

**Srpsko hemijsko društvo**  
Serbian Chemical Society



**52. SAVETOVANJE  
SRPSKOG HEMIJSKOG  
DRUŠTVA**

**PROGRAM**

**i**

**KRATKI IZVODI RADOVA**

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the Serbian Chemical Society**

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*Ova knjiga sadrži kratke izvode  
tri plenarna predavanja (PP),  
tri predavanja po pozivu (PPP) i  
137 saopštenja prihvaćenih  
za prezentovanje na Savetovanju,  
od čega 12 usmenih (O) i 125 postera (P).*

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*na desnoj strani iznad naslova rada.*

*This book contains short abstracts of  
3 Plenary Lectures (PP),  
3 Invited Lectures (PPP),  
137 contributions accepted  
for the presentation at the Meeting,  
of which 12 oral (O) and 126 poster (P) presentations*

*The Proceedings of the papers (consisting of four pages)  
are published on compact-disk (CD),  
as an integral part of the Meeting material.  
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# PROGRAM

**Petak, 29. maj 2015. / Friday, May 29, 2015**

09.00 – 15.00 **Registracija – Tehnološki fakultet, Sala 12**  
Registration – Lecture Room 12

**Hol Poljoprivrednog fakulteta / Faculty of Agriculture – Lobby**

09.00– 10.00 **Postavljanje postera / Poster Mounting (Sesija 1 / Session 1)**

**Poljoprivredni fakultet – Amfiteatar / Faculty of Agriculture – Amphitheatre**

10.00 – 10.30 **Svečano otvaranje / Opening Ceremony**

**Plenarna predavanja / Plenary Lectures**  
**Predsedavajuća / Chairperson: Ljiljana Jovanović**

10.30 – 11.15 **Nenad M. Kostić** Texas A & M University, Commerce, Texas, USA

**PP 1 Mechanisms of electron-transfer reactions between dynamic metalloproteins**

**Predsedavajuća / Chairperson: Dušanka Milojković-Opsenica**

11.15 – 12.00 **Károly Heberger**, Research Centre for Natural Sciences, Hungarian Academy of Sciences, Budapest, Hungary

**PP 2 Pattern recognition methods in food chemistry**

12.00 – 12.15 **Pauza / Pause**

**Predavanja po pozivu / Invited Lectures**

**Predsedavajući / Chairperson: Slavica Ražić**

12.15 – 12.45 **Olgica Nedić Aleksandar Dekanski\***

**PPP 1**

Institut za primenu nuklearne energije (INEP), Univerzitet u Beogradu, Srbija

\*Institut za hemiju, tehnologiju i metalurgiju, CEH, Univerzitet u Beogradu, Srbija

**Jubilej 85 godina štampanja časopisa „Journal of the Serbian Chemical Society“:**  
**Šta misle saradnici?**

Celebration of the 85 years of publishing of the Journal of the Serbian Chemical Society: What do associates think?

12.45 – 13.15 **Daniela Šojić, Biljana Abramović**

**PPP 2**

Univerzitet u Novom Sadu, Prirodno-matematički fakultet, Departman za hemiju, biohemiju i zaštitu životne sredine, Novi Sad, Srbija

**Fotokatalitička razgradnja pesticida u vodenoj sredini**

Photocatalytic degradation of pesticides in aqueous media

**Hol Poljoprivrednog fakulteta / Faculty of Agriculture – Lobby**

13.15 – 14.30 **Koktel / Cocktail**

14.15 – 15.30 **Posterska sesija 1 / Poster Session 1**

**(AH P 1 – AH P 9; EH P 1 – EH P 12; FH P 1 – FH P 9; HTH P 1 – HTH P 5; HTM P 1 – HTM P 4; NIH P 1 – NIH P 5)**

15.30 – 15.45 **Uklanjanje postera / Poster Dismounting (Sesija 1 / Session 1)**

**Poseta manastirima Krušedol i Grgeteg**

Visit to the monasteries Krušedol and Grgeteg

15.45 **Polazak autobusa sa parkinga ispred Poljoprivrednog fakulteta**

Bus departure from the parking lot in front of the Faculty of Agriculture

20.00 **Zajednička večera / Social Dinner**

**Restoran Plava frajla / Restaurant Blonde Lassie**

## **Subota, 30. maj 2015. / Saturday, May 30, 2015**

**09.00 – 12.00 Registracija – Tehnološki fakultet, Sala 12**  
Registration – Lecture Room 12

### **Hol Poljoprivrednog fakulteta / Faculty of Agriculture – Lobby**

**09.00 – 09.15 Uklanjanje postera / Poster Dismounting (Sesija 1 / Session 1)**

**09.15 – 09.30 Postavljanje postera / Poster Mounting (Sesija 2 / Session 2)**

### **Poljoprivredni fakultet – Amfiteatar / Faculty of Agriculture - Amphitheatre**

**Predsedavajući / Chairperson: Janoš Čanadi**

**Plenarno predavanje / Plenary Lecture**

**09.30 – 10.15 PP3**  
**László Somsák**, Department of Organic Chemistry, University of Debrecen, Hungary  
**Sugar derivatives against diabetes**

**Predavanje po pozivu / Invited Lecture**

**10.15 – 10.45 PPP3**  
**Igor Opsenica**, Hemijski fakultet, Univerzitet u Beogradu, Srbija  
**Sinteza i antimikrobna aktivnost azepinskih i tiepinskih derivata**  
Synthesis and antimicrobial activity of azepine and thiepine derivatives

**Usmena izlaganja / Oral Presentations**

**10.45 – 11.00 OH 01**  
**D. B. Zlatković, N. S. Radulović, K. V. Mitić, P. J. Randelović, N. M. Stojanović**  
Toksičnost i anti-acetilholinesterazna aktivnost novih hibridnih jedinjenja ferocena i indola  
Toxicity and acetylcholinesterase inhibiting activity of novel ferrocene-indole hybrids

**11.00 – 11.15 OH 02**  
**И. Н. Цвијеџић, М. Танц, И. О. Јурањић, Т. Ж. Вербић, К. Т. Сујуран, Б. Ј. Дракулић**  
Интеракције 5-арил-1H-пиразол-3-карбоксилних киселина са четири изоформе хумане карбо-анхидразе из перспективе молекулског моделовања  
Interactions of 5-aryl-1H-pyrazole-3-carboxylic acids with four human carbonic anhydrase isoforms - a molecular modelling perspective

**11.15 – 11.30 NIHO 1**  
**V. D. Milanović, D. D. Trivić**  
Istorija hemije u nastavi hemije – stavovi nastavnika hemije  
History of chemistry in chemistry education – chemistry teachers attitudes

**11.30 – 11.45 NIHO 2**  
**K. B. Putica, D. D. Trivić**  
Unapređivanje naučne pismenosti učenika kroz interdisciplinarni pristup u nastavi prirodnih nauka  
Improvement of students' scientific literacy through interdisciplinary approach to science teaching

**11.45 – 12.00 Pauza / Pause**

### **Hol Poljoprivrednog fakulteta / Faculty of Agriculture – Lobby**

**12.00 – 13.30 Posterska sesija 2 / Poster Session 2**  
(HI P 1 – HI P 16; HŽS P 1 – HŽS P 16; NH P 1 – NH P 7)

**13.30 – 14.00 Osveženje / Coffee Break**

**13.30 – 13.45 Uklanjanje postera / Poster Dismounting (Sesija 2 / Session 2)**

**13.45 – 14.00 Postavljanje postera / Poster Mounting (Sesija 3 / Session 3)**

### **Poljoprivredni fakultet – Amfiteatar / Faculty of Agriculture - Amphitheatre**

**Usmena izlaganja / Oral Presentations**

**Predsedavajući / Chairperson: Goran Bošković**

**14.00 – 14.15 TH 01**  
**D. M. Popović, I. O. Juranić**  
Uticaj proteinskog okruženja na redoks osobine kofaktora: Redoks potencijal sintetičkih citohroma b  
Influence of protein environment on redox properties of cofactors: Redox potentials of artificial cytochrome b

**14.15 – 14.30 EH 01**  
**I. Lović, D. Mijin, M. Jovanović, O. Glavaški, T. Zeremski, S. Petrović, M. Avramov Ivić**  
Electrochemical determination and degradation of tebuconazole on gold electrode followed by GC-MS analysis  
Elektrohemijsko određivanje i degradacija tebukonazola na elektrodi od zlata praćena GC-MS analizom



- 14.30 – 14.45** **AH O 1** **U. Gašić, J. Trifković, Z. Kelemen, B. Marošanić, Ž. Tešić, D. Milojković-Opsenica**  
Određivanje botaničkog porekla meda na osnovu sadržaja šećera i odnosa stabilnih izotopa ugljenika  
*Botanical discrimination of honeys by sugar and stable carbon isotope ratio analysis*
- 14.45 – 15.00** **HI O 1** **J. M. Vuksanović, N. M. Todorović, M. Lj. Kijevčanin, S. P. Šerbanović, I. R. Radović**  
Separation of heptane + methanol azeotrope using choline chloride + dl-malic acid deep eutectic solvent  
*Razdvajanje azeotropne smeše heptan + metanol korišćenjem eutektičke smeše holin hlorida i dl-jabučne kiseline kao rastvarača*
- 15.00 – 15.15** **BT O 1** **M. Šokarda Slavić, Z. Vujčić, N. Božić**  
Hidroliza koncentrovane suspenzije sirovog skroba  $\alpha$ -amilazom iz *Bacillus licheniformis* 9945a  
*Hidroliza koncentrovane suspenzije sirovog skroba  $\alpha$ -amilazom iz Bacillus licheniformis 9945a*
- 15.15 – 15.30 \*** **HTH O 1** **N. R. Ivanović, V. B. Mihailović, A. R. Ćirić, S. Kreft, Lj. G. Joksović, P. T. Đurđević**  
Optimizacija uslova ultrazvučne ekstrakcije za određivanje sadržaja fenola i antioksidativni kapacitet divljih jabuka  
*Optimization of ultrasound-assisted extraction conditions for phenolic content and antioxidant capacity of wild apple fruits*
- 15.30 – 15.45** **BH O 1** **A. Cvetanović, J. Švarc-Gajić, Z. Zeković, P. Mašković, M. Radojković, D. Adamović**  
Anti-proliferativna i anti-mikrobna aktivnost ekstrakata kamilice dobijenih pregrijanom vodom  
*Anti-proliferative and anti-microbial activity of chamomile extracts obtained by superheated water*
- 15.45 – 16.00** **Pauza / Pause**

**Hol Poljoprivrednog fakulteta / Faculty of Agriculture – Lobby**

- 16.00 – 17.15** **Posterska sesija 3 / Poster Session 3**  
**(BH P 1 – BH P 10; BT P 1 – BT P 3; MAT P 1 – MAT P 3; OH P 1 – OH P 26)**
- 17.15 – 17.30** **Uklanjanje postera / Poster Dismounting (Sesija 3 / Session 3)**
- 17.30** **Dodela IUPAC poster nagrada i zatvaranje Savetovanja**  
*IUPAC poster prize award and Closing Ceremony*

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\* Rad **BT O 2** - J. Trbojević Ivić, D. Veličković, A. Dimitrijević, D. Bezbradica, M. Gavrović Jankulović, N. Milosavić: Stabilizacija lipaza iz *Candida rugosa* jednostavnom i efikasnom imobilizacijom na hidroksiapatitu / *Candida rugosa lipase stabilization by simple and effective immobilisation on hydroxyapatite*, prvobitno predviđen za usmeno izlaganje u ovom terminu, biće prezentovan kao poster: **Posterska sesija 3, subota 30. maj, 16.15 – 17.45.**



**KRATKI IZVODI**  
**ABSTRACTS**





## Plenarna predavanja / Plenary Lectures

PP 1

### Mechanisms of electron-transfer reactions between dynamic metalloproteins

Tijana Ž. Grove, G. Matthias Ullmann\* and Nenad M. Kostić\*\*

Iowa State University, Ames, Iowa 50011, USA

\*University of Bayreuth, BGI 95447 Bayreuth, Germany

\*\*Texas A&M University, Commerce, Texas 75429, USA.

Oxidoreduction reactions of metalloproteins are important because they occur in photosynthesis and respiration. Cytochrome *c*<sub>6</sub> and cytochrome *f* react by three different electron-transfer mechanisms under similar conditions. Protein docking and kinetics of the photoinduced electron-transfer reaction  ${}^3\text{Zncyt } c_6 + \text{cyt } f(\text{III}) \rightarrow \text{Zncyt } c_6^+ + \text{cyt } f(\text{II})$  were studied by laser photolysis. This net reaction occurs between associated proteins with the rate constant  $k^{\text{pr}}$  and between colliding proteins with the rate constant  $k^{\text{tr}}$ . The viscosity independence of  $k^{\text{pr}}$ , the heme-heme electronic coupling, and reorganizational energy show that the reaction between associated proteins is *true* electron transfer. The viscosity dependence of  $k^{\text{tr}}$  and a remarkable break at 30 °C in the Eyring plot for the reaction between colliding proteins reveal mechanisms that are reversibly switched as temperature changes. Protein friction parameters below and above 30 °C differ. Colliding proteins undergo *coupled* electron transfer below 30 °C and *gated* electron transfer above 30 °C. Brownian dynamics simulations reveal two dynamic ensembles of protein-protein configurations “bridged” by relatively few configurations through which the dynamic ensembles interconvert.

### Mehanizmi reakcija prenosa elektrona između dinamičnih metaloproteina

Tijana Ž. Grouv, G. Matijas Ulman\* i Nenad M. Kostić\*\*

Državni univerzitet Ajove, Ejms, Ajova 50011, SAD

\*Univerzitet Bajrota, BGI 95447 Bajrot, Nemačka

\*\*Teksasški A&M univerzitet, Komers, Teksas 75429, SAD.

Oksidoredukcione reakcije metaloproteina važne su jer učestvuju u fotosintezi i respiraciji. Citohrom *c*<sub>6</sub> i citohrom *f* reaguju trima različitim mehanizmima prenosa elektrona pod sličnim uslovima. Uzajamno “uklapanje” proteina i kinetika fotoindukovanog prenosa elektrona  ${}^3\text{Zncyt } c_6 + \text{cyt } f(\text{III}) \rightarrow \text{Zncyt } c_6^+ + \text{cyt } f(\text{II})$  proučavani su laserskom fotolizom. Ova ista ukupna reakcija ima konstantu brzine  $k^{\text{pr}}$  kad se događa među asosovanim proteinima, ali konstantu brzine  $k^{\text{tr}}$  kad se događa među sudarenim proteinima. Nezavisnost  $k^{\text{pr}}$  od viskoznosti rastvora, hem-hem elektronsko kuplovanje i energija reorganizacije ukazuju da je reakcija među asosovanim proteinima *pravi* prenos elektrona. Zavisnost  $k^{\text{tr}}$  od viskoznosti rastvora i značajan prelom na 30 °C u Ajringovom grafiku za reakciju među sudarenim proteinima otkrivaju mehanizme koji se reverzibilno smenjuju sa promenom temperature. Parametri trenja proteina ispod i iznad 30 °C razlikuju se. Sudareni proteini učestvuju u *kuplovanom* prenosu elektrona ispod 30 °C i prenosu elektrona “*sa zadržskom*” iznad 30 °C. Simulacije Braunove dinamike ukazuju na dva dinamična ansambla protein-protein konfiguracija “premošćena” relativno malobrojnim konfiguracijama kroz koje se ona dva dinamična ansambla pretvaraju jedan u drugi.

## Pattern Recognition Methods in Food Chemistry Metode prepoznavanja obrazaca u hemiji hrane

Károly Heberger

Research Centre for Natural Sciences, Hungarian Academy of Sciences,  
H-1117 Budapest XI., Magyar tudósok krt 2

Multivariate (chemometric) data analysis has become a fundamental task in analytical chemistry due to the great quantity of analytical information provided by modern analytical instruments. The multivariate methods can be distributed in two fundamental class supervised pattern recognition and unsupervised ones: the first one aims to establish a classification model based on experimental data in order to assign unknown samples to a previously defined sample class based on its pattern of measured features. The unsupervised techniques do not use any class membership information in the modeling phase. The supervised pattern recognition techniques mostly used in food analysis were reviewed some time ago and *common misconceptions and errors were highlighted* [1].

Principal component analysis is strongly advisable to be performed before using any supervised pattern recognition technique. Linear Discriminant Analysis is the most commonly used supervised technique; whereas Quadratic Discriminant Analysis and Classification and Regression Trees are rarely used, despite the good results that they can achieve.

Some interesting application shows high potential:

Impact assessment for attributes of Just About Right (JAR) scale on overall liking can be solved by generalized pair-wise correlation method. (GPCM) [2]. In product development using JAR scales, it is important to identify precisely, which direction of a given attribute affects hedonic scores the most. JAR data needs to be transformed based on the dummy variable approach. Then, GPCM gives those attributes in that order, which should be increased/decreased to gain higher consumer liking score. Moreover, GPCM utilizes correct statistical tests for ranking: McNemar's, Chi-square, Conditional Fisher's and the Williams' *t*-test.

Mineral, spring and tap water samples of different geographical origins (7 classes) were distinguished using sensory evaluation, electronic tongue measurement, inductively coupled plasma atomic emission spectroscopy and ion chromatography. Samples from the same geographical origin were correctly classified by chemical analysis and electronic tongue (100 %), but only ~80 % classification rate can be achieved by sensory evaluation. The two brands bottled at the same district are rooted in identical geographical layers. So these waters sold under different brand names, but originated from the same geographical region cannot be distinguished [3].

### References

1. Luis A. Berrueta, Rosa M. Alonso-Salces, Károly Héberger, Supervised pattern recognition in food analysis, *Journal of Chromatography A*, **1158** (2007) 196–214.
2. Attila Gere, László Sipos, Károly Héberger Generalized pair-wise correlation and method comparison: impact assessment for JAR attributes on overall liking, *Food Quality and Preference*, **43** (2015) 88–96. DOI:10.1016/j.foodqual.2015.02.017
3. László Sipos, Zoltán Kovács, Virág Sági-Kiss, Tímea Csiki, Zoltán Kókai, András Fekete, Károly Héberger, Discrimination of mineral waters by electronic tongue, sensory evaluation and chemical analysis, *Food Chemistry*, **135** (2012) 2947–2953.

PP 3

## Sugar derivatives against diabetes

László Somsák

*Department of Organic Chemistry, University of Debrecen,  
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*Diabetes mellitus* (DM) is one of the most frequent causes of death and disability all over the world first of all due to its long term complications. Epidemic spread of the non-insulin-dependent (or type II) DM, representing more than 90 % of the diagnosed cases, has induced extensive efforts in both academia and industry to find new therapies. Several fields of antidiabetic research are based on carbohydrate derivatives. An outlook will be given to the most important glycomimetic drugs in current clinical practice, e. i. inhibitors of intestinal  $\alpha$ -glucosidases and renal sodium dependent glucose cotransporters.

The main focus of the lecture will be directed to monosaccharide derived inhibitors of glycogen phosphorylase (GP), the main regulatory enzyme of glycogen metabolism, a validated target in combating type II DM. Presented will be the design principles, synthetic challenges, results of enzyme kinetic, crystallographic, computational and physiological studies related to such inhibitors as well as the interplay of all of these investigations in producing potentially antidiabetic new sugar derivatives.





## Predavanja po pozivu / Invited Lectures

PPP 1

### Јубилеј 85 година штампања часописа Journal of the Serbian Chemical Society: Шта мисле сарадници?

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Поводом 85 година штампања часописа "Journal of the Serbian Chemical Society" и изласка свеске број 80, Уредништво је спровело анкету ради утврђивања шта аутори, рецензенти и подручни уредници мисле о часопису. Анкета је спроведена електронски, а послата је укупно на 2422 адресе. Добијена су 832 одговора, који су показали да је већина сарадника задовољна постојећим начином рада и уређивачком политиком. Просечне додељене оцене су биле 4,3 код подручних уредника, 4,0 код рецензента и 3,7 код аутора. Подручни уредници су указали на тешкоће у налажењу добрих рецензента и великом степену одбијања или неодговарања позваних рецензента. Рецензенти су сугерисали једноставнији "on-line" систем за слање рецензија и дужи рок за рецензирање. Аутори су, такође, предложили једноставнији "on-line" систем за подношење радова, тражили су јаснија упутства за израду слика, бољи избор рецензента и, пре свега, бржи поступак рецензирања. Реакција Уредништва на резултате анкете је увођење потпуно аутоматизованог "on-line" система пријаве и руковања радовима, поједностављење Упутства за ауторе и Рецензентског формулара, а у току је тражење решења за убрзање рецензирања. Новине су уведене марта 2015.

### Celebration of the 85 years of publishing of the Journal of the Serbian Chemical Society: What do associates think?

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This year we are celebrating 85 years of publishing of the Journal of the Serbian Chemical Society and the 80<sup>th</sup> volume. On this occasion, members of the Editorial board investigated what do authors, reviewers and Sub-editors think about the journal. E-mail questionnaire was sent to 2422 addresses and 832 responses were received. Most associates were satisfied with the journal and the managing policy, giving overall grades 4.3 (Sub-Editors), 4.0 (reviewers) and 3.7 (authors). Sub-Editors pointed to the difficulties in finding good reviewers. Reviewers asked for complete automatic „on-line“ system for sending reports and longer period for reviewing. Authors also suggested „on-line system“ for submitting articles, more precise instructions for figures, better choice of reviewers and faster reviewing. Consequently, since March 2015, automatic „on-line“ system was introduced both for submitting and reviewing, instructions were simplified and we are searching for the solution to speed-up reviewing.

PPP 2

## **Fotokatalitička razgradnja pesticida u vodenoj sredini**

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Poslednjih decenija se poklanja velika pažnja proučavanju procesa uklanjanja perzistentnih organskih polutanata iz vode, kakvi su pesticidi, koji ispoljavaju hemijsku stabilnost i rezistentnost na biorazgradnju. Mnoga istraživanja iz ove oblasti su zasnovana na primeni vodene suspenzije poluprovodnika u prisustvu odgovarajućeg izvora zračenja u cilju uklanjanja polutanata. Proces heterogene fotokatalize se zasniva na stvaranju visoko-reaktivnih radikala koji mogu da mineralizuju organske supstance do jedinjenja koja nisu toksična, kao što su voda, ugljenik(IV)-oksid i odgovarajući neorganski joni. Na primeru razgradnje odabranih herbicida biće prikazan uticaj različitih faktora (početna koncentracija polaznog jedinjenja, masena koncentracija katalizatora, elektron-akceptori i njihova uloga u rekombinaciji, pH rastvora i drugi) na kinetiku heterogene fotokatalize, kao i mehanizam razgradnje primenom UVA i simuliranog sunčevog zračenja. U cilju praćenja nestajanja polaznog jedinjenja i nastajanja/nestajanja intermedijera tokom procesa fotokatalitičke razgradnje herbicida, korišćeni su različiti analitički postupci kao što su tečna hromatografija, spektrofotometrija, jonska hromatografija, tehnika određivanja ukupnog organskog ugljenika i druge.

## **Photocatalytic degradation of pesticides in aqueous media**

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In the recent decades great attention is paid to the study of the removal of persistent contaminants from water, including pesticides, which exhibit chemical stability and resistance to biodegradation. Numerous studies have been based on the application of water suspensions of semiconductors in the presence of appropriate light source to achieve removal of the pollutants. Photocatalytic processes are based on the formation of highly reactive radicals that may mineralize organic substances to non-toxic compounds, such as water, carbon dioxide and appropriate inorganic ions. Influence of various factors (initial concentration of substrate, loading of catalyst, electron acceptors and their role in recombination, reaction pH, etc.) on the kinetics of heterogeneous catalysis of some herbicides, as well as mechanism of degradation using UVA light and simulated sunlight will be discussed. In order to evaluate the disappearance of starting compound and appearance/disappearance of intermediates formed during the photocatalytic degradation of herbicides, different analytical techniques such as liquid chromatography, spectrophotometry, ion chromatography, TOC technique, etc. were used.

**Acknowledgment:** *The authors acknowledge financial support of the Ministry of Education, Science and Technological Development of the Republic of Serbia (Project No. 172042) and Provincial Secretariat for Science and Technological Development of Vojvodina, Republic of Serbia.*

## Sinteza i antimikrobna aktivnost azepinskih i tiepinskih derivata

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Heterociklična jedinjenja 5*H*-dibenz[*b,f*]azepinske i dibenzo[*b,f*]tiepinske strukture našla su primenu u medicini. Karbamazepin **1** koji pripada klasi dibenzazepina našao je primenu u lečenju epilepsije, neuropatskih bolova i bipolarnih poremećaja, dok se dibenzotiepinski derivat Zotepin **2** upotrebljava u lečenju šizofrenije.

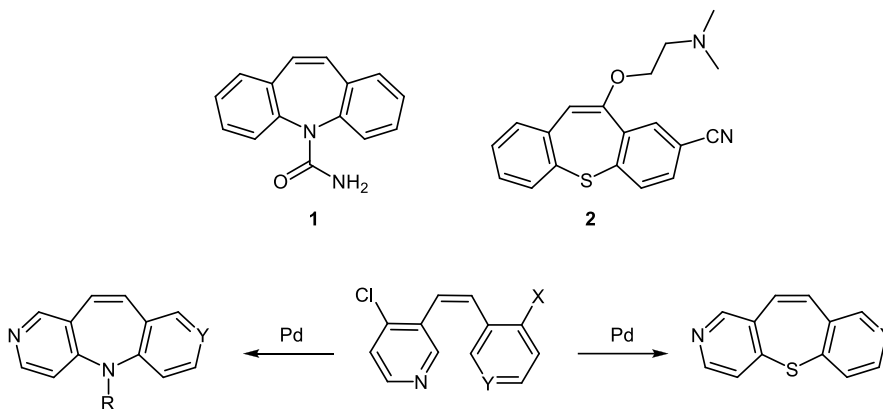
U okviru naših istraživanja razvili smo novu metodologiju za formiranje azepinskog i tiepinskog prstena iz odgovarajućih *Z*-stilbena. Dvostruko *N*- i *S*-arilovanje *orto*-halogen supstituisanih *Z*-stilbena odvija se u prisustvu paladijuma kao katalizatora. Sintetisani azepinski i tiepinski derivati pokazuju izraženu antimikrobnu aktivnost.

## Synthesis and antimicrobial activity of azepine and thiepine derivatives

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The tricyclic moieties of 5*H*-dibenz[*b,f*]azepine and dibenzo[*b,f*]thiepine character are important heterocyclic pharmacophores of a number of drugs. Carbamazepine **1**, a member of dibenzazepine class, is an anticonvulsant used to treat seizures, nerve pain and bipolar disorder, while dibenzothiepine derivative Zotepine **2** is used to treat schizophrenia. Recently, we have developed a simple and efficient Pd-catalyzed method for the synthesis of 5*H*-pyridobenzazepine and pyridobenzothiepine compounds. Our approach, based on a Pd-catalyzed formation of C–N and C–S bonds, enables the synthesis of azepine and thiepine derivatives from corresponding *Z*-stilbene precursors. All synthesized compounds were evaluated for their *in vitro* antimicrobial activity against bacterial and fungal pathogenic strains.



**Acknowledgment:** This research was supported by the Ministry of Education, Science and Technological development of the Republic of Serbia (grant no. 172008).

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## Saopštenja / Contributions

### Analitička hemija / Analytical Chemistry

AH O 1

#### **Određivanje botaničkog porekla meda na osnovu sadržaja šećera i odnosa stabilnih izotopa ugljenika**

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Uprkos dugoj tradiciji proizvodnje meda u Srbiji, rezultati ispitivanja koja se odnose na sadržaj šećera i odnos stabilnih izotopa ugljenika u srpskom medu do sada nisu publikovani, suprotno velikom broju sličnih radova iz drugih zemalja. Botaničko poreklo tri najzastupljenija srpska monofloralna meda (bagremov, suncokretov i lipov) određeno je na osnovu sadržaja šećera, analiziranog primenom visoko-efikasne jonske hromatografije sa pulsno amperometrijskom detekcijom, i odnosa stabilnih izotopa ugljenika, analiziranog primenom masene spektrometrije odnosa stabilnih izotopa. Rezultati hemometrijske analize primenjene na podacima potvrđuju mogućnost razdvajanja uzoraka prema botaničkom poreklu i ukazuju na najvažnije parametre koji dovode do razdvajanja. Prema našem saznanju, ovo je prvi rad u kojem je uspešno određeno botaničko porekla meda na osnovu odnosa stabilnih izotopa ugljenika.

*Zahvalnica: Ova istraživanja su podržana od strane Ministarstva prosvete, nauke i tehnološkog razvoja Republike Srbije (projekat br. 172017).*

#### **Botanical discrimination of honeys by sugar and stable carbon isotope ratio analysis**

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Despite the long beekeeping tradition, the issues of sugar content and stable carbon isotope ratio of Serbian honey have not, insofar been covered by scientific publications, contrary to the large number of foreign papers dealing with these issues. The botanical origin of three most common Serbian unifloral honey types (acacia, sunflower and lime) was investigated by analysis of the sugar content using High-Performance Anion-Exchange Chromatography with Pulsed Amperometric Detection (HPAEC-PAD) and the stable carbon isotope ratio using Elemental Analyzer - Isotope Ratio Mass Spectrometry ( $\delta^{13}\text{C}$ -EA-IRMS). Pattern recognition methods applied on the data, confirm the possibility of distinguishing among samples of specific botanical origin and reveal the most important variables for such discrimination. To our best knowledge, this is a first report of successful determination of botanical origin of honey based on stable carbon isotope ratio data.

*Acknowledgements: This research was supported by the Ministry of education, science and technological development of the Republic of Serbia (Grant No. 172017).*



## АН Р 1

### Спектрофотометријско одређивање садржаја платине у Pt/TiO<sub>2</sub> композитном катализатору

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Испитивана је могућност одређивања садржаја платине у композитним катализаторима индиректном спектрофотометријском методом. Одређен је састав платине у композиту Pt/TiO<sub>2</sub> који је синтетисан сол–гел поступком. Испитивани чврсти узорак композита је третиран раствором HCl и HNO<sub>3</sub> како би се платина превела у јонски облик, а потом је снимљен UV–Vis спектар добијеног раствора и упоређен са спектрима припремљених раствора хлороплатинасте киселине. Спектри припремљених раствора садрже изражени апсорпциони врх у области таласних дужина од 260–265 nm, који је искоришћен за квантитативно одређивање платине. Резултати спектрофотометријског одређивања показали су добро слагање са просечним садржајем платине добијеним методом спектроскопије X-зрака са дисперзијом енергије (EDS).

### Spectrophotometric determination of Pt in Pt/TiO<sub>2</sub> composite catalyst

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Possibility for spectrophotometric determination of platinum loading in Pt/TiO<sub>2</sub> composite was examined. The platinum from a composite sample is converted into ionic form by a mixture of hydrochloric and nitric acid and the absorbance of a sample was measured. The Pt concentration is determined from the calibration curve gained by UV–Vis measurements on chloroplatinic acid solutions. The results are compared to those obtained by EDS method. Spectrophotometric measurement revealed 18.4 wt. % and EDS method showed around 18 wt.% of platinum. The data obtained by the two analytical methods give valuable insights into characteristic structure–activity relationship for the synthesized composite.

AH P 2

### **Ispitivanje mogućnosti primene bizmut film elektrode za određivanje 3-dehidro-dezoksiholne kiseline**

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Voltametrijska karakterizacija i određivanje 3-dehidro-dezoksiholne kiseline (3-DH-DHK) je izvršeno u Britton-Robinsonovom puferu kao pomoćnom elektrolitu primenom optimizovane adsorptivne voltametrijske metode sa pravougaonim talasima i elektrode na bazi staklastog ugljenika modifikovane *ex situ* pripremljenim bizmutovim filmom. Najbolje definisan i najintenzivniji redukcionni signal 3-DH-DHK javlja se pri pH 11,82 sa maksimumom pika na oko -1,5 V, zbog toga je ova pH vrednost odabrana za analitičke svrhe. Relativno dobra linearnost ove metode je postignuta u nižoj  $\mu\text{g mL}^{-1}$  oblasti. Reproductivnost analitičkih signala je okarakterisana relativnom standardnom devijacijom manjom od 5,0 %.

Za praćenje toka elektrolize 3-DH-DHK pri konstantnom potencijalu primenjena je  $^1\text{H}$  NMR tehnika.

**Zahvalnica:** Autori se zahvaljuju za finansijsku podršku Ministarstvu nauke i tehnološkog razvoja Republike Srbije (Projekti br. 172059 i 172012).

### **Investigation of the applicability of bismuth film electrode for the determination of 3-dehydro-deoxycholic acid**

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Voltammetric characterization and determination of 3-dehydro-deoxycholic acid (3-DH-DCA) were performed in aqueous Britton-Robinson buffer solution as supporting electrolyte by optimized adsorptive square-wave voltammetry and glassy carbon electrode modified with *ex situ* prepared bismuth film. The well shaped and most intensive reduction signal of 3-DH-DCA was obtained at pH 11.82 with peak maximum at about -1.5 V, therefore this pH value was chosen for analytical purposes. Relative good linearity of this method was obtained at the lower  $\mu\text{g mL}^{-1}$  range. The reproducibility of the analytical signals was characterized by the relative standard deviation less than 5.0 %.

The progress of 3-DH-DCA electrolysis at constant potential was monitored by  $^1\text{H}$  NMR technique.

**Acknowledgement:** The authors acknowledge the financial support of the Ministry of Science and Technological Development of the Republic of Serbia (Projects No. 172059 and 172012).

AH P 3

**Određivanje odnosa stabilnih izotopa kiseonika vode u vinima iz Srbije**

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Merenje odnosa izotopa  $^{18}\text{O}/^{16}\text{O}$  jedna je od najvažnijih primena masene spektrometrije odnosa izotopa. Primenjuje se u ispitivanjima hidrološkog ciklusa, u kontroli autentičnosti pića i za praćenje metabolita koji su obeleženi izotopom  $^{18}\text{O}$ . Određivanje odnosa  $^{18}\text{O}/^{16}\text{O}$  u vodi iz vina se primenjuje da bi se definisalo geografsko poreklo, i predloženo je kao metoda za otkrivanje dodatka vode u procesu proizvodnje vina. Analizirano je ukupno 46 uzoraka vina (berbe 2009-2014), iz različitih regiona Srbije. Sve analize su urađene na Finnigan GasBench II periferalu, sa autosemplerom, povezanim sa Delta V Advantage masenim spektrometrom. Dobijene vrednosti odnosa izotopa  $^{18}\text{O}/^{16}\text{O}$ , u ispitivanim uzorcima vina, ukazuju na razliku između berbi. Naši rezultati pokazuju da se odnos izotopa potencijalno može primeniti za razlikovanje sorti vina i berbi, kao i za ispitivanje zavisnosti geografskog porekla vina od geografskih karakteristika (geografska visina i širina) i klimatskih uslova (vlažnost i količina padavina).

**Determination of oxygen stable isotope ratio of water content  
in wines from Serbia**

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Rada M. Baošić and Živoslav Lj. Tešić

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The measurement of the  $^{18}\text{O}/^{16}\text{O}$  ratio of water is one of the most important applications of isotope ratio mass spectrometry. Applications range from studies of variations in natural abundance in the hydrologic cycle to authenticity control on beverages and to the use of  $^{18}\text{O}$  enriched tracers in human and animal metabolic studies. The determination of oxygen ratio ( $^{18}\text{O}/^{16}\text{O}$ ) from the water content of the wine (wine water), have been used to define the geographical origin, and it is proposed as a method to detect the addition of water to wine. Total of 46 samples of wines, vintages 2009-2014, from different regions of Serbia were analyzed by continuous flow technique in which the sample is entrained in a carrier gas. All analyses were done on Finnigan GasBench II with autosampler-assisted loop injection coupled with Delta V Advantage mass spectrometer. Our results showed differences between vintages.  $^{18}\text{O}/^{16}\text{O}$  values from the wine water could potentially be used to distinguish between the varieties of grapes and the vintages of wines, and to study the relationship between the place of origin and its geographical characteristics (altitude and latitude) and climatic conditions (temperature, precipitation and humidity).



AH P 4

## Određivanje fenolnog profila vrsta *Matricaria chamomilla* i *Matricaria inodora* pomoću UHPLC/HRMS tehnike

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Kamilica je jedna od najpopularnijih lekovitih biljaka. Zahvaljujući prisustvu različitih fitohemikalija, ispoljava antiinflamatornu, antioksidantnu i druge biološke aktivnosti. U ovom radu, određen je fenolni profil *M. chamomilla* i *M. inodora*. U metanolnim ekstraktima cvasti, herbe i rizoma fenolna jedinjenja su kvantifikovana po prethodno razvijenoj metodi Kečkeš i dr. (2013). Trideset sedam jedinjenja je identifikovano i kvantifikovano u ekstraktima pomoću UHPLC-LTQ OrbiTrap masenog spektrometra. Većina jedinjenja prisutna je u većoj koncentraciji u spojenim ekstraktima nadzemnih i podzemnih delova biljke, u odnosu na ekstrakte cvasti (Tabela 1).

**Tabela 1: Dominantna jedinjenja u ekstraktima *M. chamomilla* i *M. inodora***

Biljka	Ekstrakt	Dominantna jedinjenja (µg fenola po L ekstrakta)
<i>Matricaria chamomilla</i>	cvast	Galna kiselina(57.34), Vanilinska kiselina (8.077)
	herba & rizom	3-O-Kafeoilhinska kiselina (2688), Luteolin 7-O-glukozid (1536), Luteolin (72.95), Kvercetin (244.6), Eskuletin (662.4), Katehin 3-galat (184.6)
<i>Matricaria inodora</i>	cvast	Galna kiselina (70.29), p-Hidroksibenzoeva kiselina (10.47)
	herba & rhizom	3-O-Kafeoilhinska kiselina (6079), Luteolin 7-O-glukozid (761.2), Luteolin (448.8), Kvercetin (317.2), Vanilinska kiselina (243.6), Kvercetin 3-O-galaktozid (155.8)

## The determination of phenolic profile of *Matricaria chamomilla* and *Matricaria inodora* using UHPLC/HRMS technique

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Chamomile is one of the most popular medicinal plants. Due to the presence of various phytochemicals, it exhibits anti-inflammatory, antioxidant and other biological activities. In this paper, the phenolic profile of *M. chamomilla* and *M. inodora*, was determined. Plant phenolics were quantified in methanolic extracts of flowers, herbs and rhizomes by previously developed method by Kečkeš *et al.* (2013). Thirty seven compounds were identified and quantified in the extracts by UHPLC-LTQ OrbiTrap mass spectrometer. Most compounds were more abundant in joined extracts of above and underground plant parts, comparing to the flower extracts (Table 1).

**Table 1. Dominant compounds in *M. chamomilla* and *M. inodora* extracts**

Plant	Extract	Dominant compounds (µg of phenolics per L of extract)
<i>Matricaria chamomilla</i>	flower	Gallic acid (57.34), Vanillic acid (8.077)
	herb & rhizome	3-O-Caffeoylquinic acid (2688), Luteolin 7-O-glucoside (1536), Luteolin (72.95), Quercetin (244.6), Aesculetin (662.4), Catechin 3-gallate (184.6)
<i>Matricaria inodora</i>	flower	Gallic acid (70.29), p-Hydroxybenzoic acid (10.47)
	herb & rhizome	3-O-Caffeoylquinic acid (6079), Luteolin 7-O-glucoside (761.2), Luteolin (448.8), Quercetin (317.2), Vanillic acid (243.6), Quercetin 3-O-galactoside (155.8)

### **Određivanje sadržaja kiselih funkcionalnih grupa na površini biougljeva primenom Boehm-ovih titracija**

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Biougalj se dobija pirolizom iz organskog otpada. Smatra se „modifikatorom“ zemljišta i koristi se kao đubrivo i kao rezervoar za skladištenje ugljenika. Kvantifikacija funkcionalnih grupa je važna zbog hemijske reaktivnosti biougljeva koji imaju uticaj na svojstva zemljišta. U istraživanju su korišćena 3 uzorka biouglja različitog porekla i jedan referentni, aktivni ugalj. Sadržaj kiselih funkcionalnih grupa je određivan titracijom po Boehm-u na dva načina: uvođenje N<sub>2</sub> tokom titracije i tretman sa BaCl<sub>2</sub> [1]. Optimizovan je način pripreme tri Boehm-ova rastvora (NaOH, Na<sub>2</sub>CO<sub>3</sub>, NaHCO<sub>3</sub>, koncentracije 0,05 mol/dm<sup>3</sup>). Ispitivan je način pripreme standardnog rastvora NaOH kao i način uklanjanja CO<sub>2</sub>, pre ili u toku titracije osetljive na prisustvo CO<sub>2</sub> i rastvorljivih organskih komponenti iz ekstrakta biouglja i vreme uvođenja N<sub>2</sub>. Da bi se proverila efikasnost ovih metoda, ekstrakti su snimani spektrofotometrijski na talasnoj dužini 250 nm.

### **Determination of oxygen surface groups on biochars using Boehm-titration method**

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Biochar is obtained from the pyrolysis of organic waste. Biochar is a soil modifier due to its fertilizing effect and its potential for carbon storage. Quantification of functional groups is interesting because chemical reactivity of biochar can have effect on soil properties. In this research different samples of biochar are used and one as referent sample. Oxygen surface groups were determined by Boehm titration in two manners: sparging with N<sub>2</sub> during titration and the treatment with BaCl<sub>2</sub>[1]. The preparation of three Boehm's solutions was optimized (NaOH, Na<sub>2</sub>CO<sub>3</sub>, NaHCO<sub>3</sub>, conc. 0,05 mol/dm<sup>3</sup>). The preparation of the NaOH standard solution was investigated as well as the method of removing CO<sub>2</sub> before or during titration which is sensitive in the presence of CO<sub>2</sub> and dissolved organic compounds and the time of sparging by N<sub>2</sub>. In order to check the efficiency this methods, spectrophotometry is used at a wavelength of 250 nm.

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### **Reference**

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## **Određivanje dihidrotestosterona i testosterona u tranzitornoj zoni prostate primenom LC/MS hromatografije**

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Dihidrotestosteron (DHT) nastaje dejstvom  $5\alpha$  reduktaze (SRD5A1/2) na testosteron u tkivu prostate. Povećane vrednosti DHT su odgovorne za nastanak benigne hiperplazije prostate (BHP), a verovatno podstiču i rast karcinoma prostate (CaP). Osim toga, DHT dovodi i do androgene alopecije, tj. ćelavosti.

Cilj ovog rada je određivanje odnosa DHT i testosterona u tkivu prostate, primenom visoko efikasne tečne hromatografije sa MS detekcijom.

Kod 93 bolesnika je zbog sumnje na postojanje CaP, vršena biopsija prostate vođena pomoću transrektalnog ultrazvuka (TRUS). Tokom biopsije su uzimana i dva dodatna uzorka iz tranzitorne zone (TZ) prostate, za određivanje tkivne koncentracije testosterona i DHT. Uzorak tkiva je zamrzavan u tečnom azotu, usitnjen i prečišćen uz pomoć SPE ekstrakcije. Koncentracija testosterona i DHT je određivana primenom reverzno fazne tečne hromatografije uz elektron-sprej jonizaciju i masenu spektroskopiju.

Koncentracija testosterona je iznosila 0.104 – 3.219 ng/g (medijana: 0.787 ng/g), a koncentracija DHT, 0.983- 24.985ng/g (medijana: 10.268 ng/g).

## **Determination of dihydrotestosterone and testosterone in prostate transition zone by LC/MS chromatography**

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Dihydrotestosterone (DHT) is produced manly by the action of  $5\alpha$  reductase (SRD5A1/2) on testosterone in prostate tissue. Increased concentration of DHT in prostatic tissue is the main cause of benign prostatic hyperplasia (BPH) development and one of the factors that support prostate cancer (PCa) growth. In addition, DHT is responsible for the androgenic alopecia.

The framework of this study was to determine concentration of testosterone and DHT and their ratio in prostate tissue by the use of high performance liquid chromatography coupled with mass spectrometry.

The group of 93 patients underwent transrectal ultrasonography (TRUS) guided prostate biopsy, due to potential PCa. During the biopsy, two additional samples of the transition zone (TZ) of the prostate were taken, for the determination of testosterone and DHT. The tissue samples were frozen in liquid nitrogen, minced and purified by SPE extraction. High performance liquid chromatography coupled with electron spray ionization and mass spectrometry was used for the determination of testosterone and DHT.

Concentration of testosterone was 0.104 -3.219ng/g, (median: 0.787 ng/g) whereas concentration of DHT was 0.983-24.985ng/g (median: 10.268 ng/g).

## Primena kinetičke metode za određivanje tragova tiocijanata u realnim uzorcima

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Kinetičke metode su brze i visoko osetljive analitičke metode za određivanje tragova analiziranih supstanci ali su ograničene u njihovoj direktnoj primeni svojom niskom selektivnošću, koja obično zahteva primenu predhodne separacije. U radu je predstavljena primena jednostavne i reproduktivne kinetičko metode za određivanje tragova tiocijanata u realnim uzorcima. Primenom kinetičke metode je uspešno kvantifikovan sadržaj tiocijanata u uzorcima sa složenim matriksom - pljuvačka nepušača i pušača (dinamički opseg metode 0.29 – 5.81  $\mu\text{g cm}^{-3}$ ). Kako interferirajući joni nisu prisutni u ovom matriksu, kinetička metoda je nakon centrifugiranja direktno primenjena u analiziranim uzorcima. Odgovarajuće zapremine rastvora uzorka su analizirane po propisanoj proceduri kinetičkog određivanja, nakon dodatka 0.1 mol  $\text{dm}^{-3}$  rastvora NaOH (pH pljuvačke je  $\sim 7.5$ ), u cilju regulisanja radnog pH metode. Dobijeni rezultati su u dobroj saglasnosti sa rezultatima dobijenih uporednom spektrofotometrijskom metodom (Recovery 102.52 % za uzorke nepušača i 101.64% za uzorke pušača). U uzorcima pljuvačke nepušača nađeno je  $1.63 \pm 0.07 \mu\text{g cm}^{-3}$ , RSD = 4.20 %, dok je u uzorcima pušača nađeno  $3.71 \pm 0.12 \mu\text{g cm}^{-3}$ , RSD = 3.22 %.

## Application of kinetic method for the determination of thiocyanates traces in real samples

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Kinetic methods are simple and high sensitive for determination of traces of analysed specie, but there is limitation in their direct application because of low selectivity. Usually, separation is needed prior to analysis. In this work the application of a simple and reproducibile kinetic method for the determination of traces of thiocyanates in real samples was presented. This method was successfully applied for determination of thiocyanates in samples with complex saliva matrices of smokers and s non-mokers (dynamic range: 0.29 –5.81  $\mu\text{g cm}^{-3}$ ). Direct analysis was possible after centigution, because of absence of interference. Aproprate amounts of a sample solution were analysed according to te recommended procedure, after adding of 0.1 mol  $\text{dm}^{-3}$  solution of NaOH for adjusting pH. pH of saliva is  $\sim 7.5$ . The obtained results were in a good agreement with those obtained by comparing spectrophotometric method. The recovery value for samples of non-smokers was 102.52% and for samples of smokers 101. 64%. Concentration of thiocyanates in samples of saliva non-smokers were  $1.63 \pm 0.07 \mu\text{g cm}^{-3}$ , RSD = 4.20 %, while in saliva of smokers were  $3.71 \pm 0.12 \mu\text{g cm}^{-3}$ , RSD = 3.22 %.

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### **Staklasti ugljenik modifikovan kompozitom od bizmut-oksihlorida i višezidnih ugljeničnih nanocevi za anodno inverzno voltometrijsko određivanje Zn(II)**

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Kompozitni materijal od bizmut-oksihlorida i višezidnih ugljeničnih nanocevi u kombinaciji sa NAFION<sup>®</sup>-om je primenjen kao površinski modifikator elektrode od staklastog ugljenika (BiOCl-MWCN/GCE) za određivanje tragova Zn(II) primenom anodne inverzne voltometrije sa pravougaonim talasima (SWASV). Optimizacija metode obuhvata izbor pomoćnog elektrolita i operacione parametre SWASV metode. BiOCl-MWCN/GCE pokazuje linearni odziv Zn(II) u ispitivanoj oblasti koncentracija od 2,5 do 120  $\mu\text{g L}^{-1}$  sa računatom granicom detekcije od 0,75  $\mu\text{g L}^{-1}$  pri vremenu elektrodepozicije od 120 s i potencijalu elektrodepozicije od -1,40 V vs ZKE. Pouzdana reproduktivnost merenja od 4,8% je dobijena u slučaju analize model rastvora od 10  $\mu\text{g L}^{-1}$  Zn(II).

### **Bismuth-oxychloride-multiwalled carbon nanotube composite modified glassy carbon electrode for anodic stripping voltammetric determination of Zn(II)**

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Bismuth-oxychloride-multiwalled carbon nanotube composite material was applied as surface modifier of glassy carbon electrode (BiOCl-MWCN/GCE) in combination with NAFION<sup>®</sup> for the trace level determination of Zn(II) by square wave anodic stripping voltammetry (SWASV). The method optimization encompasses the selection of the supporting electrolyte and the operational parameters of the SWASV method. The BiOCl-MWCN/GCE showed an excellent linear response towards Zn(II) in the concentration range examined from 2.5 to 120  $\mu\text{g L}^{-1}$  with the limit of detection estimated to be 0.75  $\mu\text{g L}^{-1}$ , associated with accumulation for 120 s at a potential of -1.40 V vs SCE. A satisfactory reproducibility of 4.8% was obtained when analysing a model solution with 10  $\mu\text{g L}^{-1}$  Zn(II).

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**Štampane ugljenične elektrode modifikovane nanokompozitom na bazi paladijuma i višezidnih ugljeničnih nanocevi za određivanje H<sub>2</sub>O<sub>2</sub>**  
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Izvršena je osnovna karakterizacija nanokompozitnog materijala na bazi paladijuma i višezidnih ugljeničnih nanocevi (PdNP-MWCNT) primenom SEM-EDS-a. Zatim su pripremljene štampane ugljenične elektrode, kako nemonifikovane, tako i modifikovane sa PdNP-MWCNT na dva različita načina: zapreminski i površinski. Voltametrijsko ponašanje H<sub>2</sub>O<sub>2</sub> na pripremljenim elektrodama je proučavano primenom ciklične voltametrije. Vršeno je hidrodinamičko hronoamperometrijsko određivanje H<sub>2</sub>O<sub>2</sub> u model sistemima pri različitim radnim potencijalima u fosfatnom puferu pH 7,5 kao pomoćnom elektrolitu, pri čemu je pogodan interval radnog potencijala od -0,2 V do -0,7 V. U poslednjem koraku, na površine pripremljenih elektroda imobilizovan je enzim glukoza-oksidaza i elektrode su testirane za određivanje glukoze. Na osnovu dobijenih rezultata, zapreminska modifikacija je pokazala bolje analitičke performanse za određivanje H<sub>2</sub>O<sub>2</sub> i glukoze.

**Screen printed carbon electrodes modified with nanocomposite based on palladium and multiwalled carbon nanotubes for determination of H<sub>2</sub>O<sub>2</sub>**  
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The basic characterization of a nanocomposite material consisting of palladium and multiwalled carbon nanotubes (PdNP-MWCNT) was performed by SEM-EDS. After that, screen printed carbon electrodes were prepared, unmodified as well as modified with -MWCNT in two different ways: bulk and surface. The voltammetric behavior of H<sub>2</sub>O<sub>2</sub> at the prepared electrodes was studied by cyclic voltammetry. Hydrodynamic chronoamperometric determination of H<sub>2</sub>O<sub>2</sub> was performed at different working potentials in model systems in phosphate buffer pH 7.5 as supporting electrolyte, and the appropriate working potential ranges from -0.2 V to -0.7 V. In the last step, the enzyme glucose-oxidase was immobilized on the surface of the electrodes which were tested for the determination of glucose. Based on the obtained results, bulk modification showed better analytical performance in terms of H<sub>2</sub>O<sub>2</sub> and glucose determination.

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## Fizička hemija / Physical Chemistry

FH P 1

### **Volumetrijske osobine binarnih smeša odabrane jonske tečnosti sa vodom i etanolom**

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Gustine binarnih smeša novosintetisane jonske tečnosti 1-butyl-3-ethylimidazolijum bromida, [beim]Br, sa vodom i etanolom, su izmerene na atmosferskom pritisku u temperaturnom intervalu  $T = 293,15 - 323,15$  K u pri različitim molalitetima jonske tečnosti (za binarnu smešu jonske tečnosti sa vodom:  $0,01301 \leq m[\text{beim}]Br \leq 0,03900$  mol·kg<sup>-1</sup>; za binarnu smešu jonske tečnosti sa etanolom:  $0,00827 \leq m[\text{beim}]Br \leq 0,04083$  mol·kg<sup>-1</sup>). Iz eksperimentalnih podataka izračunate su prividne molarne zapremine, prividne molarne zapremine pri beskonačnom razblaženju, parcijalne molarne zapremine, termički koeficijent širenja, a u cilju dobijanja informacija o interakcijama između odabrane jonske tečnosti i komercijalnih molekularnih rastvarača.

### **Volumetric properties of selected ionic liquid binary mixtures with water and ethanol**

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Densities of binary mixtures of newly synthesized ionic liquid 1-butyl-3-ethylimidazolium bromide, [beim]Br, with water and ethanol, were measured at atmospheric pressure and at temperatures from 293.15 K to 323.15 K over the whole composition range (for a binary mixture of ionic liquids and water:  $0.01301 \leq m[\text{beim}]Br \leq 0.03900$  mol·kg<sup>-1</sup>; for a binary mixture of ionic liquids and ethanol:  $0.00827 \leq m[\text{beim}]Br \leq 0.04083$  mol·kg<sup>-1</sup>). From the experimental data were calculated the apparent molar volumes, apparent molar volumes at infinite dilution, partial molar volumes, thermal expansion coefficients, in order to obtain information about interactions between the selected ionic liquids and commercial molecular solvents.



## FH P 2

**Uporedni pregled molekulskih deskriptora 3-(4-supstisuani benzil)-5-fenilhidantoina koji se koriste za predviđanje sličnosti sa lekom**

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Hidantoini (imidazolidin-2,4-dioni) spadaju u grupu jedinjenja koja ispoljavaju širok spektar biološke aktivnosti. Do sada su derivati hidantoina identifikovani kao antikonvulzivi, antiaritmici, antidiabetici i antitumor agensi. Poznato je da biološka aktivnost neke supstance u velikoj meri zavisi od njenih fizičkih, hemijskih i strukturnih osobina. U ovom radu određeni su hromatografski parametri lipofilnosti novosintetisanih 5-fenilhidantoina primenom podeone hromatografije na obrnutim fazama. Korišćena je komercijalna HPTLC nepokretna faza C-18 modifikovanog silika-gela i pokretna faza metanol-voda. Diskutovan je uticaj strukture hidantoina na hromatografske parametre lipofilnosti. Primenjeno je pravilo Lipinskog sa ciljem da se proverí da li ispitivani hidantoini imaju dobru (teorijsku) oralnu biodostupnost.

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**A comparative study of molecular descriptors for predicting drug-likeness of 3-(4-substituted benzyl)-5-phenylhydantoins**

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Hydantoins (imidazolidine-2,4-diones) are a group of compounds that exhibit a wide range of biological activities. Until today hydantoin derivatives have been identified as anticonvulsants, antiarrhythmics, antidiabetics, and antitumor agents. It is well known that the biological activity of the substance greatly depends on physical, chemical and structural properties. In this paper the chromatographic lipophilicity parameters of the new synthesized 5-phenylhydantoins were determined using reversed-phase partition chromatography. Chromatography was performed on a commercially available HPTLC octadecyl silica stationary phase and aqueous mobile phase methanol-water. The relationship between structure and lipophilicity was discussed. Lipinski rule of five was applied in order to verify whether the investigated hydantoins exhibit good (theoretical) oral bioavailability.

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### **Intrerakcije smeša jonske tečnosti na bazi taurata i vode**

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U ovom radu su proučavane binarne smeše, novosintetisane jonske tečnosti, 1-butil-3-metilimidazolijum taurata ([bmim][Tau]) i vode. Usled nedostatka informacija o fizičko-hemijskim svojstvima jonskih tečnosti na bazi taurata, detaljno su ispitana termodinamička i transportna svojstva, kao i interakcije katjona sa tauratom i interakcije između katjona i anjona sa vodom. Rezultati i zaključci su izvedeni iz eksperimentalnih podataka (volumetrijska i transportna ispitivanja) i korišćenjem računarskih simulacija (molekulska dinamika). Dobijeni podaci daju značajne informacije o „structure making“ osobinama jonskih tečnosti na bazi taurata i o interakcijama između [bmim][Tau] i vode.

### **Interactions of binary mixtures of taurate based ionic liquids and water**

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In this work, binary mixtures of newly synthesized 1-butyl-3-methylimidazolium taurate, [bmim][Tau], ionic liquid and water was studied. Due to the lack of information about the physical-chemical properties of taurate ILs, interactions of cation with taurate and interaction of both cation and taurate with water molecules, a detailed investigation of thermodynamic and transport properties of this mixtures was performed. The results and conclusions were conducted applying both experimental (volumetric and transport properties) and computational approach (molecular dynamics). The obtained data provided significant information about structure making properties of taurate based ionic liquid and interactions of [bmim][Tau] and water.

FHP 4

**Fotokatalitička aktivnost TiO<sub>2</sub>/polianilin nanokompozitnih katalizatora primenom različitih izvora zračenja**

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Ispitana je fotokatalitička aktivnost nanočestičnog čistog TiO<sub>2</sub>, odnosno kompozita TiO<sub>2</sub>/polianilin u različitim molskim odnosima (TP-50, TP-100 i TP-150) u slučaju razgradnje herbicida klomazona i kvinmeraka primenom simuliranog sunčevog zračenja. Isto tako je ispitana i fotokatalitička efikasnost pomenutih katalizatora primenom UVA zračenja u slučaju razgradnje klomazona. Na osnovu dobijenih rezultata može se zaključiti, da se pod dejstvom simuliranog sunčevog, kao i UVA zračenja kao najefikasniji pokazao TP-150. U cilju praćenja efikasnosti razgradnje polaznog jedinjenja primenjena je HPLC-DAD tehnika, a za određivanje stepena mineralizacije odabrana je TOC tehnika. Dobijeni rezultati pokazuju da je mineralizacija u prisustvu svih navedenih katalizatora sporija u poređenju sa brzinom razgradnje polaznih jedinjenja.

**Photocatalytic activity of TiO<sub>2</sub>/polyaniline nanocomposite powders using different sources of radiation**

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Photocatalytic activities of the bare TiO<sub>2</sub>, and TiO<sub>2</sub>/polyaniline nanocomposite powders synthesized in different mole ratios (TP-50, TP-100, and TP-150) were investigated in the case of degradation of the herbicides quinmerac and clomazone using simulated solar radiation. Also, the photocatalytic efficiency of the mentioned catalysts using UVA radiation in the case of degradation of clomazone was studied. On the basis of the obtained results it can be concluded that under the simulated solar radiation, as well as UVA light the highest photocatalytic activity had TP-150. In order to check the efficiency of degradation of the parent compound, HPLC-DAD measurements were carried out, while for determination of the degree of mineralization TOC technique was applied. The obtained results show that the mineralization in the presence of all the mentioned catalysts was slower compared to the rate of degradation of the parent compounds.

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## **Proučavanje solvatohromnih osobina novosintetisanih derivata 2-piridona**

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2-Piridoni čine deo strukture mnogih prirodnih jedinjenja, od kojih neka pokazuju značajnu fiziološku i farmakološku aktivnost. Ovim jedinjenjima pripadaju i 4,6-disupstituisani-3-cijano-2-piridoni sa izrazito kardiotoničnim, antiinflamatornim, antidijabetskim i analgetičnim svojstvima. Istraživanja su pokazala da aktivnost ovih jedinjenja zavisi od njihove strukture i elektronskih efekata prisutnih supstituenata na prstenu 2-piridona. U ovom radu ispitivan je uticaj rastvarača i supstituenata na apsorpcione spektre novosintetisanih 2-piridona. Snimljeni su spektri 15 novosintetisanih derivata 3-cijano-4(fenilsubstituisani)-6-fenil-2-piridona u 10 rastvarača, različitih osobina. Apsorpcioni spektri ispitivanih jedinjenja su snimljeni u oblasti od 200 – 450 nm. Solvatohromizam ispitivanih derivata tumačen je primenom metode višestruke linearne korelacije slobodnih energija. Primenom Hammett-ove jednačine analiziran je uticaj supstituenta na spektralno ponašanje ispitivanih derivata. Dobijeni rezultati daju podatke o vrsti i dominantnosti interakcija između ispitivanih derivata i okolnog medijuma, koje imaju značajnu ulogu za njihovu dalju primenu.

## **Investigation of solvatochromic properties of newly synthesized 2-pyridone derivatives**

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2-Pyridone are structural parts of many natural compounds, and some of them show significant physiological and pharmacological activity. Among these compounds there are also 4,6-disubstitued-3-cyano-2-pyridone which have highly cardiotonic, antiinflammatory, antidiabetic and analgetic properties. Studies have showed that the activities of these compounds depend on their structure and electronic effects of substituents present on the 2-pyridone ring. The purpose of this work was to show dependence of absorption spectra of newly synthesized 2-pyridones on solvents and structure. Spectra of 15 newly synthesized 3-cyano-4(phenyl-substituted)-6-phenyl-2-pyridone derivatives were recorded in 10 solvents with different characteristics. Absorption spectra of the tested compounds were recorded in the range of 200-450 nm. Solvatochromism of the investigated compounds was characterized by applying the linear solvation energy relationships (LSER) principles. The effect of substituents on the spectral behavior of the investigated derivatives was analyzed by using the Hammett equation. The obtained results provide information on the type and dominance of interactions between investigated derivatives and the surrounding medium, which have an important role in their further application.

FH P 6

**Optimizacija i karakterizacija smeše jonske tečnosti i  $\gamma$ -butirolaktona kao elektrolita za litijum-jonske baterije**

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Volumetrijska i transportna svojstva binarne smeše jonske tečnosti 1-butil-1-metilpirolidinium dicianamida i  $\gamma$ -butirolaktona su prikazane na različitim temperaturama i atmosferskom pritisku u celom opsegu molskih udela. Dobijeni rezultati su upoređeni sa literaturnim podacima za binarni sistem 1-butil-1-metilpirolidinium bis(trifluorometil-sulfonil)imid +  $\gamma$ -butirolakton. Negativne vrednosti dodatnih molarnih zapremina ukazuju na solvataciju jonske tečnosti molekulima GBL zbog jakih jon-dipol interakcija pri čemu dolazi do slabljenja jon-jon interakcija između [bmpyrr]<sup>+</sup> i [DCA]<sup>-</sup>. Negativne vrednosti devijacije viskoznosti su verovatno posledica nastajanja heteronuklearnih kompleksa formiranjem intermolekulske vodonične veze između atoma vodonika na C-2 atomu GBL-a i atoma azota anjona jonske tečnosti, što je u skladu sa izračunatim volumetrijskim podacima. Poređenjem eksperimentalnih podataka električne provodljivosti opažaju se više vrednosti u sistemu [bmpyrr][DCA] + GBL nego u sistemu [bmpyrr][NTf<sub>2</sub>] + GBL, posebno pri niskim koncentracijama jonske tečnosti što ukazuje na izraženije razdvajanje jona u slučaju smeše [bmpyrr][DCA] + GBL

**Optimization and characterization of ionic liquid and  $\gamma$ -butyrolactone mixture based electrolyte for Li-ion battery application**

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Volumetric and transport properties of binary liquid mixtures of 1-butyl-1-methylpyrrolydinium dicyanamide ionic liquid with  $\gamma$ -butyrolactone are presented at various temperatures at atmospheric pressure in the whole composition range and compared with literature values for 1-butyl-1-methylpyrrolydinium dicyanamide +  $\gamma$ -butyrolactone binary system. Negative values of excess molar volumes indicate solvation of IL by GBL molecules due to strong ion-dipol interactions which results in weakening ion-ion interactions between [bmpyrr]<sup>+</sup> and [DCA]<sup>-</sup>. Negative viscosity deviations values are probably the consequence of formation of heteronuclear complexes due to intermolecular hydrogen bonding between hydrogen atom in the position C-2 of GBL and nitrogen atom of DCA, which is in accordance with calculated volumetric data. From electrical conductivity data it can be noticed that molar conductivities are much higher for [bmpyrr][DCA] + GBL binary mixtures compared to [bmpyrr][NTf<sub>2</sub>] + GBL system, especially in the IL low concentration range, indicating better separation of the ions in case of [bmpyrr][DCA] + GBL mixtures comparing to those with [bmpyrr][NTf<sub>2</sub>].

FH P 7

## Uticaj dužine bočnog niza jonskih tečnosti na fizičko-hemijske osobine binarnih smeša jonskih tečnosti sa $\gamma$ -butirolaktonom

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Budući da je limitirajući faktor za praktičnu primenu jonskih tečnosti njihova velika viskoznost, binarne smeše jonskih tečnosti sa odabranim laktonom,  $\gamma$ -butirolaktonom, bile su istražene u cilju prevazilaženja visoke viskoznosti. Volumetrijske i transportne osobine binarnih smeša 1-butil-3-metilimidazolijum *bis*(trifluorometilsulfonil)imida, [bmim][NTf<sub>2</sub>], sa  $\gamma$ -butirolaktonom (GBL), 1-metil-3-propilimidazolijum [NTf<sub>2</sub>] sa GBL i 1-etil-3-metilimidazolijum [NTf<sub>2</sub>] sa GBL, merene su u intervalu  $T = 293,15 - 323,15$  K na atmosferskom pritisku i u celom opsegu koncentracija. Iz eksperimentalnih podataka izračunate su dodatne molarne zapremine koje su fitovane Redlich-Kisterovom polinomnom jednačinom. Na osnovu rezultata dobijenih merenjem viskoznosti, računat je *Angell strength* parametar koji ukazuje na to da su ispitivane jonske tečnosti "lomljive", a na osnovu konduktometrijskih merenja izračunat je *Walden*-ov proizvod. Ispitivan je uticaj bočnog niza odabranih jonskih tečnosti na binarne smeše sa GBL u cilju pronalaženja najpogodnijeg medijuma za buduću primenu elektrolita u litijum-jonskim baterijama.

## Influence of the ionic liquids side chain length on physico-chemical properties of binary mixtures of ionic liquids with $\gamma$ -butyrolactone

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Since the limiting factor for the ionic liquids application is their high viscosity, binary liquid mixtures of ionic liquids with selected lactone,  $\gamma$ -butyrolactone, were investigated in order to overcome high viscosity. Volumetric and transport properties of binary liquid mixtures of 1-butyl-3-methylimidazolium *bis*(trifluoromethylsulfonyl)imide, [bmim][NTf<sub>2</sub>] with  $\gamma$ -butyrolactone (GBL), 1-methyl-3-propylimidazolium [NTf<sub>2</sub>] with GBL, and 1-ethyl-3-methylimidazolium [NTf<sub>2</sub>] with GBL, were measured in the interval  $T = 293.15 - 323.15$  K and under atmospheric pressure over the whole composition range. Excess molar volumes have been calculated from the experimental densities and fitted with the Redlich-Kister polynomial equation. From the viscosity results, the *Angell strength* parameter was calculated, indicating that investigated ionic liquids are "fragile" liquids and from conductivity results *Walden* plot is presented. The effect of side chain selected ionic liquids was investigated in binary mixture with GBL in order to find the most appropriate medium for future application as electrolyte for lithium-ion batteries.

FH P 8

### Uticaj odabranih amida na proces adsorpcije *p*-nitrofenola na aktivnom uglju

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Ispitivan je mehanizam adsorpcije *p*-nitrofenola na aktivnom uglju NORIT SA2 u prisustvu različitih amida kroz kinetičke i termodinamičke eksperimente. Određeni su koeficijent površinske difuzije ( $D_s$ ) i koeficijent difuzije u graničnom sloju ( $k_f$ ). Takođe, određeni su termodinamički parametri za interakcije adsorbat-adsorbent. Uticaj amida na adsorpciju *p*-nitrofenola razmatran je kroz uočene razlike u difuzionim i termodinamičkim parametrima.

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### Influence of selected amides on adsorption process of *p*-nitrophenol on activated carbon

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We report the investigation on mechanism of adsorption of *p*-nitrophenol on activated carbon NORIT SA2 in the presence of different amides in kinetic and thermodynamic experiments. Coefficients of surface diffusion ( $D_s$ ) and of film diffusion ( $k_f$ ) have been determined. Furthermore, thermodynamic parameters for adsorbate-adsorbent interactions were estimated. Influence of amides on adsorption of *p*-nitrophenol has been considered in view of the observed differences in diffusion and thermodynamic parameters.

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FH P 9

## **Fotokatalitička razgradnja triarilmetanske boje na zlatom modifikovanom cink oksidu**

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Fotokatalitička efikasnost poluprovodničkih nanočestica je često ograničena brzom rekombinacijom fotogenerisanih nosilaca naelektrisanja. Jedan od najčešće korišćenih načina za prevazilaženje ovog problema je depozicija plemenitih metala na površinu poluprovodnika, koja sprečava rekombinaciju parova elektron-šupljina i shodno tome povećava fotokatalitičku aktivnost. U ovom radu su različiti nedomodifikovani i zlatom modifikovani poluprovodnički fotokatalizatori bili primenjeni za potpuno obezbojavanje nepufisanog rastvora kristal violeta. Fotokatalitička efikasnost komercijalno dostupnog ZnO i TiO<sub>2</sub> (nanoprah anataze) je bila upoređena sa aktivnošću novopripremljenih zlatom modifikovanih čestica ZnO (Au/ZnO). Au/ZnO su napravljene od čistog ZnO praha pomoću depozicije zlata naparavanjem. Morfološka karakterizacija Au/ZnO je izvedena pomoću skenirajuće elektronske mikroskopije. Katalizatori na bazi ZnO su pokazali znatno veću aktivnost nego TiO<sub>2</sub>. Au/ZnO su pokazali znatno veću aktivnost nego nedomodifikovani ZnO.

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## **Photocatalytic degradation of a triarylmethane dye on gold-modified zinc oxide**

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The photocatalytic efficiency of semiconductor nanoparticles is often limited by a quick recombination of the photogenerated charge carriers. One of the mostly used ways to overcome this problem is deposition of noble metals on the semiconductor surface, which prevents recombination of the electron-hole pairs and consequently increases the photocatalytic activity. In this work different unmodified and gold-modified semiconductor photocatalysts were used for the complete decolorization of an unbuffered crystal violet solution. The photocatalytic efficiency of commercially available ZnO and TiO<sub>2</sub> (anatase nanopowder) was compared with that of newly prepared gold-modified ZnO (Au/ZnO). The Au/ZnO particles were prepared from pure ZnO powder through deposition of gold by sputtering. The morphological characterization of the Au/ZnO was done with the aid of scanning electron microscopy. ZnO-based catalysts show significantly higher decolorization power in comparison with TiO<sub>2</sub>. Au/ZnO shows even higher activity than unmodified ZnO.

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**Elektrohemija / Electrochemistry**

EH O 1

**Electrochemical determination and degradation of tebuconazole on gold electrode followed by GC-MS analysis**

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Tebuconazole, pesticide which presents the risk for ecosystems, groundwater and human health, was quantitatively determined at gold electrode in 0.05 M NaHCO<sub>3</sub> using its electrochemical activity by CV and SWV. The linear relationships (currents vs. concentrations) in the range: (0.076, 0.19, 0.38, 0.57, 0.76 μmol dm<sup>-3</sup>) were obtained. The gold electrode is highly sensitive for tebuconazole molecule giving values of LOD and LOQ of 0.045 and 0.15 μmol dm<sup>-3</sup>, respectively. The degradation of pesticide was performed by long term cycling and followed by GC-MS analysis. The possible mechanism of tebuconazole electrochemical degradation is presented.

**Elektrohemijsko određivanje i degradacija tebukonazola na elektrodi od zlata praćena GC-MS analizom**

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Tebukonazol, pesticid koji predstavlja rizik za ekosistem, podzemne vode i ljudsko zdravlje, kvantitativno je određen na elektrodi od zlata u rastvoru bikarbonata metodom ciklične i pulsne voltametrije. Ispitivana je koncentraciona zavisnost ovog pesticida i za pet različitih koncentracija, od 0.076 μmol dm<sup>-3</sup> do 0.76 μmol dm<sup>-3</sup>, dobija se pravolinijska zavisnost, ukazujući na značajnu osetljivost elektrode od zlata. Određene su granica detekcije (LOD) i granica kvantifikacije (LOQ) koje iznose 0.045 i 0.15 μmol dm<sup>-3</sup>, redom. Degradacija ovog pesticida ostvarena je dužim cikliziranjem, a da bi se utvrdili proizvodi degradacije tebukonazola korišćena je gasno masena hromatografija (GC-MS). Na osnovu određenih proizvoda razgradnje tebukonazola predložen je mehanizam elektrohemijske degradacije.





EN P 1

**Псеудокапацитивне карактеристике нанокристалиничног оксида рутенијума неуобичајене структуре добијене хидротермалном синтезом у микроталасном реактору**

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Једноставна синтеза хидратисаног нанокристалиничног оксида рутенијума неуобичајене морфологије и структуре изведена је под контролисаним режимом температуре и притиска у микроталасном реактору. Изузетно висока псеудокапацитивност преко 800 F/g, регистрована за узорак синтетисан на 200 °С, показује незнатну зависност од брзине пуњења/пражњења. Синтетисан оксид тежи да формира високо уређене ромбоидне и призматичне облике, који скоро потпуно излажу активну површину електролиту при високим, као и при ниским снагама капацитивног пражњења. Карактеристике синтетисаног оксида последица су примењених услова микроталасне синтезе.

**Pseudocapacitive characteristics of the unusually-shaped nano-crystalline ruthenium oxide prepared by hydrothermal synthesis in a microwave reactor**

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One-pot and simple synthesis of hydrated nano-crystalline ruthenium oxide of an unusual morphology and structure is performed under controllable temperature–pressure regime in a microwave reactor. The extremely high pseudocapacitance of above 800 F/g is registered for the sample synthesized at 200 °C, showing negligible dependence on charging/discharging rate. The synthesized oxide tends to form highly ordered rhomboidal and prismatic shapes, able to expose almost complete active surface area in high- and low-power capacitive demands. The characteristics of the synthesized oxide appear intrinsic to applied microwave synthesis conditions.

EH P 2

**Upotreba nemodifikovane borom dopovane dijamantske elektrode za analizu vitamina B<sub>12</sub>**

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Elektrohemijsko ponašanje i određivanje vitamina B<sub>12</sub> je predloženo korišćenjem nemodifikovane borom dopovane dijamantske (BDD) elektrode. Ciklična voltometrija i polarografija sa pravougaonim talasima su korišćene za ispitivanje karakteristika dijamantske elektrode. Kalibraciona kriva, konstruisana kao zavisnost struje oksidacionog pika, u Britton-Robinson-ovom puferu na pH=2 je linearna u širokom opsegu koncentracija od 2 do 35  $\mu\text{M}$  sa detekcionim limitom 0,7  $\mu\text{M}$ . Uticaj najčešćih interferirajućih supstanci je ispitan i predložena metoda je uspešno primenjena za kvantifikaciju vitamina B<sub>12</sub> u analizi realnih uzoraka sa zadovoljavajućim rikaverijem (98-104,8%)

**Unmodified boron-doped diamond electrode for Vitamin B<sub>12</sub> analysis**

Darko Kuzmanović, Vesna Vukojević, Slađana Đurđić, Dalibor M. Stanković,  
Ivan Anđelković, Dragan Manojlović, Goran Roglić  
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Electrochemical behavior and sensitive determination of vitamin B<sub>12</sub> was proposed by using unmodified boron doped diamond (BDD) electrode. Cyclic voltammetry and square wave voltammetry were used to investigate the performance of BDD electrode. The calibration curve, constructed by plotting oxidation peak current, obtained in Britton-Robinson buffer solution at pH 2 was linear in wide concentration range of 2-35  $\mu\text{M}$  with detection limit of 0.7  $\mu\text{M}$ . Influence of the most common interfering compounds was investigated and proposed procedure was successfully applied for quantification of vitamin B<sub>12</sub> in real sample analysis with satisfactory recovery (98-104.8 %).

EH P 3

### **Brza elektrohemijska metoda za određivanje L-DOPA u ekstraktu ulja iz *Mucuna prurita***

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Ispitano je elektrohemijsko ponašanje L-DOPA primenom ciklične voltametrije u Britton – Robinson-ovom puferu. Kao elektroda korišćena je borom dopovana dijamantska (BDD) elektroda. Takođe je prikazana primena pomenute elektrode za određivanje L-DOPA u ekstraktu ulja iz semenki biljke *Mucuna prurita*. Na potencijalu od +0,8V (u odnosu na Ag/AgCl referentnu elektrodu u puferskom rastvoru čija je pH=3) L-DOPA pokazuje dobro definisan i jasan pik oksidacije. Optimizovani su eksperimentalni parametri kao što su pH elektrolita, radni parametri za SWV (*square wave voltammetry*), frekvencija i amplituda pulsa. Uticaj mogućih smetnji na određivanje L-DOPA je takođe ispitan. Korišćenjem optimalnih parametara detekcioni limit predložene metode je 0,8  $\mu\text{M}$ , a kalibraciona kriva ima opseg linearnosti od 2 do 40  $\mu\text{M}$ . Metoda je uspešno primenjena za određivanje L-DOPA u ekstraktu ulja iz biljke *Mucuna prurita*. Dobijeni rezultati se dobro slažu sa standardnom ABTS metodom i onima ranije publikovanih u literaturi.

### **Rapid electrochemical method for determination of L-DOPA in oil extract from *Mucuna prurita***

Sladana Đurđić, Vesna Vukojević, Darko Kuzmanović, Sandra Škrivanj,  
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This work present electrochemical behavior of L-DOPA at boron-doped diamond electrode using cycling voltammetry in Britton-Robinson buffer solution and application of the proposed electrode for determination of L-DOPA in oil extract from the seeds of indigenous hypoglycaemic herb *Mucuna prurita*. L-DOPA provides well defined and single oval-shape oxidation peak at +0.8 V vs. Ag/AgCl reference electrode in buffer solution pH 3. Experimental parameters, such as pH of supporting electrolyte, square wave voltammetry operating parameters frequency and pulse amplitude, were optimized. Effects of possible interferences were evaluated. Under optimal conditions detection limit of proposed method was 0.8 $\mu\text{M}$  and calibration curve has linear range from 2 to 40  $\mu\text{M}$ . Proposed method was successfully applied for determination of L-DOPA in oil extract from *Mucuna prurita* herb. Obtained result was in good agreement with result obtained by ABTS method and those reported in the literature.

EH P 4

### **Glukoza-oksidaza imobilisana na modifikovanu elektrodu od zlata za detekciju glukoze**

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Imobilizacija redoks enzima, poput lakaza i glukoza-oksidaza, na površinu elektrode predstavlja veoma važnu oblast u razvoju senzora. Zbog velike specifičnosti prema supstratu koju enzimi poseduju i koja obezbeđuje maksimalnu osetljivost, enzimi imaju veliku ulogu kao bioreceptori.

U ovom radu glukoza-oksidaza (GOx) je uspešno imobilisana na prethodno modifikovanu površinu elektrode od zlata. Površina elektrode je najpre modifikovana pomoću cisteina uspostavljanjem Au-S veze, a zatim su pomoću glutaraldehida uvedene aldehidne grupe. Ovako aktivirana elektroda korišćena je kao nosač za imobilizaciju enzima uspostavljanjem kovalentne veze sa amino grupama sa površine enzima. Dobijeni biosenzor testiran pomoću ciklične voltametrije u 0,1 M fosfatnom puferu (pH 7) u odsustvu i prisustvu 5 mM glukoze pokazao je odličnu elektrokatalitičku aktivnost. Ciklična voltametrija je pokazala da je aktivan samo sistem koji sadrži GOx. Takođe, promena morfologije površine praćena je pomoću AFM-a pri svakom koraku modifikacije.

### **Glucose oxidase immobilized on modified gold electrode for use as a glucose biosensor**

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The functional immobilization of redox enzymes, such as laccase and glucose oxidase, onto electrode surface is very important in the area of sensors development. Due to very high specificity that leads to minimal risk of false positive response, enzymes play a great role as bioreceptor molecules.

In this work glucose oxidase (GOx) was successfully immobilized on previously modified gold electrode. First, the gold electrode surface was activated with cysteine by forming Au-S bond and thereafter with glutaraldehyde to introduce aldehyde groups. The prepared electrode was then used as a support for the immobilization of GOx by forming the Schiff base with the enzyme amino groups. Fabricated biosensor was tested using cyclic voltammetry in 0.1 M phosphate buffer solution (pH 7) and in the absence and presence of 5 mM glucose and exhibited excellent electrocatalytic activity. In order to verify if electrocatalytic oxidation of glucose originates from GOx, cyclic voltammetry was performed after each assembly step and they showed catalytically inactive. In addition surface morphology was monitored using AFM.

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### **Uticaj 5-hlor-1H-benzotriazola na koroziono ponašanje bakra u kiselom sulfatnom rastvoru**

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Ispitivan je uticaj 5-hlor-1H-benzotriazola na koroziono ponašanje bakra u 0,01 M H<sub>2</sub>SO<sub>4</sub>. U cilju istraživanja korišćene su potenciodinamička polarizaciona metoda i merenje potencijala otvorenog kola. Na osnovu dobijenih rezultata utvrđeno je da se 5-hlor-1H-benzotriazol ponaša kao anodni inhibitor. Takođe je uočeno da se inhibiciona efikasnost povećava sa povećanjem koncentracije ispitivanog jedinjenja. Adsorpcija inhibitora na površini bakra odvija se po Lengmirovoj adsorpcionoj izotermi.

### **Influence of 5-chloro-1H-benzotriazole on copper corrosion behavior in acidic sulfate solution**

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The influence of 5-chloro-1H-benzotriazole on copper corrosion behavior in 0.01 M H<sub>2</sub>SO<sub>4</sub> was investigated. The potentiodynamic measurements and the open circuit potential measurements were used. The obtained results indicated that 5-chloro-1H-benzotriazole act as anodic type of inhibitor. The inhibition efficiency is increased with increasing concentration of investigated compound. The adsorption of inhibitor molecules onto copper surface follows Langmuir adsorption isotherm.

### **Inhibicija korozije bakra u kiselom sulfatnom rastvoru primenom derivata tiazola**

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Ispitivana je koroziona otpornost bakra u kiselom rastvoru  $\text{Na}_2\text{SO}_4$  (pH 3) u odsustvu i prisustvu 5-(5'-metilfurfuriliden-2')-2,4-dioksotetrahydro-1,3-tiazola (MFDT) na četiri različite temperature: 15, 25, 35 i 45 °C. Potenciostatička polarizaciona merenja pokazala su da ispitivani derivat tiazola inhibira koroziju bakra pri datim uslovima sa tendencijom da deluje kao dominantno katodni tip inhibitora. Zaštita bakra ostvaruje se adsorpcijom molekula inhibitora na metalnoj površini, koja se odigrava prema modelu Bokris-Svinkels-ove izoterme ( $X = 1$ ). Izračunati kinetički i termodinamički parametri ukazuju na to da je dominantan mehanizam adsorpcije hemisorpcija.

### **Inhibition of copper corrosion in acidic sulphate solution by thiazole derivative**

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Copper corrosion resistance in acidic  $\text{Na}_2\text{SO}_4$  solution (pH 3) in the absence and in the presence of 5-(5'-methylfurfurylidene-2')-2,4-dioxotetrahydro-1,3-thiazole at four selected temperatures: 15, 25, 35 and 45 °C, was investigated. Potentiostatic polarization measurements showed that investigated thiazole derivative inhibit copper corrosion at given condition with tendency to act as predominant cathodic type inhibitor. Copper protection is achieved by adsorption of inhibitor molecules on metal surface which obeys Bockris-Svinkels isotherm ( $X = 1$ ). Obtained kinetic and thermodynamic parameters indicated that adsorption mechanism is predominant chemisorption.

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EHP 7

## Voltametrijska karakterizacija novih kompleksa Cr(III) sa semi- i tiosemi-karbazonom piridoksala

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Voltametrijski su okarakterisani DMF rastvori kompleksa Cr(III) sa semi-(PLSC) i tiosemi-(PLTSC) karbazonom piridoksala sledećih formula:  $[Cr(PLSC)(PLSC-H)](NO_3)_2 \cdot H_2O$ ,  $K[Cr(PLSC-H)(NCS)_3]$ ,  $[Cr(PLTSC)(PLTSC-H)](NO_3)_2 \cdot 2H_2O$ ,  $[Cr(PLTSC)(NCS)_3] \cdot 2H_2O$ . Na cikličnim voltamogramima na radnoj elektrodi od staklastog ugljenika registrovani su redoks procesi za Cr(III) komplekse i ligande. Pokazalo se da odgovarajući kompleksi podležu reakcijama ligandne izmene tipa  $NCS^-$  - DMF kako u rastvoru, tako i posle reakcije na elektrodi. Nestabilnost ispitivanih kompleksa u DMF, naročito bis(ligandnih), se odražava na voltamogramima u pojavi više pikova sa strujom niskog intenziteta. Svi kompleksi se redukuju u po dva jednoelektronska procesa centrirana na centralnom atomu, a registruju se i redoks procesi na koordinovanom ligandu. Utvrđene su karakteristike redoks procesa, reakciona sekvenca i za  $NCS^-$  komplekse predložen je mehanizam elektrodnih procesa.

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## Voltammetric characterization of new Cr(III) complexes with pyridoxal semi- and thiosemi- carbazone

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The new Cr(III) complexes with pyridoxal semi-(PLSC) and thiosemi-(PLTSC) carbazones of the following formulas:  $[Cr(PLSC)(PLSC-H)](NO_3)_2 \cdot H_2O$ ,  $K[Cr(PLSC-H)(NCS)_3]$ ,  $[Cr(PLTSC)(PLTSC-H)](NO_3)_2 \cdot 2H_2O$  and  $[Cr(PLTSC)(NCS)_3] \cdot 2H_2O$  were voltammetrically characterized in DMF. On the cyclic voltammograms at a glassy carbon electrode redox processes for complexes and ligands were recorded. It appeared that the corresponding complexes show the ligand exchange reactions  $NCS^-$  - DMF both in solution and following the electrode processes. The instabilities of these complexes, especially of the bis(ligand) ones, were observed through multiple peaks with low intensity currents. All the complexes reduce in two one-electron processes centered prevailingly at the central atom, while also the processes at the coordinated ligands are present. The characteristics of redox processes and reaction sequences are established and for the  $NCS^-$  complexes a possible reaction mechanism proposed.

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**EH P 8**

**In situ ECAFM proučavanje morfologije platinskih katalizatora**

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U ovom radu je ispitivana morfologija platine deponovane na elektrodu od staklastog ugljenika primenom in situ elektrohemijske mikroskopije atomskih sila (ECAFM). Platinski katalizatori su dobijeni elektrohemijskim i hemijskim taloženjem platine na podlogu od poliranog i elektrohemijski tretiranog staklastog ugljenika iz  $H_2SO_4 + H_2PtCl_6$  rastvora. Elektrokatalitička aktivnost tako dobijenih katalizatora je ispitivana za reakciju oksidacije metanola u  $H_2SO_4$  rastvoru. Dobijena aktivnost katalizatora je dalje proučavana sa aspekta površinske tj. strukturne osetljivosti reakcije oksidacije metanola kao jednog od potencijalnih goriva u gorivim ćelijama sa polimernom membranom.

**In situ ECAFM investigation of platinum catalyst morphology**

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In this work surface morphology of platinum deposited on glassy carbon electrode was investigated by in situ electrochemical atomic force microscopy (ECAFM) and correlated with electrochemical results. Platinum catalysts were prepared by potentiostatic and chemical deposition onto the polished and electrochemically treated glassy carbon support from  $H_2SO_4 + H_2PtCl_6$  solution. The catalyst electrocatalytic activity was studied for methanol oxidation reaction in  $H_2SO_4$  solution. The activity of the electrodes was investigated from the viewpoint of the sensitivity of methanol electro oxidation on the surface structure.



EHP 9

## Zaštita čelika u armiranom betonu primenom epoksidne prevlake na čeliku modifikovanom prevlakama legura Zn

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Čelik u armiranom betonu je podložan koroziji usled prodiranja ugljen-dioksida i hloridnih jona kroz pore betona do njegove površine. Najrašireniji postupak zaštite čelika u ovom okruženju su prevlake cinka, epoksidna prevlaka, ili njihova kombinacija. Nedostatak primene epoksidne prevlake na čeliku je slaba adhezija prevlake i njeno odvajanje, kako od čelika tako i od betona (katodno odvajanje).

Cilj ovog rada je ispitivanje modifikacije površine čelika prevlakama legura Zn (Zn-Ni i Zn-Fe) u cilju poboljšanja adhezije epoksidne prevlake prilikom primene ovakvog zaštitnog sistema u betonu. Prevlake Zn-Ni i Zn-Fe legure sa raziličitim sadržajem legirajućeg elementa su galvanostatski taložene na čeliku gustinama struje 20 – 70 mA cm<sup>-2</sup> (debljina 15 μm) i one su bile supstrat za nanošenje epoksidne prevlake. Koroziona otpornost zaštitnih sistema je ispitivana spektroskopijom elektrohemijske impedancije u rastvoru koji simulira elektrolit u porama betona (0,3 mol dm<sup>-3</sup> NaHCO<sub>3</sub> + 0,1 mol dm<sup>-3</sup> Na<sub>2</sub>CO<sub>3</sub> + 0,5 mol dm<sup>-3</sup> NaCl). Ispitivana je i adhezija epoksidne prevlake i sklonost prevlake prema katodnom odvajanju. Tokom početnog delovanja agensa korozije najveću stabilnost je pokazao zaštitni sistem na bazi Zn-Ni legure dobijene taloženjem sa 30 mA cm<sup>-2</sup>.

## Corrosion protection of reinforcing bars by epoxy coating on steel modified by Zn alloy coatings

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Reinforcing steel in concrete corrodes due to the migration of carbon dioxide and chloride ions through the concrete pores. Widely accepted protection methods for steel in such environment are coatings, such as galvanized zinc, epoxy coating, or their combination. The drawback of epoxy coating on steel is a weak wet-adhesion, both to the substrate as well as coated rebar and concrete (cathodic disbondment).

In this work the steel surface modification with Zn alloy coatings (Zn-Ni and Zn-Fe) was examined with the aim of enhancing the adhesion of top epoxy coating and decreasing its susceptibility to cathodic disbondment. The 15 μm thick Zn-Ni and Zn-Fe alloy coatings, with different amounts of alloying element, were obtained by galvanostatic electrodeposition at current densities of 20 – 70 mA cm<sup>-2</sup> and used as a substrate for the top epoxy coating. The corrosion behavior of the samples was examined by electrochemical impedance spectroscopy in the simulated concrete pore solution (0,3 mol dm<sup>-3</sup> NaHCO<sub>3</sub> + 0,1 mol dm<sup>-3</sup> Na<sub>2</sub>CO<sub>3</sub> + 0,5 mol dm<sup>-3</sup> NaCl). The adhesion of epoxy coating to the substrate and its susceptibility to cathodic disbondment was also examined. It was shown that epoxy coating on steel modified with Zn-Ni alloy coating deposited at 30 mA cm<sup>-2</sup> showed the highest corrosion stability during early corrosion testing.

EH P 10

**Priroda izuzetne aktivnosti Pt<sub>3</sub>Ni(111) katalizatora u reakciji CO oksidacije**

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Oksidacija CO je ispitivana na Pt(111) monokristalu i Pt<sub>3</sub>Ni(111) monokristalnoj leguri primenom elektrokemijske (EK), skenirajuće tunelirajuće mikroskopije (STM) i infracrvene adsorpcione spektroskopije (IRAS). CO polarizacione krive su pokazale da je Pt<sub>3</sub>Ni(111) mnogo aktivniji katalizator u odnosu na Pt (111). Rezultati dobijeni korišćenjem IRAS i STM tehnika su pokazali da za visoku aktivnost Pt<sub>3</sub>Ni(111) katalizatora za oksidaciju CO nije odgovoran elektronski efekat izazvan prisustvom atoma Ni u drugom sloju već površinska morfologija. Površinska ostrva, sastavljena od niskokoordinisanih Pt atoma, koja pokrivaju površinu Pt na oba katalizatora su detektovana kao aktivna mesta za oksidaciju CO u predoksidacionoj oblasti. Superiornija aktivnost Pt<sub>3</sub>Ni(111) u odnosu na Pt(111) katalizator je posledica većeg broja površinskih ostrva.

**The origin of exceptional activity of Pt<sub>3</sub>Ni(111) catalyst in CO oxidation reaction**

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Bulk CO oxidation was studied at a Pt(111) single crystal and a Pt<sub>3</sub>Ni(111) single crystal alloy using electrochemical (EC), scanning tunneling microscopy (STM) and infrared adsorption spectroscopy (IRAS) techniques. CO polarization curves showed that Pt<sub>3</sub>Ni(111) is much more active compared to Pt(111). The results obtained by IRAS and STM pointed out that the high activity for CO oxidation of Pt<sub>3</sub>Ni(111) is not caused by an electronic effect induced by a Ni containing subsurface layer but by the surface morphology. Adislands, composed of highly undercoordinated Pt atoms, covering the Pt surface at both catalysts are detected and assigned as the active sites for CO oxidation in the preignition potential region. The enhanced activity of Pt<sub>3</sub>Ni(111) compared to Pt(111) is related to a larger number of adislands on Pt skin surface.

EHP 11

### **Elektrohemijska karakterizacija Pt/C katalizatora sintetizovanih mikroemulzionom metodom pomoću oksidacije adsorbovanog CO**

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Pt katalizatori na ugljeničnom nosaču sintetizovani su mikroemulzionom metodom, gde je vodena faza dispergovana u uljanoj, pri čemu je korišćen polietilenglikol-dodeciletar (BRIJ30) kao površinski aktivna supstanca. Ova metoda je korišćena u cilju poboljšanja površinskih karakteristika katalizatora potrebnih za reakcije u gorivnim spregovima. Površina katalizatora ispitivana je elektrohemijski, cikličnom voltametrijom u osnovnom elektrolitu (0.5 M H<sub>2</sub>SO<sub>4</sub>) i oksidacijom adsorbovanog CO. Elektrohemijski aktivne površine izračunate su integracijom krivih za oksidaciju CO, uzimajući da 420  $\mu\text{C cm}^{-2}$  odgovara adsorbovanom monosloju CO. Daljim izračunavanjem dobijene su vrednosti veličine Pt čestica. Oksidacija adsorbovanog CO koristi se kao „otisak prsta“ morfologije površine katalizatora, a korišćena je ne samo u cilju izračunavanja elektrohemijski aktivne površine katalizatora, već i za ispitivanje tolerancije sintetizovanih katalizatora prema CO.

### **CO stripping surface characterisation of Pt/C catalysts prepared by water in oil microemulsion method**

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Carbon supported Pt catalysts were prepared by water in oil microemulsion method, with polyethyleneglycol-dodecylether (BRIJ30) as capping agent. This method was used in order to improve Pt surface characteristics required for fuel cell reactions. Catalyst surface was investigated by electrochemical characterisation methods, such as cyclic voltammetry in the supporting electrolyte (0.5 M H<sub>2</sub>SO<sub>4</sub>) and CO stripping voltammetry. Electrochemically active surface areas (EASA) were calculated from the integration of CO oxidation curves, assuming that 420  $\mu\text{C cm}^{-2}$  corresponds to adsorbed CO monolayer. Further calculations determined the size of Pt particle diameters. CO stripping voltammetry can be used as a fingerprint of catalyst surface morphology, and was employed not only to determine EASA but also to study the CO tolerance of the investigated catalysts.

**EH P 12**

**Uticaj termičkog tretiranja na morfologiju platinskih katalizatora**

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U ovom radu je ispitivana promena morfologije površine platine deponovane na elektrodu od staklastog ugljenika ili Au pod uticajem termičkog tretiranja na 400° C u inertoj, oksidujućoj ili redukujućoj atmosferi. Promena morfologije površine praćena je mikroskopijom atomskih sila (AFM) cikličnom voltametrijom i oksidacijom adsorbovanog CO. Platina je na podlogu nanošena elektrohemijskim taloženjem iz rastvora H<sub>2</sub>SO<sub>4</sub> + H<sub>2</sub>PtCl<sub>6</sub>. Pored promene morfologije ispitivana je i promena katalitičke aktivnosti dobijenih površina za reakciju oksidacije mravlje kiseline. Dobijena aktivnost katalizatora je dalje proučavana sa aspekta strukturne osetljivosti reakcije.

**Influence of thermal treatment on morphology of platinum catalyst**

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Surface morphology changes of platinum deposited on glassy carbon and Au electrodes upon thermal treatment at 400° C in inert, oxidizing and reducing atmosphere were investigated in this work. Morphology changes were followed by atomic force microscopy (AFM), cyclic voltammetry and CO stripping polarization curves. Platinum catalysts were prepared by potentiostatic deposition from H<sub>2</sub>SO<sub>4</sub> + H<sub>2</sub>PtCl<sub>6</sub> solution. The electrocatalytic activity of the catalysts obtained was also studied for the oxidation of formic acid. The activity of the electrodes was investigated from the viewpoint of the surface structure.

## Hemijsko inženjerstvo / Chemical Engineering

HI O 1



### **Separation of heptane + methanol azeotrope using choline chloride + dl-malic acid deep eutectic solvent**

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With rapid industrial development existing processes need to enable high efficiency with a special emphasis on environmental protection. In the field of green chemistry non-toxic organic solvents have attracted major attention. Deep eutectic solvents (DESs), as a new generation of ionic liquids, exhibit exceptional characteristics such as low toxicity and volatility, biodegradability and they are made from naturally occurring substances. In this work extraction efficiency of choline chloride + dl-malic acid deep eutectic solvent (molar ratio 1:1) was examined for separation of heptane + methanol azeotrope through liquid-liquid extraction. Ternary liquid-liquid equilibrium experiments were performed at 298.15 K and at atmospheric pressure. Determined selectivity and distribution ratio values prove promising extraction ability of the investigated deep eutectic solvent comparing to some ionic liquids and conventional solvents.

### **Razdvajanje azeotropne smeše heptan + metanol korišćenjem eutektičke smeše holin hlorida i dl-jabučne kiseline kao rastvarača**

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Nagli industrijski razvoj zahteva primenu procesa visoke efikasnosti uz sprečavanje zagađenja životne sredine. U oblasti zelene hemije netoksični organski rastvarači privlače veliku pažnju. Eutektičke smeše kao nova generacija jonskih tečnosti koje se dobijaju iz prirodnih izvora pokazale su izuzetna svojstva kao što su niska toksičnost, isparljivost i biorazgradivost. U ovom radu ispitivana je ekstrakciona efikasnost eutektičke smeše holin hlorida i dl-jabučne kiseline u molarnom odnosu 1:1 za razdvajanje azeotropne smeše heptan + metanol primenom ekstrakcije tečno-tečno. Ternarni tečno-tečno ravnotežni eksperimenti su sprovedeni na 298,15 K i na atmosferskom pritisku. Iz eksperimentalnih podataka određeni su selektivnost i distribicioni koeficijent koji ukazuju na odličnu ekstrakcionu efikasnost ispitivane eutektičke smeše u poređenju sa konvencionalni ekstrakcionim sredstvima i nekim ranije ispitivanim jonskim tečnostima.

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HI P 1

### Višestruke V/U/V emulzije sa enkapsuliranim ekstraktom belog luka

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Cilj ovog rada je inkorporiranje ekstrakta belog luka u unutrašnju vodenu fazu višestrukih voda-u-ulju-u-vodi (V/U/V) emulzija. Za dobijanje stabilnih primarnih V/U emulzija kao najbolji emulgator izabran je poliglicerol poliricinoleat (PGPR). U prisustvu soli (NaCl) koja ima ulogu kostabilizatora, PGPR omogućava dobijanje emulzija sa kapima vode nanometarskih veličina. Kao uljana faza korišćena je smeša tikvinog i suncokretovog ulja u odnosu 1:1. Prečnik kapi emulzija sa inkorporiranim etanolnim ili vodenim ekstraktom belog luka poređen je sa veličinom kapi primarnih V/U emulzije koje ne sadrže ekstrakt, odnosno vodena faza je zapravo 0.15M rastvor NaCl. Prečnik kapi svih pripremljenih primarnih emulzija bio je manji od 200 nm, sa uskom raspodelom veličina formiranih kapi.

Dvosruke V/U/V emulzije sa inkorporiranim ekstraktom belog luka u unutrašnjoj vodenoj fazi pripremane su: upotrebom homogenizera i membranskim emulgovanjem (ME) u ćeliji sa mešanjem. Kao hidrofilni emulgator korišćen je Tween 20. Veličina kapi duplih emulzija dobijenih na homogenizeru bila je manja od kapi koje su dobijene ME (na  $\Delta P_{tm} = 30$  kPa i  $N = 1000$  rpm), pri čemu je odgovarajući prečnik bio  $D_{4,3} = 46.44 \pm 3.9$   $\mu\text{m}$  u poređenju sa  $D_{4,3} = 54.77 \pm 0.04$   $\mu\text{m}$  koji je dobijen ME. Međutim, uniformnost kapljica prilično je poboljšana korišćenjem ME, pri čemu emulzija dobijena u ćeliji sa mešanjem ima vrednost *span*-a 0,65, dok emulzija dobijena homogenizacijom ima *span* 1,08 (za isti sadržaj disperzne faze od 0 % (v/v)).

### Double W/O/W emulsions with encapsulated garlic extract

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The aim of this work was preparation of emulsions with incorporated garlic extract into internal water phase of water-in-oil-in-water (W/O/W) emulsions. Polyglycerol polyricinoleate was chosen as the best emulsifier for preparation of stable W/O emulsions which together with the sodium chloride (has a role of co-stabiliser) give the possibility of production stable nanoemulsions. As an oil phase mixture of pumpkin seed and sunflower oil (mass ratio 1:1) was used. Additionally, comparison between droplet diameter of primary W/O emulsions (without incorporated garlic extract) and droplet diameter of W/O emulsions containing ethanol or aqueous garlic extract were made. The droplet diameter of all prepared emulsions was smaller than 200 nm and emulsions were quite monodisperse.

The double W/O/W emulsions containing garlic extract in internal water phase were prepared by two techniques: using high-speed homogenizer and stirred cell membrane emulsification (ME), with the Tween 20 as a hydrophilic emulsifier. Droplet size of double emulsions obtained by homogenisation was lower than  $D_{4,3}$  obtained by ME ( $\Delta P_{tm} = 30$  kPa and  $N = 1000$  rpm), with volume-weighted mean diameter of  $46.44 \pm 3.9$   $\mu\text{m}$  in comparison to  $54.77 \pm 0.04$   $\mu\text{m}$ . However, the uniformity of droplets was enhanced using ME and the resulting emulsion had a *span* value of 0.65, while the emulsions obtained by homogenisation had *span* of 1.08 for the dispersed phase content of 2 0% (v/v).

HI P 2

**Izolovanje i optimizacija steroidnih sapogenina iz semena  
*Trigonella foenum-graceum* L. superkriticnom ekstrakcijom  
pri različitim procesnim uslovima**

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Steroidni sapogenini su poznati kao polazni materijal za dobijanje steroidnih hormona u farmaceutskoj industriji, pri čemu je od posebnog interesa njihovo dobijanje iz prirodnih sirovina kao što je seme *Trigonella foenum-graecum* L. Natkritična ekstrakcija sa ugljenik(IV)-oksidom iz semena *Trigonella foenum-graecum* L. je optimizovana u cilju izolovanja steroidnih sapogenina, a nakon predtretmana odmaščivanja i kisele hidrolize polaznog materijala. Prinos ekstrakta steroidnih sapogenina je analiziran pri različitim uslovima procesnih parametara natkritične ekstrakcije uključujući pritisak, temperaturu i vreme trajanja ekstrakcije u cilju optimizacije efikasnosti procesa.

**Optimization and isolation of steroid sapogenins from  
*Trigonella foenum-graecum* seeds L. for various process parameters  
of supercritical extraction**

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Steroidal sapogenins are known as precursors for obtaining steroidal hormones in pharmaceutical industry and their isolation has been of importance especially from natural sources such as seeds of *Trigonella foenum graecum* L. A focused supercritical extraction method was developed for the extraction of steroidal sapogenins from fenugreek seeds (*Trigonella foenum-graecum* L.), after pretreatment of defatted and acid hydrolyzed seeds. The yield of steroidal sapogenins was studied for various process parameters of supercritical extraction, such as pressure, temperature and extraction time in order to optimize the extraction efficiency.

HI P 3

### **Difuzija kofeina iz lipidozoma modifikovanih površinski aktivnim komponentama**

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Fosfolipidne mikročestice (lipozomi) su pokazale značajan potencijal u oblasti kontrolisanog otpuštanja aktivnih komponenti.

Predmet ovog rada je ispitivanje uticaja sastava membrane lipozoma na brzinu oslobađanja inkapsuliranog kofeina. Kofein je multifunkcionalna aktivna komponenta koja se sve više koristi u kozmetičkim i farmaceutskim proizvodima. Inkapsulacija u lipozome sa različitim sastavom membrane bi trebalo da omogući kontrolisanje brzine otpuštanja kofeina.

Eksperimentalno je ispitivan uticaj sastava membrane lipozoma na prenos mase. Praćena je difuzija iz lipozoma čija je membrana sastavljena isključivo od fosfolipida i lipozoma čija je membrana smeša fosfolipida i površinski aktivnih materija: Tween 20 (polisorbat-20), Tween 40 (polisorbat-40) i Tween 60 (polisorbat-60). Difuzioni eksperimenti izvedeni su u standardnoj Franz-ovoj difuzionoj ćeliji.

Na osnovu difuzionih eksperimenata određeni su koeficijenti difuzije kofeina iz konvencionalnih i modifikovanih lipozoma. Rezultati eksperimentalnih ispitivanja pokazali su da se modifikacijom membrane površinski aktivnom materijom ubrzava difuzija kofeina i da se promenom sastava membrane može kontrolisati brzina otpuštanja aktivnih komponenti.

### **Diffusion of caffeine from liposomes modified by surfactants**

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Phospholipid microparticles (liposomes) have shown significant potential in the controlled release of actives.

The subject of this paper is to investigate the influence of the membrane of the liposomes on the release rate of encapsulated caffeine. Caffeine is a multifunctional active component that is increasingly used in cosmetic and pharmaceutical products. Encapsulation in liposomes with different membrane composition should allow controlling the release rate.

The effect of the liposome membrane composition on mass transfer was studied experimentally. The diffusion from the liposome whose membrane is composed solely of phospholipids and liposomes whose membrane was mixture of phospholipids and surfactants: Tween 20, Tween 40 and Tween 60, was studied. Diffusion experiments were performed in a standard Franz diffusion cell.

Based on experimental results diffusion coefficients of caffeine from conventional and modified liposome are determined. The results show that diffusion from surfactant modified liposomes is significantly faster, and that membrane composition affects diffusion rate.



HI P 4

## Uticaj protoka tečnosti na zapreminski koeficijent prenosa mase kiseonika u koloni sa fontansko-fluidizovanim slojem i centralnom cev

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Uticaj brzine tečnosti, vode, na zapreminski koeficijent prenosa mase kiseonika iz vazduha,  $k_{L,a}$  je ispitivan u trofaznoj 2D polukoloni sa fontansko-fluidizovanim slojem i centralnom cevi. Treću, čvrstu fazu su činile staklene sferične čestice prečnika 6 mm. U kolonu je voda uvedena kroz mlaz protokom od 2,5 m<sup>3</sup>/s i anulus protocima 1,0, 1,5, i 2,0 m<sup>3</sup>/s. Vazduh je protocima 300-800 l/h uveden samo kroz mlaz odnosno kroz centralnu cev, samo kroz anulus i kroz oba ova dela kolone istovremeno. Rezultati istraživanja su pokazali da je najveća vrednost  $k_{L,a}$  dobijena kada je vazduh uveden u anularni deo kolone, za razliku od nižih vrednosti ovog koeficijenta dobijenih kada je vazduh uveden samo kroz centralnu cev, ili kada je protok vazduha bio raspodeljen po čitavom poprečnom preseku kolone. U celom sistemu se zbog uvođenja dva nezavisna toka tečnosti (mlazni i anularni) i njihove međusobne interakcija preko ulaznog regiona i cirkulacije čestica ne ostvaruju uniformi uslovi za prenos mase kiseonika. Pored količine uvedenog vazduha u sistem i formiranih mehurova usled interakcija sa staklenim česticama, važnu ulogu ima brzina tečnosti. Sa jedne strane brzina tečnosti utiče na povećanu turbulenciju u sistemu i doprinosi povećanom prenosu mase kiseonika, dok sa druge strane utiče na smanjenje vremena boravka mehurova vazduha, a samim tim i na smanjenje  $k_{L,a}$ .

## Influence of liquid flow rate on the volumetric mass transfer coefficient of oxygen in a spout-fluidized bed with a draft tube

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The influence of fluid velocity (water) on the volumetric mass transfer coefficient of oxygen from air,  $k_{L,a}$ , have been studied in three phase 2D semicolon with spout- fluidized bed and a draft tube. Third phase, the solid phase, was formed by glass spherical particles with a diameter of 6 mm. The liquid flow in the column had a flow rate of 2.5 m<sup>3</sup>/s and annulus flow rates of 1.0, 1.5, and 2.0 m<sup>3</sup>/s. Air, with flow rates of 300-800 l/h was fed through the jet or through a central tube, through the annulus or through both of these columns simultaneously. The results showed that the highest  $k_{L,a}$  value was produced when the air is introduced into the annulus of the column, as opposed to the lower value of this ratio obtained when the air is fed only through the central tube or when the airflow was distributed throughout the cross section of the column. Due to the introduction of two independent fluid flows (jet and the annulus) and their mutual interaction via the input region and the circulation of particles, uniform conditions for mass transfer of oxygen are not achieved in the system. In addition to the amount of air introduced into the system and bubbles formed due to interaction with glass particles, an important role is played by the liquid velocity. On one hand the fluid velocity affects the increased turbulence in the system, contributing to increased mass transfer of oxygen, on the other hand it affects the reduction in the residence time of bubbles of air, and thus to a reduction of  $k_{L,a}$ .

HI P 5

**Prenos mase sa i bez hemijske reakcije u partikulativno fluidizovanom sloju**

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U ovom radu su prikazani rezultati eksperimentalnog ispitivanja prenosa mase između zida kolone i fluidizovanog sloja uz brzu, nepovratnu hemijsku reakcije. Ispitivanja su vršena u koloni prečnika 34 mm sa unutrašnjim žljebom ispunjenim benzoevom kiselinom. Fluidizovani sloj su činile inertne, sferne staklene čestice prečnika 1,94 mm, a kao fluidizacioni medijum korišćen je vodeni rastvor natrijum hidroksida. Da bi se dobio jasan rezultat o uticaju prisutnih čestica na intenzitet prenosa mase eksperimenti bez i sa hemijskom reakcijom su urađeni i u sistemima bez čestica.

Utvrđena je vrednost faktora uvećanja koeficijenta prenosa mase zbog prisutne hemijske reakcije. Pokazalo se da uticaj prisutnih čestica i uticaj hemijske reakcije na prenos mase imaju aditivni karakter. Prisutna hemijska reakcija u sistemima bez čestica uvećava koeficijent prelaza mase 20 puta, dok prisustvo fluidizovanih čestica uvećava koeficijent prelaza mase 28 puta. Dobijeni rezultati su pokazali dobra slaganja sa literaturnim korelacijama.

Rezultati su prikazani i kao bezdimenzioni faktor prenosa mase u funkciji bezdimenzione poroznosti u cilju postavljanja analogije prenosa količine kretanja i mase u fluidizovanom sloju sa prisutnom hemijskom reakcijom.

**Mass transfer without and with chemical reaction in particulate fluidized beds**

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This work presents the results of an experimental investigation of the mass transfer between the column wall and fluidized bed accompanied with fast, irreversible chemical reaction. Experiments were performed in the column 34 mm in diameter with inner channel filled with benzoic acid. The inert, spherical glass particles with diameter of 1.94 mm were used as fluidized bed and the fluidization medium was aqueous solution of sodium hydroxide. In order to obtain a clear result of the impact of particles on the intensity of the mass transfer, experiments with and without chemical reaction are also done in systems without particles. The value of the enhancement factor of the mass transfer coefficient due to the presence of chemical reactions was found using experimental results. It was shown that the influence of the particles presence and the effect of chemical reactions have additive character on the mass transfer. The chemical reaction in systems without particles increases mass transfer coefficients 20 times, while the presence of fluidized particles increases the mass transfer coefficients 28 times. Obtained results showed good agreement with literature correlations. In order to establish analogies between momentum and mass transfer in a fluidized bed, with chemical reaction, experimental results are also presented as dimensionless mass transfer factor as a function of dimensionless porosity.

HI P 6

### **Analogija prenosa količine kretanja i toplote u partikulativno fluidiziranim slojevima**

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Proučavanje partikulativno fluidiziranih slojeva kao i odgovarajućih fenomena prenosa od velikog je značaja za hemijske i biohemijske procese. Pri projektovanju uređaja sa fluidiziranim slojem tečnost–čestice neophodno je poznavati zavisnosti koje povezuju hidrodinamičke parametre sa koeficijentima prelaza toplote i mase.

U ovom radu prikazani su rezultati eksperimentalnih ispitivanja prenosa količine kretanja i prenosa toplote u partikulativno fluidiziranim slojevima sferičnih čestica. Eksperimenti su izvedeni sa sferičnim staklenim česticama prečnika od 0.80 do 2.98 mm, sa vodom, u bakarnoj koloni prečnika 25.4mm, sa omotačem kroz koji je propuštana vodena para kao grejni fluid.

Dobijeni podaci za prenos toplote, prikazani su u zavisnosti od međufaznog koeficijenta trenja fluid–čestice dobijenim pomoću varijacionog modela<sup>1</sup>, i upoređeni su sa rezultatima ranijih ispitivanja prenosa mase<sup>2</sup>. Svi podaci, za prenos količine kretanja, toplote i mase u partikulativno fluidiziranim slojevima sferičnih čestica, ukazuju na postojanje analogije, a ovako uspostavljena analogija daje mogućnost primene modela za određivanje koeficijenta trenja fluid–čestice i za određivanje koeficijenta prelaza toplote i mase.

### **Analogy between momentum and heat transfer in particulate fluidized beds**

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The study of particulate fluidized beds as well as the appropriate transfer phenomena is of great importance for the chemical and biochemical processes. The design of equipment with liquid–solid fluidized beds is mainly based on knowledge of the hydrodynamics and heat and mass transfer coefficients.

Wall–to–bed heat transfer in particulate fluidized beds of spherical particles was studied. The experiments were performed using spherical glass particles of 0.80 to 2.98 mm in diameter, with water, in a 25.4 mm I.D. copper tube equipped with a steam jacket.

Heat transfer data related to the fluid–particle interphase drag coefficient predicted by the variational model<sup>1</sup>, were obtained and compared with previous results for the wall–to–bed mass transfer in fluidized beds<sup>2</sup>. All the data for momentum, heat and mass transfer in particulate fluidized beds of spherical particles, show that an analogy among these three phenomena exists. An analogy established in this work gives possibility of application of the proposed model for determination of fluid–particle interphase drag coefficient and for heat and mass transfer coefficients in liquid–solid fluidized beds.

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## Предвиђање експанзије слоја полидисперзних смеша кварцног песка флуидизованих са водом

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У циљу одређивања експанзије слоја полидисперзних смеша честица песка током флуидизације слоја, извршени су експериментална мерења са 19 различитих фракција филтрационог песка чији је сејани пречник у интервалу  $d_m = 0.359$  до 2.596 мм.

Експериментални резултати експанзије слоја корелисани су користећи неколико једначина доступних у литератури са циљем да се испита примена једначина за предвиђање експанзије слоја полидисперзних смеша песка током флуидизације. Неколико корелација оригинално изведених за моно-сферичне честице, као и за моно-несферичне честице, испитивано је користећи  $d_{SV}$  као репрезентативни пречник.

На основу експерименталних мерења, предложен је нови модел за предвиђање експанзије слоја полидисперзних смеша песка флуидизованих са водом. Ново предложени модел даје средњу апсолутну грешку од 2,66% у предвиђању порозности слоја,  $\epsilon$ , за дату вредност површинске брзине воде,  $U$ .

## Prediction of bed expansion of polydisperse quartz sand mixtures fluidized with water

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In order to investigate minimum fluidization velocity and bed expansion of the polydisperse sand mixtures, fluidization experiments were conducted using 19 different fractions of quartz filtration sand with sieve diameters in the interval  $d_m = 0.359$  to 2.596 mm. The experimental results of bed expansion were correlated using a number of equations available in the literature and their applicability to polydisperse mixtures was investigated. A number of correlations developed for monosized spherical particles as well as for monosized non-spherical particles were tested using  $d_{SV}$  as representative diameter.

Based on the experimental measurements, a new model for the prediction of bed expansion for water fluidized beds of sand particles was proposed. In predicting the bed porosity,  $\epsilon$ , for a given superficial velocity,  $U$ , the new model showed a mean absolute deviation of 2.66%.

## **Hidrodinamičko modelovanje vertikalnog suprotnostrujnog toka gas-čvrste čestice**

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U ovom radu prikazan je i eksperimentalno potvrđen jednodimenzioni model suprotnostrujnog toka fluida i čestica u zoni ubrzanja, merenjem pritiska duž transportne cevi. Model bazira na jednačinama kontinuiteta, bilansima količine kretanja za fluid i čestice i varijacionom modelu za predviđanje koeficijenta trenja fluid-čestice.

Eksperimenti su izvedeni sa sferičnim staklenim česticama prečnika 1,94 mm, u vertikalnoj transportnoj akrilnoj cevi prečnika 16 mm i dužine 2230 mm, pri konstantnom masenom fluksu čestica. Kao transportni fluid korišćen je vazduh.

Eksperimentalni podaci za statički pritisak fluida duž transportne cevi u dobroj su saglasnosti sa predviđenim vrednostima po modelu. Vrednosti za gradient pritiska, poroznost, brzinu čestica i relativnu brzinu između fluida i čestica, duž transportne cevi izračunate su prema formulisanom modelu. U ovim izračunavanjima, koeficijent trenja čestice-zid transportne cevi određen je indirektno.

Izračunavanja takođe pokazuju, da je dužina zone ubrzanja za iste čestice (1,94 mm), kod suprotnostrujnog toka fluida i čestica oko dva puta veća od dužine zone ubrzanja kod istostrujnih gas-čestice sistema na gore. U ispitivanom sistemu, "brzina zagušivanja" javlja se na relativnoj brzini između fluida i čestica, koja je oko 73 % od brzine odnošenja usamljene čestice.

### **Hydrodynamic modeling of vertical counter-current gas-solids flow**

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The one-dimensional model of accelerating turbulent downward counter-current gas-solids flow of coarse particles was formulated and experimentally verified by measuring the pressure distribution along the transport tube. The continuity and momentum equations were used in the model formulation and variational model was used for the prediction of the fluid-particle interphase drag coefficient.

Experiments were performed by transporting spherical glass particles 1.94 mm in diameter with air, in a 16 mm i.d. acrylic tube, the length 2230 mm, at constant solids mass flux.

Experimental data for the static fluid pressure distribution along the transport tube agree quite well with the model predictions. The values of the pressure gradient, porosity, particle velocity and slip velocity along the tube were calculated according to the formulated model. In these calculations, particle-wall friction coefficient was determined indirectly.

Calculations show that the acceleration length for the same particles (1.94 mm) in downward counter-current gas-solids flow is about two times higher than the acceleration length in upward co-current gas-solids flow. In the system investigated, "choking" occurs at slip velocity which is about 73 % of the single particle terminal velocity.



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**Hydrodynamic modeling of co-current gas/coarse particle flow in downer**

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The one-dimensional model of accelerating turbulent downward co-current gas-solids flow of coarse particles ( $d_p=1.94$  mm) was formulated and experimentally verified by measuring the pressure distribution along the transport tube. The continuity and momentum equations were used in the model formulation and variational model was used for the prediction of the fluid-particle interphase drag coefficient. The values of the pressure gradient, porosity, particle velocity and slip velocity along the tube were calculated according to the formulated model. The distance from the transport tube inlet where the slip velocity changes its sign from positive to negative is the function of the gas superficial velocity. At positive slip velocity both gravity and drag contribute to particle acceleration. At negative slip velocity the drag force acts in upward direction resisting the particle acceleration. In downward co-current gas-solid flow acceleration length is relatively long, about two times longer compared to the upward co-current gas-solid flow.

**Хидродинамичко моделовање истострујног тока гас/крупне честице при струјању наниже**

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Развијен је једнодимензиони модел за турбулентни истострујни ток гас-крупне чврсте честице (1.9 mm) у зони убрзавања и експериментално је верификован мерењем дистрибуције притиска дуж транспортне цеви. Коришћене су једначине континуитета и количине кретања у формулисању модела, а варијациони модел је коришћен за предвиђање међуфазног коефицијента трења течност-честице. Вредности градијента притиска, порозности, брзине честица и релативне брзине између флуида и честица дуж цеви су рачунате помоћу предложеног модела. Растојање од улаза у транспортну цев на коме релативна брзина између честица и флуида мења знак из позитивног у негативни је функција површинске брзине гаса. При позитивним вредностима релативне брзине између флуида и честица убрзању честица доприносе и гравитација и сила трења. При негативним вредностима релативне брзине између флуида и честица сила трења делује у смеру навише пружајући отпор убрзању честица. У истострујном току гас-крупне честице при струјању наниже зона убрзавања честица је релативно дуга, око два пута већа у односу на истострујни ток гас- крупне честице при струјању навише.

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## **Mehanohemijaska sinteza i karakterizacija Ca- odnosno Zn-glicerolata i primena kao katalizatora za sintezu biodizela**

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U ovom radu Ca-glicerolat (CaG) i Zn-glicerolat (ZnG) dobijeni su mehanohemijskim tretmanom CaO, ZnO i glicerola tokom 2 časa. Nakon ovog vremena dodat je alkohol (metanol ili etanol), a mlevenje nastavljeno još 15 minuta. Karakterizacija sintetizovanih glicerolata izvršena je rendgenskom strukturnom analizom (XRD), termogravimetrijskom analizom (TGA/DTA), infracrvenom spektroskopijom (FTIR) i analizom baznosti titracijom pomoću Hammetovih indikatora. Rezultati su pokazali da je moguće dobiti CaG i ZnG pod određenim uslovima mehanohemijske sinteze. Pripremljeni katalizatori testirani su u metanolizi suncokretovog ulja na 60 °C pri molarnom odnosu metanola i ulja od 10:1 i sa 2 % katalizatora u odnosu na masu ulja.

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## **Mechanochemical synthesis and characterization of Ca- and Zn-glycerolate and their application as catalyst for biodiesel synthesis**

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In this study Ca-glycerolate and Zn-glycerolate were synthesized by mechanochemical treatment of ZnO or CaO with glycerol (2 hours). At the end of ball milling, alcohol (methanol or ethanol) was added, and the milling was continued for another 15 minutes. Characterisation of prepared glycerolate was performed by X-ray diffraction (XRD), thermogravimetric analysis (TGA/DTA), infrared spectroscopy (FTIR), and Hammett indicator method for base strength determination. The results showed that both Ca-glycerolate (CaG) and Zn-glycerolate (ZnG) could be obtained by mechanochemical treatment under certain conditions. Prepared glycerolates were tested in the methanolysis of sunflower oil at 60 °C with the molar ratio of methanol to oil of 10:1 and with 2 wt% of catalyst based on oil weight.

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**Liquid liquid equilibria of binary and pseudo binary systems of Aniline or N,N-Dimethylaniline + Water Solutions: Effects of Solid Poly (ethylene glycols) addition**

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Reliable phase equilibrium data are essential for development and design of chemical processes. In this work, liquid-liquid equilibria for two binary and four pseudobinary systems, containing polyethylene glycol 2050 (PEG2050), polyethylene glycol 35000 (PEG35000), aniline, N, N-dimethylaniline, and water, in the temperature range 298.15-350.15 K and at ambient pressure of 0.1 MPa, were investigated. The temperature-composition phase diagrams for those six systems were obtained, using visual method of cloud point determination. Our experimental results showed that the only useful co-solvent is PEG2050 for aniline in water. On the other hand, the addition of PEG35000 even provoked an anti-solvent effect.

**Ispitivanje ravnoteže tečno-tečno binarnih i pseudobinarnih sistema vodenih rastvora Anilina i N,N-dimetilanilina i efekti dodavanja čvrstih polietilen glikola**

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Pouzdana podaci fazne ravnoteže značajni su za razvijanje i projektovanje hemijskih procesa. U ovom radu, ispitivana je ravnoteža tečno-tečno dva binarna i četiri pseudobinarna sistema, koji sadrže polietilen glikol 2050, polietilen glikol 35000, anilin, N,N-dimetilanilin i vodu, u temperaturnom opsegu 298,15-350,15 K i na atmosferskom pritisku od 0,1 MPa. Dobijeni su fazni dijagrami za ovih šest sistema koristeći vizuelnu metodu određivanja. Rezultati eksperimentalnih merenja pokazali su mogućnosti korišćenja samo polietilen glikola 2050 kao ko-rastvarača za anilin u vodi.

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### **Apsorpcija ozona praćena trenutnom hemijskom reakcijom**

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U ovom radu ispitivana je apsorpcija ozona praćena trenutnom hemijskom reakcijom u tečnoj fazi u barbotажnoj koloni sa poroznom ploćom kao raspodeljivaćem gasa. Za generisanje ozona korišćen je generator ozona, *OzoneLab OL100/DS* ozonizator, dok je za praćenje promene boje rastvora korišćen računarski program "Sigma Scan Pro 5". Cilj ovih ispitivanja bio je određivanje zapreminskog koeficijenta prenosa mase ozona za fizićku apsorpciju, zapreminskog koeficijenta prenosa mase ozona za apsorpciju praćenu trenutnom hemijskom reakcijom ozona i kalijum indigotrisulfonata i vrednosti faktora uvećanja. Za određivanje brzine apsorpcije pretpostavljeno je idealno mešanje u tečnoj fazi. Na osnovu dobijenih vrednosti zapreminskih koeficijenata prenosa mase određena je vrednost faktora uvećanja koja pokazuje koliko puta je apsorpcija praćena trenutnom hemijskom reakcijom brža od čiste fizićke apsorpcije. Rezultati su pokazali da je izraz koji definiše faktor uvećanja za velike vrednosti faktora uvećanja pogodan i za proraćune malih vrednosti faktora uvećanja.

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### **Absorption of ozone accompanied by instantaneous chemical reaction**

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The absorption of ozone accompanied by instantaneous chemical reaction in the liquid phase, in bubble column with porous plate as the distributor of gas, are presented in this work. Ozone generator, *OzoneLab OL100 / DS* ozonizer was used for ozone generation, while monitoring the color change of the solution was performed by using a computer program "Sigma Scan Pro 5". The main objective was to determine the ozone volumetric mass transfer coefficient for physical absorption, the ozone volumetric mass transfer coefficient for absorption accompanied by instantaneous chemical reaction and the value of the enhancement factor. To determine the absorption rate, ideal mixing in the liquid phase was assumed. The value of the enhancement factor shows how many times the absorption accompanied by instantaneous chemical reaction is faster than pure physical absorption was experimentally determined. The results showed that the formula that defines the enhancement factor, for large values of enhancement factor is suitable for calculations of small values of enhancement factors.

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## **Kinetika termičke oksidacije neprečišćenih i prečišćenih višeslojnih ugljeničnih nanocevi**

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Izvršena je kinetička analiza neizotemske oksidacije neprečišćenih i prečišćenih višeslojnih ugljeničnih nanocevi (UNC), a dobijeni kinetički parametri su korelisani sa njihovim termičkim i teksturalnim osobinama. Za kinetičku analizu korišćeni su eksperimentalni podaci dobijeni primenom TG/DTG, u temperaturnom opsegu od 25-1000°C, pri brzini zagrevanja od 10°C/min. Podaci su potom fitovani primenom nekoliko odabranih matematičkih modela u cilju odabira kinetičkog modela koji najbolje opisuje proces dekompozicije UNC. Dobijene vrednosti energije aktivacije i predeksponencijalnog faktora neprečišćenih uzoraka nanocevi potvrđuju njihovu različitu međusobnu termičku stabilnost i mehanizam dekompozicije, što je i u saglasnosti sa njihovim teksturalnim karakteristikama. Kinetički parametri ukazuju i na promenu oksidativne stabilnosti UNC nakon njihovog prečišćavanja, što je naročito naglašeno u slučaju nanocevi poreklom iz katalizatora sa aluminom kao nosačem. Kinetička analiza termičke oksidacije uzorka prečišćenih UNC poreklom iz katalizatora sa nosačem od silike zahteva primenu eksperimentalnih podataka dobijenih termičkom analizom pri različitim uslovima sagorevanja cevi, kao i primenu više različitih matematičkih modela za njihovu obradu. Stoga je odabir odgovarajućih eksperimentalnih uslova, kao i matematičkih metoda od izuzetnog značaja za adekvatan opis celokupnog mehanizma termičke degradacije ugljeničnih nanocevi.

## **Thermal oxidation kinetics of as-grown and purified multi-walled carbon nanotubes**

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The aim of this work was to study the nonisothermal oxidation kinetics of as-grown and purified carbon nanotubes (CNTs) and to correlate kinetic parameters to their thermal and textural properties. The kinetic analysis was performed by using the TG/DTG experimental data collected in the temperature range 25-1000°C, at a heating rate of 10°C/min. The data were fitted by using selected model-fitting methods to find an appropriate kinetic model of NCTs decomposition. The determined values of activation energy and pre-exponential factor for the as-grown samples confirm different thermal stability among CNTs, while the different mechanisms of their decomposition are in accordance with tubes specific textural properties. The kinetic parameters confirmed CNTs oxidative stability evolution due to the applied purification treatment, however, only for alumina-based CNTs. The kinetic study of the purified sample of silica-based catalyst origin requires experimental data obtained by thermal analysis performed at different conditions, and also the application of several kinds of mathematical models for processing the results. Therefore, the selection of appropriate experimental conditions, as well as mathematical methods is very important in order to get an adequate description of the complete thermal degradation reaction mechanism of CNTs and their kinetics.



HI P 14

## Experimental measurements of density for the ternary system ethylbutyrate + diethylsuccinate + isobutanol

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Systematic studies of density, as well as excess molar volumes, of multicomponent liquid mixtures on different temperatures are significant since it allows better insight into the liquid state theory and intermolecular interactions. This work is a continuation of our study of the thermodynamic and transport properties of liquid mixtures containing different important esters. Density data of the ternary system ethylbutyrate + diethylsuccinate + isobutanol have been measured in the temperature range 288.15–323.15 K, with a temperature step of 5 K, and at atmospheric pressure. The measurements were performed on Anton Paar DMA 5000 digital vibrating tube densimeter. Excess molar volumes ( $V^E$ ) were determined and fitted by the Nagata-Tamura polynomial.

## Eksperimentalno određivanje volumetrijskih svojstava ternernog sistema etilbutirat + dietilsukcinat + izobutanol

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Sistematična analiza gustine, kao i dopunske molarne zapremine, višekomponentnih tečnih smeša na različitim temperaturama je veoma značajna i omogućava nam bolji uvid u međumolekulske interakcije prisutne u smešama. Ovaj rad je nastavak istraživanja termodinamičkih i transportnih veličina tečnih smeša koje sadrže različite značajne estre. Eksperimentalno su određene gustine ternernog sistema etilbutirat + dietilsukcinat + izobutanol u temperaturnom intervalu 288.15–323.15 K, sa korakom od 5 K i na atmosferskom pritisku. Eksperimentalna merenja su izvršena na digitalnom gustinomeru Anton Paar 5000. Izračunate su dopunske molarne zapremine ( $V^E$ ) i njihova analitička zavisnost od sastava smeše je obrađena Nagata-tamura polinomom.

*The authors gratefully acknowledge the financial support received from the Research Fund of Ministry of Education, Science and Technological Development (project No 172063), Serbia and the Faculty of Technology and Metallurgy, University of Belgrade.*



HI P 15

### **Density of biodiesels from sunflower oil at high pressures**

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In the past few decades, the use of biodiesel as a fuel has greatly increased in order to overcome the shortage of fossil fuels and to reduce the environmental pollution. One of the major advantages of biodiesel is the possibility of its use in most injection pump diesel engines, pure or mixed with petroleum diesel, without significant corrections of an engine design. The replacement of a diesel with a biodiesel still affects the process of injecting the fuel in the engine and the speed of ignition so it is necessary to have knowledge of a lot of biodiesel properties under various operating conditions. Some of the most important are the density and in the isothermal compressibility and the isobaric thermal expansivity that can be derived from it. Hence, the densities of the methyl and the ethyl esters of the fatty acids from sunflower oil were measured at temperatures 288.15–413.15 K and at pressures 0.1–60 MPa. The Anton Paar DMA HP densimeter was used for the measurements and the densities were calculated applying classical one-fluid calibration method. The obtained values were fitted to the modified Tammann-Tait equation and the optimized parameters were used in the isothermal compressibility and the isobaric thermal expansivity calculation.

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### **Gustine biodizela dobijenih iz suncokretovog ulja na višim pritiscima**

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U poslednjih nekoliko decenija upotreba biodizela kao goriva se znatno povećala u cilju prevazilaženja nestašice fosilnih goriva i smanjenja zagađivanja životne sredine. Jedna od glavnih prednosti biodizela jeste mogućnost njegove upotrebe u većini dizel motora, čistog ili u smeši sa dizelom gorivom, bez značajnijih promena u dizajnu motora. Zamena dizela biodizelom ipak utiče na proces ubrizgavanja goriva u motor i na brzinu paljenja pa je neophodno poznavanje velikog broja osobina biodizela na različitim uslovima. Neke od najvažnijih su gustina, kao i izotermna kompresibilnost i izobarska termička ekspanzivnost, koje se iz nje mogu izvesti. Stoga su merene gustine metil i etil estara masnih kiselina iz suncokretovog ulja na temperaturama 288.15–413.15 K i pritiscima 0.1–60 MPa. Za merenja je korišćen DMA HP gustinomer, proizvođača Anton Paar i gustine su računane primenom klasične kalibracione metode sa jednim referentnim fluidom. Dobijene vrednosti su modelovane modifikovanom Tammann-Tait-ovom jednačinom i optimizovani parametri su poslužili za računanje izotemske kompresibilnosti i izobarske termičke ekspanzivnosti.



## **Analiza organske supstance iz uglja u cilju procene njegovog stepena karbonifikacije**

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Radovi većeg broja istraživača pokazali su da se organska supstanca ugljeva, počev od lignita do antracita, sastoji najvećim delom od nerastvornog dela (kerogena) koji je makromolekulske prirode i od relativno male količine (manje od 10%) u organskim rastvaračima rastvornog dela (bitumena).

Veliki doprinos u ispitivanju organske supstance uglja daju organsko-geohemijske analize kojima se dolazi se do podataka o poreklu, načinu postanka i hemijskoj strukturi uglja. Ova istraživanja su aktuelna za zemlje bogate ugljem, a posebno za Republiku Srbiju koja raspolaže velikim rezervama uglja.

U ovom radu je ispitivana organska supstanca uglja iz centralnog, južnog i istočnog dela kosovskog basena. Analizirani su uzorci iz ugljenog sloja bliže povlata, sredine i ugljenog sloja bliže podini. Akcenat ovog istraživanja je dat na određivanje ukupne (nekorigovane i korigovane) organske supstance, određivanje sadržaja i sastava slobodnog bitumena, određivanje sadržaja i sastava vezanog bitumena i određivanje sadržaja i elementarnog sastava kerogena. Dobijeni rezultati mogu poslužiti za procenu porekla kao i stepena zrelosti organske supstance u cilju određivanja stepena karbonifikacije ispitivanog uglja.

## **Analysis of organic substances from coal in order to assess its level carbonification**

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The works of a number of researchers have shown that the organic substance of coal, ranging from lignite to anthracite, consisting mainly of insoluble part (kerogen), which is the macromolecular nature and of relatively small amounts (less than 10%) of soluble (in organic solvents) part (bitumen).

Great contribution in the study of organic substance of coal provides organic-geochemical analysis that leads to information about the origin, genesis and chemical structure of coal. These studies are substantial for countries rich in coal, and in particular for the Republic of Serbia, which has large reserves of coal.

In this study, the organic substances of coal from the central, southern and eastern part of Kosovo Basin were studied. We analyzed samples from the coal seam closer to the roof, middle and the coal seam closer underlain. The emphasis of this research is given to the determination of the total (unadjusted and adjusted) organic substance, determine the content and composition of free bitumen, determining the content and composition in bound and determination of the elemental composition of kerogen. The obtained results can be used to estimate the origin and degree of maturity of organic matter in order to determine the degree of carbonification of the tested coal.

## Nauka o materijalima / Material Science

MAT P 1

### **Sinteza i karakterizacija filmova na bazi pektina i glicidil metakrilata dobijenih u reakciji fotopolimerizacije**

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U ovom radu visoko metil-esterifikovani pektin je hemijski modifikovan u reakciji umrežavanja sa glicidilmetakrilatom (GMA) u cilju dobijanja "bio-based" filma. Predloženom modifikacijom uvedene su vinil grupa u strukturu polisaharida. Nakon fotoinicijacije (365 nm) vinil grupe reaguju preko slobodnih radikala i proizvode umreženu strukturu sa povećanom hidrofobnošću i poboljšanim mehaničkim svojstvima. U prvom koraku sinteze glicidilmetakrilat je kalemljen na lanac pektina, dok se umrežavanje odigrava u drugom koraku UV - indukovanom reakcijom polimerizacije GMA koristeći benzofenon kao fotoinicijator. U ovom radu će biti predstavljena sinteza filma, utvrđivanje interakcija između pektina i GMA pomoću FTIR -ATR i NMR analize, kao i termijska analiza dobijenog filma.

### **Synthesis and characterizaton of films based on pectin and glycidyl methacrylate obtained in photopolymerization reaction**

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In this work high-methylated pectin was chemically modified in a cross-linking reaction using glycidyl methacrylate (GMA) in order to obtain a bio-based film. The objective of this modification is the introduction of vinylic groups in the polysaccharide structure. After the photoinitiation (365 nm) vinylic groups will react through free radicals producing cross-linked structure with increased hydrophobicity and improved film-forming and mechanical properties. Namely, in the first step glycidyl methacrylate was grafted to the pectin backbone, whereas the system was additionally cross-linked in the second step by UV-induced polymerization reaction of GMA with benzophenone as a photoinitiator. In this work, we will present the synthesis, elucidation of interactions between pectin and GMA by FTIR-ATR and NMR analysis, as well as thermal behavior of gained film.

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**MAT P 2**

**Novel protective materials for Cultural Heritage monuments**

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Porous mineral building materials are susceptible to a variety of degradation processes as the result of the entry and/or presence of water and environmental pollution. The pollution products are usually deposited on the materials surface and could penetrate further into the bulk. Degradation of building materials in the field of Cultural Heritage due to presence of biological contaminants on the surfaces, represents a serious problem. Newly developed protective materials - two types of consolidant formulations (for carbonate and silicate based substrates) and the protective coating (based on double layered hydroxides associated with TiO<sub>2</sub>) were applied on porous mineral model substrates (brick and render), in order to assess surface properties and antifungal activity. The surface properties of the treated brick and render model substrates were assessed through the measurements of the contact angle values and contactless diffusion reflection FTIR analysis (DRIFT mode) after different consolidation periods (1, 2 4 and 7 months). Due to the lack of the standardized method for antifungal activity assessment of the photocatalytic coatings on the porous model substrates, a modification of the ISO/DIS 13125 standard was evaluated and implemented. The obtained results shows better antifungal activity for the samples treated with the developed protective coating and consolidants (as a protective system) in comparison to the samples treated only with the developed consolidants.

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**Novi materijali u zaštiti spomenika kulturne baštine**

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Porozni građevinski materijali su podložni raznim degradacionim procesima usled dejstva vode iz okolne atmosfere. Polutanti se najčešće deponuju na površini materijala odakle mogu da prodiru dalje u unutrašnjost. Degradacija građevinskih materijala u oblasti kulturne baštine usled prisustva mikroorganizama na površini predstavlja ozbiljan problem. Novodizajnirani zaštitni materijali – dva tipa konsolidanata (za karbonatne i silikatne substrate) i zaštitna prevlaka (na bazi dvostrukih slojevitih hidroksida sa TiO<sub>2</sub>) su aplicirani na porozne mineralne model substrate (cigla i render) sa ciljem valorizacije površinskih karakteristika i antifungalne aktivnosti. Površinske karakteristike tretiranih materijala su analizirane na osnovu merenja ugla kvašenja i FTIR bezkontaktne metode difuzione refleksije nakon određenog perioda konsolidacije (1, 2, 4 i 7 meseci). Antifungalna aktivnost je ocenjena na osnovu modifikovanog ISO/DIS 13125 standarda. Dobijeni rezultati ukazuju na bolju antifungalnu aktivnost uzoraka tretiranih sa novorazvijenim zaštitnom prevlakom i konsolidantima (kao zaštitnim sistemom) u odnosu na uzorke koji su tretirani samo sa novodizajniranim konsolidantima.

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**MAT P 3**

**Mezoporozni silika i titanijum-dioksid kao potencijalni nosači za kontrolisano dostavljanje gentamicina**

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Jedan od načina za prevazilaženje problema koji se javljaju pri konvencionalnoj upotrebi lekova je razvoj funkcionalnih biokompatibilnih nosača za ove supstance. Smatra se da su, između ostalih, mezoporozni materijali obećavajuće rešenje za nosače jer mogu poslužiti za kontrolisano dostavljanje širokog spektra lekova. Ovo se posebno odnosi na mezoporoznu siliku i titan-dioksid s obzirom na njihove hemijske osobine i termičku stabilnost, kao i na veliku biokompatibilnost i malu toksičnost.

U radu su ispitani mezoporozni silika i  $\text{TiO}_2$  u cilju sagledavanja njihovog potencijala kao nanonosaa za kontrolisano dostavljanje gentamicina. Praćene su adsorpcija i desorpcija gentamicina na različitim vrednostima pH, kao i kinetika desorpcije. Takođe je ispitan uticaj modifikacije površine silika čestica silanizacijom na adsorpciju i desorpciju leka. Rezultati su pokazali da su nemođifikovani silika i  $\text{TiO}_2$  adsorbovali 80 mg, odnosno 65 mg gentamicina po gramu materijala na pH 7,5, odnosno pH 5. Na pH 5 se sa silike tokom 6 časova desorbovalo približno 50% gentamicina, pri čemu su tri četvrtine ove kolićine otpuštene sa materijala tokom prva dva sata.

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**Mesoporous silica and titanium dioxide as potential carriers for controlled gentamicin delivery**

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One of the ways to overcome problems arising from conventional drug administration and usage is to develop functional biocompatible drug carriers. Among them mesoporous materials appeared as promising candidates which can deliver a variety of drug molecules in a controllable manner. In particular, mesoporous silica and  $\text{TiO}_2$  nanoparticles are readily investigated as delivery systems because they possess favorable chemical properties and thermal stability, high biocompatibility and low toxicity.

With the aim to perceive the potential of nanoparticles to act as gentamicin carrier for controlled drug delivery, mesoporous silica and  $\text{TiO}_2$  were tested. Adsorption and desorption of gentamicin into materials at different pH values were investigated as well as kinetic of desorption. The influence of surface modification of silica nanoparticles by silanization on drug adsorption and desorption was also studied. Results showed that unmodified silica and  $\text{TiO}_2$  adsorbed 80 mg and 65 mg gentamicin per gram material at pH 7.5 and pH 5, respectively. Approximately 50% gentamicin was desorbed from silica at pH 5 during 6 hours with three quarters of desorbed drug released during the first two hours.

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## Neorganska hemija / Inorganic Chemistry

NH P 1

**Sinteza i spektroskopska karakterizacija oktaedarskih kobalt(III) kompleksa sa ( $\pm$ )-1,3-pentandiamin-*N,N,N',N'*-tetraacetato ligandom**

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U reakciji između ( $\pm$ )-1,3-pentandiamin-*N,N,N',N'*-tetrasirćetne kiseline (1,3- $H_4$ pndta) i  $CoCl_2 \cdot 6H_2O$  u 1:1 molskom odnosu i pH vrednosti 6,0 – 7,0 izolovana su tri oktaedarska, 3-pndta-Co(III) kompleksa koji sadrže različite kontra-katjone,  $Li[Co(1,3-pndta)] \cdot 2H_2O$ ,  $K[Co(1,3-pndta)] \cdot H_2O$  i  $Ca[Co(1,3-pndta)]_2 \cdot 5H_2O$ . Za karakterizaciju ovih kompleksa upotrebljena je elementarna mikroanaliza, infra-crvena, elektronska apsorpciona i NMR ( $^1H$  i  $^{13}C$ ) spektroskopija. Hemijski sastav ovih kompleksa je potvrđen metodom difrakcije X-zraka sa kristala od strane Hemijskog fakulteta, „Adam Mickijević“ Univerziteta iz Poznanja, Poljska. Dobijeni spektroskopski podaci ispitivanih 1,3-pndta-Co(III) kompleksa su poređeni sa odgovarajućim podacima za  $K[Co(1,3-pdta)] \cdot 2H_2O$  kompleks poznate kristalne strukture.<sup>1-3</sup>

**Synthesis and spectroscopic characterization of octahedral cobalt(III) complexes with ( $\pm$ )-1,3-pentanediamine-*N,N,N',N'*-tetraacetate ligand**

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In the reaction between ( $\pm$ )-1,3-pentanediamine-*N,N,N',N'*-tetraacetic acid (1,3- $H_4$ pndta) and  $CoCl_2 \cdot 6H_2O$  in 1:1 molar ratio and pH range 6.0 – 7.0 three octahedral 1,3-pndta-Co(III) complexes having different counter cation,  $Li[Co(1,3-pndta)] \cdot 2H_2O$ ,  $K[Co(1,3-pndta)] \cdot H_2O$  and  $Ca[Co(1,3-pndta)]_2 \cdot 5H_2O$ , were obtained and characterized by applying elemental analysis, infrared, electronic absorption and NMR ( $^1H$  and  $^{13}C$ ) spectroscopy. The chemical constitution of these complexes has been confirmed by X-ray diffraction carried out at the Faculty of Chemistry, Adam Mickiewicz University, Poznań, Poland. The spectroscopic data of 1,3-pndta-Co(III) complexes were compared with those for the analogous  $K[Co(1,3-pdta)] \cdot 2H_2O$  complex of known crystal structure.<sup>1-3</sup>

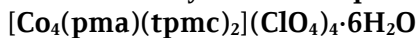
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NH P 2

**Cytotoxic *in vitro* activity of the complex compound**



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In order to investigate the biological activity, we tested on the cytotoxic activity of the complex compound  $[\text{Co}_4(\text{pma})(\text{tpmc})_2](\text{ClO}_4)_4 \cdot 6\text{H}_2\text{O}$ . The cytotoxicity was determined by MTS test on estrogen-receptor-positive breast cancer (MCF-7) cells, human malignant melanoma (Fem-x), human cervix adenocarcinoma (HeLa), myelogenous leukemia (K562), acute monocytic leukaemia cell line (THP1), and human Caucasian Burkitt's lymphoma (Ramos) cells.

A test complex compound showed significant cytotoxic activity on all tested cell lines.

Testing of the cell cycle of the tested lines, in the presence of complex compound  $[\text{Co}_4(\text{pma})(\text{tpmc})_2](\text{ClO}_4)_4 \cdot 6\text{H}_2\text{O}$ , indicates that the possible mechanism of action of this compound induction of programmed cell death (apoptosis).

**Antiproliferativna *in vitro* aktivnost kompleksa  $[\text{Co}_4(\text{pma})(\text{tpmc})_2](\text{ClO}_4)_4 \cdot 6\text{H}_2\text{O}$**

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U cilju ispitivanja biološke aktivnosti, testirana je citotoksična aktivnost kompleksa  $[\text{Co}_4(\text{pma})(\text{tpmc})_2](\text{ClO}_4)_4 \cdot 6\text{H}_2\text{O}$ . Citotoksičnost je određena MTS testom na estrogen-receptor-pozitivnim ćelijama karcinoma dojke (MCF-7), humanim ćelijama malignog melanoma (Fem-x), kao i na ćelijama humanog cervikalnog adenokarcinoma (HeLa), mijeloidne leukemije (K562), akutne monocitne leukemije (THP1) i Burkittov-og limfoma (Ramos). Testirano jedinjenje pokazalo je značajnu citotoksičku aktivnost na svim testiranim ćelijskim linijama.

Ispitivanje ćelijskog ciklusa testiranih linija, u prisutvu  $[\text{Co}_4(\text{pma})(\text{tpmc})_2](\text{ClO}_4)_4 \cdot 6\text{H}_2\text{O}$ , ukazuje da je mogući mehanizam delovanja ovog jedinjenja indukcija programirane ćelijske smrti (apoptoze).

## NH P 3

### Kinetika i mehanizam supstitucionih reakcija bimetalnog kompleksa platine(II) i paladijuma(II) sa biološki važnim ligandima

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Bimetalni kompleksi platine(II) i paladijuma(II) predstavljaju novu generaciju potencijalnih antitumorskih lekova.<sup>1</sup> S obzirom da su neki tumori otporni na dejstvo cisplatine istraživanja idu u smeru sinteze novih kompleksa čije strukturne karakteristike omogućavaju alternativni mehanizam antitumorskog dejstva, koji se razlikuje od mehanizma dejstva cisplatine i njenih analoga.<sup>2</sup>

Supstitucione reakcije novog bimetalnog dinuklearnog kompleksa platine(II) i paladijuma(II),  $[\{cis\text{-PtCl}(\text{bipy})_2\}\{\mu\text{-}(\text{H}_2\text{N}(\text{CH}_2)_6\text{NH}_2)\}\{cis\text{-PdCl}(\text{bipy})_2\}]\text{Cl}(\text{ClO}_4)$ , sa biološki značajnim nukleofilima: tioureom, L-metioninom, L-histidinom i guanozin-5'-monofosfatom, su ispitivane na pH = 5,0 (0.01M NaClO<sub>4</sub> i 40 mM NaCl) pri temperaturi od 37 °C. Sve reakcije su izučavane kao reakcije *pseudo*-prvog reda, a korišćene su stopp-flow i Uv-Vis spektrofotometrijske metode. Red reaktivnosti ispitivanih liganada je Tu > L-Met > L-His > 5'-GMP.

### Kinetics and mechanism of the substitution reactions of bimetallic complexes of platinum(II) and palladium(II) with biologically important ligands

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Bimetallic complexes of platinum(II) and palladium(II) represent a novel generation of potential antitumor drugs.<sup>1</sup> Considering the fact that some tumors are resistant to the effects of cisplatin, the investigations are going to the direction such as synthesis of novel complexes which structural characteristics provide an alternative mechanism of antitumor action that is different from mechanism of action of cisplatin and its analogues.<sup>2</sup>

The substitution reactions of new bimetallic dinuclear complex of platinum(II) and palladium(II),  $[\{cis\text{-PtCl}(\text{bipy})_2\}\{\mu\text{-}(\text{H}_2\text{N}(\text{CH}_2)_6\text{NH}_2)\}\{cis\text{-PdCl}(\text{bipy})_2\}]\text{Cl}(\text{ClO}_4)$ , were investigated with biologically relevant nucleophiles: thiourea, L-methionine, L-histidine and guanosine-5'-monophosphate at pH = 5.0 (0.01M NaClO<sub>4</sub> and 40 mM NaCl) at 37 °C. All reactions were followed as *pseudo*-first order by stopped-flow and Uv-Vis spectrophotometry methods. The observed order of reactivity of the investigated ligands is Tu > L-Met > L-His > 5'-GMP.

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## NH P 4

### Sinteza i karakterizacija derivata piridazina i njegovih kompleksa sa Co<sup>III</sup>, Ni<sup>II</sup>, Cu<sup>II</sup> i Zn<sup>II</sup>

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U ovom saopštenju opisana je sinteza, fizička i hemijska karakterizacija liganda bis(3-hloropiridazin-6-hidrazon)-2,6-diacetilpiridina (Hp<sub>2</sub>DAP), derivata Hidralazina, poznatog leka a za lečenje hipertenzije, kao i njegovih kompleksa formule: [Co<sub>2</sub>(μ-Hp<sub>2</sub>DAP-2H)<sub>2</sub>(μ-O<sub>2</sub>)]·4H<sub>2</sub>O (1), [Ni(Hp<sub>2</sub>DAP-2H)] (2), [Cu(Hp<sub>2</sub>DAP-2H)]·H<sub>2</sub>O (3) i [Zn(Hp<sub>2</sub>DAP-2H)]·H<sub>2</sub>O (4). U reakciji Hp<sub>2</sub>DAP sa Co<sup>II</sup>, ligand se koordinuje u dvostruko deprotonovanom obliku. Pri tome dolazi do oksidacije kobalta i formiranja dimernog kompleksa sa peroksidnim mostom između dva centralna atoma. Sastav i struktura ostalih koordinacionih jedinjenja je određena na osnovu spektroskopskih, konduktometrijskih i termičkih podataka. Utvrđeno je da se ligand koordinuje takođe u dvostruko deprotonovanom obliku, ali su ostali kompleksi monomerne strukture bez koliganda. Egzotermni efekat koji prati termičku dekompoziciju kompleksa cinka je znatno veći od odgovarajućeg efekta koji se javlja kod dekompozicije ostalih kompleksa. Ova pojava zajedno sa visoko konjugvanom krutom strukturom liganda ukazuje na kvadratno-planarni raspored atoma ligatora oko centralnog atoma ne samo u jedinjenjima 2 i 3, već i u kompleksu cinka (4).

*Rad je finansirao Ministarstvo prosvete, nauke i tehnološkog razvoja Republike Srbije (broj projekta ON172014).*

### Synthesis and characterisation of pyridazine derivative and its complexes with Co<sup>III</sup>, Ni<sup>II</sup>, Cu<sup>II</sup> and Zn<sup>II</sup>

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In this paper the synthesis, physical and chemical characterisation of bis(3-chloropyridazine-6-hydrazone)-2,6-diacetylpyridine (Hp<sub>2</sub>DAP) ligand, a derivative of the well known drug Hydralazine, used in antihypertensive therapy, and its complexes of formulae [Co<sub>2</sub>(μ-Hp<sub>2</sub>DAP-2H)<sub>2</sub>(μ-O<sub>2</sub>)]·4H<sub>2</sub>O (1), [Ni(Hp<sub>2</sub>DAP-2H)] (2), [Cu(Hp<sub>2</sub>DAP-2H)]·H<sub>2</sub>O (3) and [Zn(Hp<sub>2</sub>DAP-2H)]·H<sub>2</sub>O (4) are presented. In reaction of Hp<sub>2</sub>DAP and Co<sup>II</sup> ligand coordinates in its double-deprotonated form. At the same time Co<sup>II</sup> is oxidized to Co<sup>III</sup> and a dimeric complex is formed with a peroxide bridge between the cobalt centres. The composition and the structure of the complexes 2–4 were determined on the basis of spectroscopic, conductometric and thermal data. The data refer also to the coordination of the ligand in these compounds in its deprotonated form, but their structure is monomeric without co-ligands. The exothermic effect accompanying the decomposition of zinc complex is significantly higher than the corresponding effect in the other complexes. This fact together with the rigid, highly conjugated aromatic structure of the ligand suggests a square-planar geometry around the central atom not only in the compounds 2 and 3 but also in the complex with zinc (4).

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NIH P 5

## **Ispitivanje stabilnosti u rastvoru i biološka aktivnost kompleksa zlata(III) sa aromatičnim heterocikličnim jedinjenjima koja sadrže azot**

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U ovom radu, primenom UV-Vis spektrofotometrije ispitivana je stabilnost u rastvoru mononuklearnih kompleksa zlata(III) sa nekim diazaaromatičnim jedinjenjima, kao što su piridazin, pirimidin, pirazin, hinoksalin i fenazin, koji su sintetisani po postupku koji je ranije opisan u literaturi [1,2]. Nađeno je da koordinacija ovih liganda stabilizuje +3 oksidaciono stanje zlata, i sprečava njegovu redukciju do Au(I) i Au(0). Imajući u vidu značajnu stabilnost dobijenih kompleksa zlata(III) u rastvoru, ispitivana su njihova antibakterijska svojstva na panelu Gram-pozitivnih i Gram-negativnih mikroorganizama i određena je njihova *in vitro* citotoksičnost na humanim fibroblastima.

## **Solution study and biological activity of gold(III) complexes with aromatic nitrogen-containing heterocycles**

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In the present study, UV-Vis spectrophotometry has been applied to investigate the stability of mononuclear gold(III) complexes with some diazaaromatic ligands, namely pyridazine, pyrimidine, pyrazine, quinoxaline and phenazine, which were synthesized according to the procedure published in the literature [1,2]. It was found that coordination of these ligands stabilizes +3 oxidation state of gold and prevents its reduction to Au(I) and Au(0). Considering sufficient stability of obtained gold(III) complexes with *N*-heterocycles, their antibacterial effect has been studied against the range of Gram-positive and Gram-negative microorganisms and their *in vitro* cytotoxicity against human fibroblasts has been determined.

**Acknowledgements:** *This work was funded in part by the Ministry of Education, Science and Technological Development of the Republic of Serbia (Project No. 172036).*

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## NH P 6

**Zlato(III) kompleks sa *O,O'*-di-*n*-butil-(*S,S*)-etilendiamin-*N,N'*-di-2-(4-metil)pentanoatom – R<sub>2</sub>edda-tipom liganda: sinteza i karakterizacija**

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Kompleksi zlata(III) sa R<sub>2</sub>edda-tip ligandima su već pokazali značajnu *in vitro* anti-tumorsku aktivnost [1,2].

Novi zlato(III) kompleks [AuCl<sub>2</sub>{(*S,S*)-(*n*-Bu)<sub>2</sub>eddl}]PF<sub>6</sub>, ((*S,S*)-(*n*-Bu)<sub>2</sub>eddl = *O,O'*-di-*n*-butil-(*S,S*)-etilendiamin-*N,N'*-di-2-(4-metil)pentanoat), sintetisan je i okarakterisan elementalnom analizom, IC, <sup>1</sup>H i <sup>13</sup>C NMR spektroskopijom i masenom spektrometrijom.

Rezultati elementalne analize odgovaraju prikazanoj formuli. IC spektar pokazuje specifične apsorpcione trake: ν(C=O) na 1736 cm<sup>-1</sup>, ν(C–O) na 1246 cm<sup>-1</sup> i ν(CH<sub>3</sub>) na 2963 cm<sup>-1</sup>. U <sup>1</sup>H NMR spektru etilenski protoni iz etilendiaminskog dela molekula nalaze se na 3,55 ppm, što ukazuje na koordinaciju azotovih atoma.

U <sup>13</sup>C NMR spektru karbonilni ugljenikovi atomi nalaze se na očekivanoj vrednosti hemijskog pomeranja za ovu klasu jedinjenja kod kojih kiseonik ne učestvuje u koordinaciji (170,3 ppm).

**Gold(III) complex with *O,O'*-di-*n*-butyl-(*S,S*)-ethylenediamine-*N,N'*-di-2-(4-methyl)pentanoate – R<sub>2</sub>edda-type ligand: synthesis and characterization**

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Gold(III) complexes with R<sub>2</sub>edda-type ligands have already shown significant *in vitro* antitumor activity [1,2].

A novel gold(III) complex, [AuCl<sub>2</sub>{(*S,S*)-(*n*-Bu)<sub>2</sub>eddl}]PF<sub>6</sub>, ((*S,S*)-(*n*-Bu)<sub>2</sub>eddl = *O,O'*-di-*n*-butyl-(*S,S*)-ethylenediamine-*N,N'*-di-2-(4-methyl)pentanoate), was synthesized and characterized by elemental analysis, IR, <sup>1</sup>H i <sup>13</sup>C NMR spectroscopy and mass spectrometry. Results of elemental analysis are in agreement with the proposed formula. IR spectrum shows specific absorption bands: ν(C=O) at 1736 cm<sup>-1</sup>, ν(C–O) at 1246 cm<sup>-1</sup> and ν(CH<sub>3</sub>) at 2963 cm<sup>-1</sup>. In <sup>1</sup>H NMR spectrum ethylene protons from ethylenediamine moiety are found at 3.55 ppm, indicating nitrogen coordination.

In <sup>13</sup>C NMR spectrum, carbonyl carbon atoms show resonances at expected chemical shifts for this class of compounds where oxygen is not participating in coordination (170.3 ppm).

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NH P 7

## Sinteza i karakterizacija prvih kompleksa Cr(III) sa tiosemikarbazonom piridoksala

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Sa tiosemikarbazonom piridoksala (PLTSC) sintetisan je i okarakterisan veliki broj kompleksa sa većinom 3d metala. S obzirom na to da do sada nisu poznati kompleksi Cr(III) sa ovim ligandom, a uzimajući u obzir biološku važnost kako PLTSC, tako i Cr(III), od interesa je ispitati mogućnost sinteze njihovih kompleksa, te spektralne i strukturne karakteristike dobijenih jedinjenja.

U ovom radu opisane su sinteze prvih kompleksa Cr(III) sa PLTSC, koordinacionih formula  $[\text{Cr}(\text{PLTSC})_2](\text{NO}_3)_3$  (**1**) i  $[\text{Cr}(\text{PLTSC})(\text{NCS})_3]\cdot 2\text{H}_2\text{O}$  (**2**). Kompleksi su dobijeni reakcijom EtOH rastvora  $\text{Cr}(\text{NO}_3)_3\cdot 9\text{H}_2\text{O}$ , odnosno  $\text{K}_3[\text{Cr}(\text{NCS})_6]\cdot 4\text{H}_2\text{O}$  i  $\text{PLTSC}\cdot 3\text{H}_2\text{O}$ , u vidu tamnocrvenih štapićastih monokristala i okarakterisani elementalnom analizom, IR spektrima i konduktometrijskim merenjima, kao i rendgenskom strukturnom analizom. Nađen je uobičajen, tridentatni ONS način koordinacije liganda, preko atoma kiseonika deprotonovane fenolne OH-grupe, atoma azota imino-grupe i atoma sumpora. Deformisano oktaedarsko okruženje Cr(III) u kompleksu **1** realizovano je tridentatnom koordinacijom dva meridijalno raspoređena zwitter-jona PLTSC, dok je u kompleksu **2** Cr(III) u oktaedarskom okruženju zwitter-jona helatnog liganda i jednog  $\text{NCS}^-$  jona u ekvatorijalnoj ravni i dva  $\text{NCS}^-$  jona u aksijalnim položajima.

## Synthesis and characterization of the first Cr(III) complexes with pyridoxal thiosemicarbazone

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Here we present the synthesis and characterization of the first Cr(III) complexes with pyridoxal thiosemicarbazone PLTSC, of the formulas  $[\text{Cr}(\text{PLTSC})_2](\text{NO}_3)_3$  (**1**) and  $[\text{Cr}(\text{PLTSC})(\text{NCS})_3]\cdot 2\text{H}_2\text{O}$  (**2**). Red rod-like single crystals of the complexes were obtained in the reaction of EtOH solutions of  $\text{Cr}(\text{NO}_3)_3\cdot 9\text{H}_2\text{O}/\text{K}_3[\text{Cr}(\text{NCS})_6]\cdot 4\text{H}_2\text{O}$  and  $\text{PLTSC}\cdot 3\text{H}_2\text{O}$ . Complexes were characterized by elemental analysis, IR spectroscopy, conductometry and X-ray analysis. In both complexes ligand is coordinated in the usual tridentate ONS manner, through the phenolic oxygen, imine nitrogen and sulphur atom. In complex **1** a distorted octahedral environment of Cr(III) is achieved by the coordination of two meridional zwitterions of the ligand, while in complex **2** Cr(III) is in octahedral surroundings of the zwitter-ion of PLTSC and one  $\text{NCS}^-$  ion in the basal plane and the other two  $\text{NCS}^-$  ions in the axial positions. The research was supported by the Ministry of Education and Science of the Republic of Serbia (Grant no. 172014).

## Hemija životne sredine / Environmental Chemistry

HŽS P 1

**Raspodela odabranih lekova i pesticida u sistemu voda-sediment**

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Cilj ovog rada bio je ispitivanje načina adsorpcije i raspodele odabranih lekova i pesticida između vodene faze i sedimenta, kao i određivanje stepena adsorpcije i desorpcije na sedimentu reke Dunav. Rezultati adsorpcije leka karbamazepina i pesticida atrazina, karbendazima i karbofurana pokazuju najbolje slaganje sa Frojndlihovom izotermom, što ukazuje na reverzibilnu adsorpciju. Zbog visokog procenta desorpcije karbamazepina, atrazina i karbendazima, njihovo prisustvo u sistemu voda-sediment je očekivano, dok se karbofuran očekuje u manjoj koncentraciji u vodi, zbog nižeg procenta desorpcije. Lengmirov model opisuje monoslojnu adsorpciju pesticida dimetoata. Ovaj pesticid pokazuje i mali stepen desorpcije, što ukazuje na to da se može akumulirati u sedimentima u visokoj koncentraciji. Adsorpcija lekova trimetoprima, metoprolola i lorazepam se može opisati linearnim modelom. Ovi analiti pokazuju veliki afinitet adsorpcije i mogu se vezati za sediment u koncentracijama većim od ispitivanih. Za razliku od metoprolola i lorazepam, trimetoprim se u velikom procentu desorbuje sa sedimenta, pa se ovaj analit očekuje u vodi. Lekovi bromazepam, eritromicin i amlodipin su pokazali skoro potpunu adsorpciju na sedimentu i veoma mali stepen desorpcije, pa je njihova akumulacija u sedimentima očekivana.

**Zahvalnica:** Izradu ovog rada je finansiralo Ministarstvo prosvete, nauke i tehnološkog razvoja Republike Srbije (br. projekta ON 172007).

### **Distribution of selected pharmaceuticals and pesticides in water-sediment system**

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The aim of this study was investigation of the adsorption and distribution of the selected pharmaceuticals and pesticides between water phase and sediment, as well as determination of the adsorption and desorption extent using sediment of the Danube River. Adsorption data of the drug carbamazepine and pesticides atrazine, carbendazim and carbofuran fit well to Freundlich isotherm, implying reversible adsorption. Due to high desorption extent of carbamazepine, atrazine and carbendazim, their presence in water-sediment system is expected, whereas lower desorption level of carbofuran indicates lower expected concentration of this pesticide in water. Langmuir model describes monolayer adsorption of pesticide dimethoate. This pesticide also shows low desorption extent, indicating its high accumulation in sediments. Adsorption data of pharmaceuticals trimethoprim, metoprolol and lorazepam fit fine to the linear model. These analytes show high adsorption affinity and can be adsorbed onto sediment at concentrations higher than tested. Unlike metoprolol and lorazepam, trimethoprim exhibits higher desorption level, so it is expected in water. Pharmaceuticals bromazepam, erythromycin and amlodipine displayed almost complete adsorption onto sediment and extremely low desorption rate, so their accumulation in sediments is expected.



HŽS P 2

**Linearno modelovanje podeonog koeficijenta vazduh-voda ( $\log K_{AW}$ ) primenom tipične normalno-fazne tankoslojne hromatografije**

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Vazduh-voda podeoni koeficijent ( $K_{AW}$ ) u značajnoj meri određuje sudbinu jedinjenja u životnoj sredini. Eksperimentalno određivanje ove veličine skopčano je sa brojnim poteškoćama, pre svega u slučaju površinski aktivnih i supstanci sa niskim vrednostima  $\log K_{AW}$  ( $< -10$ ). U okviru ovog rada ispitana je primena jednostavnije, tankoslojno-hromatografske tehnike za određivanje  $K_{AW}$  vrednosti za 53 jedinjenja niske molekulske mase (94 – 325). Dvanaest normalno-faznih sistema je primenjeno na skupu molekula različitog oblika, veličine, različite sposobnosti da stupaju u interakcije sa stacionarnom fazom, te širokim opsegom rastvorljivosti u vodi ( $-8,06 < \log S < 0,70$ ). Retencija izmerena na cijano-, amino-modifikovanom i nemodifikovanom silika gelu, modelovana je sa literaturno preuzetim  $\log K_{AW}$  vrednostima: univarijantnom linearnom regresijom, regresijom glavnih komponenti (PCR) i metodom delimičnih najmanjih kvadrata (PLS), a rezultati su upoređeni sa kompjuterski izračunatim vrednostima (fragmentacione i kvantno-mehaničke metode). Najbolje procene dobijene su primenom PLS i PCR tehnika u modelovanju retencije na nemodifikovanom silika-gelu, dok cijano- i naročito amino-modifikovani sorbenti ne pokazuju zadovoljavajuće rezultate.

**Linear modeling of the air-water partition coefficient ( $\log K_{AW}$ ) by typical normal-phase thin-layer chromatography**

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Air-to-water partition coefficient ( $K_{AW}$ ) determines, to a large extent, the fate of the compounds in the environment. Experimental determination of this property is fraught with many difficulties, especially in the case of surface-active substances, and those with low  $\log K_{AW}$  values ( $< -10$ ). In the present work we have examined the application of simpler thin-layer chromatographic technique to determine  $\log K_{AW}$  for 53 compounds of low molecular weight (94 to 325). Compounds have been selected to cover the molecules of different shape, size, various ability to interact with the stationary phase, and wide range of water solubility ( $-8.06 < \log S < 0.70$ ). Retention measured on cyano, amino-modified and bare silica gels (in total 12 normal-phase chromatographic systems) was modeled with experimentally determined  $\log K_{AW}$  values obtained from the literature. Univariate linear regression, principal component regression (PCR) and partial least squares (PLS) were used for modeling and obtained results were compared with computational approaches (fragmentation and quantum-mechanical methods). The best estimates were obtained using the PLS and PCR techniques in modeling retention on unmodified silica gel. However, cyano, and particularly amino-modified sorbents do not provide values of satisfying quality.

**HŽS P 3**

**Uticaj načina određivanja na merenu vrednost fosfata u vodama**

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Prisustvo fosfata u otpadnim vodama koje se ispuštaju u životnu sredinu je jedan od osnovnih uzroka eutrofikacije voda. Određivanje fosfata se izvodi u otpadnim vodama pre i posle obrade, kao i u recipijentu. Za njihovo određivanje postoji više metoda. S obzirom da je fosfor u vodama prisutan u više oblika, predtretmanom uzoraka je potrebno sve te oblike prevesti u ortofosfate. U ovom radu je za predtretman više uzoraka model vode i otpadne vode (džibre) korišćena mokra digestija sa sumpornom i azotnom kiselinom i suva digestija (spaljivanje uzorka). U dobijenim uzorcima su ukupni fosfati određivani u više ponavljanja (1) metodom sa amonijum molibdatom, kalijum antimonil tartaratom i askorbinskom kiselinom i merenjem boje na spektrofotometru ili Hellige komparatoru i (2) metodom sa amonijum molibdatom i amonijum monovanadatom i merenjem boja na fotoelektričnom fotometru. Ustanovljeno je da način prethodne pripreme uzoraka model voda i otpadnih voda nije imao uticaj na rezultate određivanja fosfata. Statističkom analizom rezultata je ustanovljeno da je spektrofotometrijska metoda određivanja tačna i precizna. Određivanje na Hellige komparatoru je relativno precizno ali manje tačno, dok je određivanje na fotoelektričnom fotometru i manje precizno i manje tačno.

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**The influence of the determination method on the measured value of phosphates in waters**

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The presence of phosphates in wastewater discharged into the environment is one of the main causes of water eutrophication. Determination of phosphates is performed in wastewater before and after the treatment, as well as in the recipient. There are a number of methods for their determination. Since phosphorus is present in water in various forms, pretreatment of the sample is necessary in order to convert these forms into orthophosphates so its total content in water can be determined. In this paper, as the pretreatment of the various samples of model water and wastewater (stillage) wet digestion with sulfuric and nitric acid and dry digestion (burning of the sample) were performed. Total content of phosphates was determined in more reps by (1) method with ammonium molybdate, potassium antimony tartrate and ascorbic acid and measuring the color by the spectrophotometer or Hellige comparator and by (2) method with ammonium molybdate and ammonium monovanadate measuring the color by the photoelectric photometer. It was found that the method of prior water samples preparation had no effect on the results of the determination of phosphates. Statistical analysis of results showed that spectrophotometric method was accurate and precise for determination of phosphates. Determination by the Hellige comparator is relatively precise but less accurate, while the determination by photoelectric photometer was less accurate and less precise.

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HŽS P 4

## **Uticaj dodatka cementa na stabilizaciju/solidifikaciju elektropećne prašine u geopolimere na bazi pepela**

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Elektropećna prašina (ELP) je stabilizovana/solidifikovana u geopolimere na bazi lebdećeg pepela (LP) sa različitim udjelom Portland Cementa (OPC). Razmatran je uticaj dodatka OPC na pritisnu čvrstoću efikasnost imobilizacije teških metala iz ELP u geopolimere na bazi pepela. Ustanovljeno je da se čvrstoća geopolimera povećava sa povećanjem sadržaja OPC u mješavini od 10-20 %. Najveća vrijednost čvrstoće je dostignuta pri sadržaju OPC u mješavini od 20 %. Dalje povećanje sadržaja OPC dovodi do slabljenja geopolimerne structure. Sa druge strane, najefikasnija imobilizacija teških metala je postignuta pri najnižem sadržaju OPC u mješavini od 10 %.

## **Effect of cement addition on the stabilization/solidification of electric arc furnace dust in the fly ash based geopolymers**

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Electric arc furnace dust (EAFD) was stabilized/solidified in fly ash (FA) based geopolymers with different portions of ordinary Portland cement (OPC). The effect of OPC addition on the compressive strength and immobilization efficacy of heavy metals from EAFD in the FA based geopolymers was evaluated. For an increasing OPC in the mixture in the range of 10-20 %, the strength of geopolymers was found to increase and reach the highest value when the starting mixture contains no more than 20 % OPC. Further increase of OPC content leads to weakening of geopolymer structure. On the other hand, the best immobilization efficacy of heavy metals was achieved at the lowest OPC content of 10 %.

HŽS P 5

**Ispitivanje teratogenog efekta sintetisanog ditiokarbamata, K-daap, na zebra ribama (*Danio rerio*)**

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Ditiokarbamati (DTC) se dugo koriste u poljoprivredi pa je živi svijet već dugo izložen njihovom uticaju. Mehanizam njihovog djelovanja na kičmenjake se još uvijek ispituje. U ovom radu smo ispitivali toksičnost sintetisanog ditiokarbamata, Kdaap (kalijum-3-ditiokarboksi-3-aza-5-amino pentanoat), prema zebra ribama. Promjene u razvoju embriona zebrića, pod uticajem različitih koncentracija vodenog rastvora Kdaap posmatrani su u toku 96 sati i upoređivane sa negativnom kontrolom. Kod većih koncentracija Kdaap, embrioni *Danio rerio* su nakon 48 sati razvijali deformitete nekompatibilne sa životom (nedostatak rada srca i formiranja funkcionalnog krvotoka); U koncentraciji 0,1 g/l razvoj je prolazio bez vidljivih deformiteta embriona, ali je izlijeganje ebriona kasnilo od 12 do 24 h u odnosu na negativnu kontrolu. Ispitivani Kdaap je na većim koncentracijama, imao sličan efekat kao neki DTC opisani u literaturi, gdje je pokazano da oni izazivaju promjene u ekspresiji gena koji igraju bitnu signalnu ulogu u koordinaciji transkripcije povezane sa kraniofacijalnim razvojem kod *Danio rerio* (Yan et al., 2002). Međutim, dominantna posledica djelovanja nižih koncentracija Kdaap, su različiti nivoi poremećaja u razvoju somita što je, pretostavljamo, takođe, izazvano poremećajem u regulaciji genske transkripcije.

**Testing teratogenic effect of synthesized dithiocarbamate, K-daap, on zebra fish (*Danio rerio*)**

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Dithiocarbamates (DTC) is widely used in agriculture and living species have been exposed for quite some time to these compounds. The mechanism of their effects on the vertebrates are still being examined.

In the present study we examined the toxicity of synthesized dithiocarbamate, Kdaap (potassium 3-dithiocarboxy-3-aza-5-amino-pentanoate), on zebrafish. Changes in the development of the zebrafish embryo, under the influence of different concentrations of aqueous Kdaap solution, were observed during 96 hours, and compared with a negative control.

At higher concentrations of Kdaap, *Danio rerio* embryos developed within 48 hours deformities incompatible with life (no heartbeat, nor the formation of the functional blood flow). At a concentration of 0.1 g/L development passes without visible deformation of the embryo, but embryos hatching was delayed 12 to 24 h with respect to control.

The tested Kdaap at higher concentrations had a similar effect as DTC described in the literature, where it is shown that they induce changes in gene expression which play an important signaling role in coordinating transcription associated with craniofacial development in the zebrafish *Danio rerio* (Yan et al., 2002). However, dominant effects of Kdaap, present at low concentrations are disorders in development of somites, assumed also to be caused by disorders of gene transcription.

HŽS P 6

## Uticaj temperature kalcinacije TiO<sub>2</sub> nanoprahova dopiranih La(III) na efikasnost fotokatalitičke razgradnje metoprolola i propranolola

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Pri ispitivanju uticaja temperature kalcinacije na fotokatalitičku efikasnost TiO<sub>2</sub> nanoprahova dopiranih La(III) kao prekursor za sol-gel sintezu katalizatora korišćen je TiCl<sub>4</sub>, za podešavanje pH pri hidrolizi NH<sub>4</sub>OH, za alkogenolizu etanol, dok je temperatura kalcinacije varirana u intervalu od 450 do 750 °C. Katalizatori su dopirani primenom lantan(III)-hlorida heptahidrata, pri čemu je sadržaj La(III) iznosio 1 mol%. Primenom UVA zračenja ispitana je fotokatalitička aktivnost sintetisanih nanoprahova u razgradnji metoprolola (MET) i propranolola (PRO), β-blokatora koji se koriste u lečenju različitih kardiovaskularnih bolesti. Rezultati su upoređeni sa nedopiranim TiO<sub>2</sub> nanoprahom sintetisanim pod istim uslovima i komercijalno dostupnim katalizatorom TiO<sub>2</sub> Degussa P25. Na osnovu dobijenih rezultata je zaključeno da temperatura kalcinacije i dopiranje sa La(III) imaju značajan uticaj na fotokatalitičku aktivnost. Dopirani nanoprah kalcinisan na temperaturi od 650 °C se pokazao kao najefikasniji u razgradnji MET-a, dok se u razgradnji PRO-a kao najefikasniji pokazao dopirani nanoprah kalcinisan na 450 °C.

## Effect of calcination temperature of La(III)-doped TiO<sub>2</sub> nanopowders on the efficiency of photocatalytic degradation of metoprolol and propranolol

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The effect of varying the calcination temperature in the range from 450 to 750 °C on the photocatalytic efficiency of sol-gel synthesized La(III)-doped TiO<sub>2</sub> nanopowders with TiCl<sub>4</sub> as a precursor, using NH<sub>4</sub>OH for pH adjustment during hydrolysis and ethanol for alcogel production, has been investigated. Lanthanum(III) chloride heptahydrate was used to obtain TiO<sub>2</sub> nanopowders doped with 1 mol% of La. Photocatalytic activity of the synthesized catalysts in the degradation of metoprolol (MET) and propranolol (PRO), β-blockers usually used for the treatment of different cardiovascular disorders, was investigated. The results obtained were compared to the efficiency of undoped TiO<sub>2</sub> catalyst synthesized under the same conditions, as well as to the efficiency of commercially available TiO<sub>2</sub> Degussa P25. It was concluded that the calcination temperature and doping with La(III) ions are very important for photocatalytic activity. The doped catalyst calcinated at 650 °C appeared to be most efficient for degradation of MET, while the catalyst calcinated at 450 °C showed to be the most efficient for degradation of PRO.

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HŽS P 7

**Predviđanje BPK vrednosti u reci Dunav u Republici Srbiji primenom optimizovane veštačke neuronske mreže**

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U ovom radu opisana je primena modela zasnovanog na veštačkim neuronskim mrežama za predviđanje vrednosti biološke potrošnje kiseonika (BPK) u reci Dunav prateći tok reke kroz Republiku Srbiju. Za razvoj modela korišćeno je 18 ulaznih parametara koji opisuju kvalitet vode dostupnih sa mernih stanica Agencije za zaštitu životne sredine. Optimizacija modela vršena je na osnovu prostorne raspodele mernih tačaka i dostupnih podataka. Performanse modela ispitivane su pomoću korena srednje kvadratne greške (*the root mean squared error - RMSE*). Najbolji rezultati postignuti su podelom toka reke na dva segmenta: prvi koji obuhvata tok od ulaska u Republiku Srbiju do grada Novog Sada (RMSE=0,81 mgO<sub>2</sub>/l), i drugi koji predviđa vrednosti nivoa BPK od grada Novog Sada do granice sa Rumunijom (RMSE=0,75 mgO<sub>2</sub>/l).

**Predicting BOD levels in the River Danube in Serbia using optimized Artificial Neural Network**

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This study describes the application of Artificial Neural Network models for predicting biological oxygen demand (BOD) levels in the Danube River, following the river stretch passing through Serbia. For the purpose of model development, 18 water quality input variables monitored regularly by the Serbian Environmental Protection Agency were used. The model was optimized according to the spatial distribution of monitoring points and available data. Model performance was evaluated by the root mean squared error (RMSE). The best results were achieved when the river stretch was divided into two parts: one model to cover the Danube from the country border to the city of Novi Sad (RMSE=0.81 mgO<sub>2</sub>/l), and the other one to predict BOD levels from the city of Novi Sad to the border with Romania (RMSE=0.75 mgO<sub>2</sub>/l).

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## Heterogena fotokatalitička razgradnja tiakloprida primenom novosintetisanih $\text{WO}_3/\text{Fe}_3\text{O}_4$ nanoprahova

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Koprecipitacionom metodom sintetisana su četiri  $\text{WO}_3/\text{Fe}_3\text{O}_4$  nanopraha sa različitim masenim odnosom  $\text{WO}_3$  prema  $\text{Fe}_3\text{O}_4$  (0,5%, 2%, 7% i 11%). Ispitana je fotokatalitička efikasnost ovih nanoprahova u fotorazgradnji insekticida tiakloprida u prisustvu UVA ( $I_{\text{UVA}} = 3,57 \times 10^{-3} \text{ W/cm}^2$ ), kao i simuliranog sunčevog zračenja ( $I_{\text{UVA}} = 1,8 \times 10^{-4} \text{ W/cm}^2$ ;  $I_{\text{vis}} = 16,2 \times 10^{-3} \text{ W/cm}^2$ ). Kinetika fotokatalitičke razgradnje tiakloprida je praćena primenom HPLC–DAD. Pre svega je određen optimalni maseni odnos  $\text{WO}_3$  prema  $\text{Fe}_3\text{O}_4$ , kao i optimalna temperatura kalcinacije, a zatim je primenom ovog heterogenog sistema proučavana kinetika razgradnje tiakloprida u širokom opsegu koncentracija fotokatalizatora i vodonik-peroksida (heterogeni foto-Fenton), kao i pH. U prisustvu simuliranog sunčevog zračenja primenom heterogenog foto-Fentonskog sistema koji uključuje  $7\text{WO}_3/\text{Fe}_3\text{O}_4/\text{H}_2\text{O}_2$  efikasnost razgradnje tiakloprida je bila 2,2 puta veća u poređenju sa nekuplovanim  $\text{Fe}_3\text{O}_4/\text{H}_2\text{O}_2$  sistemom.

## Heterogeneous photocatalytic degradation of thiacloprid by applying newly synthesized $\text{WO}_3/\text{Fe}_3\text{O}_4$ nanopowders

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Four  $\text{WO}_3/\text{Fe}_3\text{O}_4$  nanopowders with different mass ratios of  $\text{WO}_3$  to  $\text{Fe}_3\text{O}_4$  (0.5, 2, 7, and 11%) were synthesized via the chemical co-precipitation method. The photocatalytic efficiency of these nanopowders in the photodegradation of thiacloprid insecticide in the presence of UVA ( $I_{\text{UVA}} = 3.57 \times 10^{-3} \text{ W/cm}^2$ ), as well as simulated solar radiation ( $I_{\text{UVA}} = 1.8 \times 10^{-4} \text{ W/cm}^2$ ;  $I_{\text{vis}} = 116.2 \times 10^{-3} \text{ W/cm}^2$ ) was investigated. The kinetics of the photocatalytic degradation of thiacloprid was monitored by HPLC–DAD. Optimum mass ratio of  $\text{WO}_3$  vs.  $\text{Fe}_3\text{O}_4$ , and optimum calcination temperature were determined. This heterogeneous system was then applied to examine the kinetics of thiacloprid degradation in a wide range of concentrations of the photocatalyst and hydrogen peroxide (heterogeneous photo-Fenton), as well as of pH. The degradation efficiency of thiacloprid in the presence of simulated solar radiation was 2.2 times greater using heterogeneous photo-Fenton system that includes  $7\text{WO}_3/\text{Fe}_3\text{O}_4/\text{H}_2\text{O}_2$  than the uncoupled  $\text{Fe}_3\text{O}_4/\text{H}_2\text{O}_2$  system.

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HŽS P 9

**Anaerobni tretman džibre iz proizvodnje etanola**

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Izvršen je anaerobni tretman džibre nastale u procesu proizvodnje etanola, sa visokim sadržajem organskih materija, kao prve faze prečišćavanja otpadnih voda industrije alkohola. Tretman je rađen u laboratorijskom anaerobnom reaktoru UASB tipa sa mešanjem zapremine 12 dm<sup>3</sup> koji radi neprekidno 320 dana. U toku tretmana praćene su karakteristike influenta (džibra) i efluenta (hemijska potrošnja kiseonika - HPK, ukupna, organska i neorganska suva masa), zapremina nastalog biogasa kao i parametri procesa (organsko opterećenje, hidraulično vreme zadržavanja, pH i temperatura). Tretman je bio pri mezofilnim uslovima (t = 43 °C), pH je bio 7,5 - 7,7, organsko opterećenje 3 – 6 g dm<sup>-3</sup> dan<sup>-1</sup> dok je hidraulično vreme zadržavanja bilo 15 – 25 dana. Pri ovakvim parametrima procesa ostvarena je visoka efikasnost odstranjivanja HPK od 85 – 95% (prosečno 87,5%), dok je odstranjivanje suve mase bilo 83%, organske suve mase 89% a neorganske 53%. Produkcija biogasa je bila prosečno 21,4 dm<sup>3</sup> dan<sup>-1</sup> odnosno 0,32 dm<sup>3</sup> po gramu odstranjenog HPK, dok je prosečni udeo metana u biogasu bio 58%. Može se zaključiti da je anaerobni tretman džibre pogodan kao prva faza prečišćavanja otpadnih voda industrije alkohola.

**Anaerobic treatment of stillage from ethanol production**

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Anaerobic treatment of stillage from ethanol production, with a high content of organic matter, as the first stage of wastewater treatment of alcohol industries was carried out. The treatment was performed in a laboratory anaerobic reactor of the UASB type with mixing a volume of 12 dm<sup>3</sup>, running continuously for 320 days. During treatment the following was monitored: characteristics of the influent (stillage) and effluent (chemical oxygen demand - COD, total, organic and inorganic dry matter), the volume of produced biogas and the process parameters (organic loading rate, hydraulic retention time, pH and temperature). Treatment was at mesophilic conditions (t = 43 °C), pH was 7.5 - 7.7, organic loading rate: 3 - 6 g dm<sup>-3</sup> day<sup>-1</sup> while hydraulic retention time was 15 - 25 days. At these process parameters high efficiency of COD removal of 85 - 95% (average 87.5 %) was achieved, while the efficiency of removal of total dry matter was 83 %, organic dry matter 89%, and inorganic dry matter 53%, respectively. Average production of biogas was 21.4 dm<sup>3</sup> day<sup>-1</sup> or 0.32 dm<sup>3</sup> per gram of removed COD, while the average proportion of methane in the biogas was 58%. It can be concluded that the anaerobic treatment of stillage is suitable as a first stage of wastewater treatment in alcohol industries.



HŽS P 10

### **Aktivnost TiO<sub>2</sub>/polianilin nanokompozitnih katalizatora u fotorazgradnji vodene suspenzije sulcotriona primenom UVA zračenja**

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Sulcotrion {2-[2-hlor-(4-metilsulfonil)benzoil]-1,3-cikloheksandion,  $M_r = 328,77$ , CAS No 99105-77-8, C<sub>14</sub>H<sub>13</sub>ClO<sub>5</sub>S} je herbicid iz grupe triketona koji se koristi za kontrolu širokog spektra trava i širokolisnih korova prilikom uzgajanja kukuruza. Razvijen je od strane kompanije Zeneca Ag Products i registrovan za korišćenje 1993. godine u Francuskoj. Fotokatalitička aktivnost nanočestičnog čistog TiO<sub>2</sub>, odnosno kompozita TiO<sub>2</sub>/polianilin u različitim molskim odnosima (TP-50, TP-100 i TP-150) je ispitana na primeru razgradnje sulcotriona primenom UVA zračenja. Rezultati su upoređeni sa komercijalno dostupnim katalizatorima TiO<sub>2</sub> Degussa P25 i TiO<sub>2</sub> Hombikat. Dobijeni rezultati ukazuju na to da je u UVA oblasti najveću fotokatalitičku aktivnost pokazao nosintetisani čist nanočestični katalizator TiO<sub>2</sub>. Za ovaj sistem su urađena i TOC merenja koja su pokazala da je mineralizacija sporija u poređenju sa brzinom razgradnje polaznog jedinjenja.

### **Activity of TiO<sub>2</sub>/polyaniline nanocomposite powders in photodegradation of aqueous suspension of sulcotrione using UVA light**

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Sulcotrione {2-(2-chloro-4-(methylsulfonyl)benzoyl)cyclohexane-1,3-dione,  $M_r = 328.77$ , CAS No 99105-77-8, C<sub>14</sub>H<sub>13</sub>ClO<sub>5</sub>S} is a herbicide from the family of triketone for control of a broad spectrum of grass weeds and broad-leaved weeds in corn farming. It was developed by the company Zeneca Ag Products and registered for use in France in 1993. The photocatalytic activity of neat TiO<sub>2</sub> and TiO<sub>2</sub>/polyaniline nanocomposite powders synthesized in different mole ratios (TP-50, TP-100, and TP-150) was investigated in the degradation of sulcotrione by UVA light. These results were compared with those obtained using TiO<sub>2</sub> Degussa P25 and TiO<sub>2</sub> Hombikat. The results obtained indicate that in the UVA region the highest photocatalytic activity is shown by the neat TiO<sub>2</sub> nanocomposite powder. For this system TOC measurements were carried out, and the results showed that mineralization is slower compared to the rate of decomposition of the starting compound.

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HŽS P 11

**Efikasnost brukitnih TiO<sub>2</sub> nanoprahova u fotokatalitičkoj razgradnji alprazolama primenom UVA zračenja**

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Titan(IV)-oksid (TiO<sub>2</sub>) je poznat kao polimorfni materijal sa tri prirodne kristalne modifikacije: anatas, rutil i brukit, od kojih se brukit najređe javlja u prirodi. Dve serije brukitnih TiO<sub>2</sub> nanoprahova sintetisane su primenom kombinovane sol–gel–hidrotermalne metode sa titan(IV)-hloridom kao prekursorom, variranjem hidrotermalne temperature u opsegu od 120 do 200 °C i reakcionog vremena od 12 do 48 h. Uticaj hidrotermalne temperature i vremena reakcije na fotokatalitičku aktivnost nanoprahova (1,0 mg/cm<sup>3</sup>) ispitan je na primeru fotokatalitičke razgradnje alprazolama (0,03 mmol/dm<sup>3</sup>), anksiolitika iz grupe benzodiazepina, pod uticajem UVA zračenja. Dobijeni rezultati su upoređeni sa efikasnošću komercijalno dostupnog katalizatora TiO<sub>2</sub> Degussa P25. Od svih sintetisanih uzoraka, nanoprah čistog brukita, dobijen na 200 °C tokom 24 h, pokazao je najveću efikasnost u fotorazgradnji alprazolama, praktično istu kao aktivnost katalizatora TiO<sub>2</sub> Degussa P25.

**Efficiency of brookite type TiO<sub>2</sub> nanopowders in photocatalytic degradation of alprazolam using UVA irradiation**

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Titanium(IV) oxide (TiO<sub>2</sub>) is known as a polymorphic material with three naturally occurring crystalline modifications: anatase, rutile and brookite, with brookite as the rarest one. Two series of brookite-type TiO<sub>2</sub> nanopowders have been synthesized by using combined sol–gel–hydrothermal method with titanium(IV) chloride as a precursor, varying hydrothermal temperature in the range from 120 to 200 °C and reaction time from 12 to 48 h. The influence of the hydrothermal temperature and reaction time on photocatalytic activity of produced nanopowders (1.0 mg/cm<sup>3</sup>) has been studied through the photocatalytic degradation of alprazolam (0.03 mmol/dm<sup>3</sup>), anxiolytic of benzodiazepines class, under UVA light irradiation. The results obtained are compared with the efficiency of the commercially available TiO<sub>2</sub> Degussa P25. Among all synthesized samples, pure brookite nanopowder produced at 200 °C for 24 h, has shown the highest efficiency in the photodegradation of alprazolam, practically equal to the activity of TiO<sub>2</sub> Degussa P25.

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## **Statistički značajne razlike emisije NO<sub>2</sub> u okruženju fotokopirnice**

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Istraživanje je obuhvatilo praćenje koncentracionih varijacija azot(IV) oksida tokom procesa fotokopiranja. Petodnevna merenja su sprovedena u dve fotokopirnice u Novom Sadu. U cilju utvrđivanja postojanja statistički značajne razlike emisije azot(IV) oksida među mernim mestima u ispitivanim fotokopirnicama korišćen je T test za nezavisne uzorke. Rezultati su pokazali da merno mesto A doprinosi statistički značajnoj emisiji azot(IV) oksida u obe fotokopirnice. Dobijeni rezultati su dalje poslužili za izračunavanje CO<sub>2</sub> ekvivalenta. Koncentraciona varijacija azot(IV) oksida i njegov CO<sub>2</sub> ekvivalent su pokazali suprotan trend u obe fotokopirnice. Dobijeni rezultati mogu biti korisni za procenu izloženosti azot(IV) oksidu i kreiranje ekološke stope NO<sub>2</sub> u radnom prostoru fotokopirnice u Republici Srbiji.

## **The statistically significant differences of NO<sub>2</sub> emissions in the photocopying environment**

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The investigation included monitoring of nitrogen dioxide concentration variations during the photocopying process. The five-day measurements were conducted in two photocopying shops in Novi Sad. In order to determine the existence of statistically significant differences of nitrogen dioxide emissions between the sampling points an independent-samples T test was performed. The results showed that sampling point A contributes to statistically significant emission of nitrogen dioxide in both photocopying shops. The obtained results are further used for calculation of CO<sub>2</sub> equivalents. Variation of nitrogen dioxide concentration and its CO<sub>2</sub> equivalent showed the opposite trend in both photocopying shops. The obtained results may be useful for the exposure assessment to nitrogen dioxide, as well as for the creation of NO<sub>2</sub> footprint guidelines in photocopying environment of the Republic of Serbia.

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## HŽS P 13

**Uloga mikrofiltracije u završnoj obradi otpadnih voda industrije šećera**

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Industrija šećera, kao grana prehrambene industrije je veoma razvijenu u Srbiji, a poznato je da se otpadne vode šećerana bez prečišćavanja ne mogu puštati u vodotokove zbog velikog opterećenja. Skoro 99% upotrebjene vode izlazi iz fabrike kao otpadna voda. Prema preporukama Najboljih dostupnih tehnika (BAT - Best Available Techniques) izdate od strane EU, mikro- i ultrafiltracija su jedna od mogućih rešenja za finalno prečišćavanje otpadnih voda industrije šećera. U ovom radu će biti razmatrana mogućnost primene keramičkih cevni membrana sa otvorima pora veličine od 0,2  $\mu\text{m}$  (proizvođača GEA, Nemačka) radi smanjenja hemijske potrošnje kiseonika (HPK) otpadne vode industrije šećera. Time bi se kao permeat dobila voda sa smanjenom HPK i bez mutnoće, koja bi se mogla recirkulirati u proces. Pri mikrofiltraciji otpadne vode pri transmembranskom pritisku u opsegu od 1-3 bara, protoku od 50 do 150 l/h, može se zaključiti da se mikrofiltracijom otpadne vode industrije šećera mogu smanjiti suspendovane čestice za 99%, električna provodljivost za 20%, i mutnoća permeata sa 573 NTU na 0,32 NTU, pri čemu se postiže fluks od 30 l/m<sup>2</sup>h.

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**The role of microfiltration in sugar industry wastewater treatment**

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Sugar industry, as a part of food industry, is highly developed in Serbia and it is known that the sugar refinery wastewater cannot be let out into water recipients without treatment, due to the high load of suspended solids, turbidity. Almost 99% of the used water, comes out from the factory as wastewater. According to the Best Available Techniques (BAT) issued by EU, micro- and ultrafiltration are one of the possible solution for the final treatment of sugar industry wastewater. In this work the use of the ceramic tubular membranes with 0.2  $\mu\text{m}$  pore sizes (manufacturer GEA, Germany) are investigated in order to reduce the chemical oxygen demand (COD) of the sugar industry wastewater. As a result, the permeate would have reduced COD and turbidity, which could be recirculated in the factory. After microfiltration of the wastewater at transmembrane pressure in the range of 1-3 bars and at a flow rate of 50 – 150 L/h, it could be concluded that suspended solids are reduced by 99%, electric conductivity by 20%, as well as the turbidity decreased from 573 NTU to 0.32 NTU, with an achieved flux of 30 L/m<sup>2</sup>h.

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## Uticaj O<sub>3</sub>/UV unapređenog procesa oksidacije na sadržaj prekursora trihalometana u vodi

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Trihalometani (THM) su halogenovani ugljovodonici koji se u najvećoj meri formiraju u vodi nakon hlorisanja, usled reakcije hlora sa prisutnim prirodnim organskim materijama (POM). U ovom radu ispitivan je uticaj procesa UV-C fotolize (600-6000 mJ/cm<sup>2</sup>), ozonizacije (0,5-3,0 mg O<sub>3</sub>/mg DOC) i njihove kombinacije O<sub>3</sub>/UV (0,5-3,0 mg O<sub>3</sub>/mg DOC; 600-6000 mJ/cm<sup>2</sup>) na sadržaj prekursora THM u vodi nakon hlorisanja. Sirovu vodu karakterišu vrednosti: UV apsorpcija na 254 nm (UV<sub>254</sub>) od 0,2011 cm<sup>-1</sup>, sadržaj rastvorenog organskog ugljenika (DOC) od 5,15 mg C/l, bromid 31,0 µg Br/l i potencijal formiranja trihalometana (PFTHM) od 279,5 µg/l. U poređenju sa pojedinačnim tretmanima UV-C fotolizom i ozonizacijom, njihova kombinacija (O<sub>3</sub>/UV) pokazuje veću efikasnost u uklanjanju UV<sub>254</sub> apsorbujućih organskih materija (42-82%) i potencijala formiranja THM (23-50%) u vodi, a najbolji rezultati se postižu primenom najveće ispitivane doze ozona i UV zračenja. U poređenju sa ozonizacijom, potencijal formiranja hidroksil radikala usled O<sub>3</sub>/UV je mnogo veći. Hidroksil radikali omogućavaju oksidaciju skoro svih redukujućih materija prisutnih u vodi i kombinovani O<sub>3</sub>/UV proces rezultira značajnom mineralizacijom organske materije i smanjenjem PFTHM.

## The effect of O<sub>3</sub>/UV advanced oxidation process on the content of trihalomethane precursors in water

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Trihalomethanes (THMs) are halogenated hydrocarbons and are mostly formed in water after chlorination, when chlorine reacts with the present natural organic matter (NOM). This study investigated the impact of UV-C photolysis (600-6000 mJ/cm<sup>2</sup>), ozonation processes (0.5-3.0 mg O<sub>3</sub>/mg DOC) and their combination O<sub>3</sub>/UV (0.5-3.0 mg O<sub>3</sub>/mg DOC; 600-6000 mJ/cm<sup>2</sup>) on the content of THM precursors in water during chlorination. Raw water is characterized by values: UV absorbance at 254 nm (UV<sub>254</sub>) of 0.2011 cm<sup>-1</sup>, dissolved organic carbon (DOC) content of 5.15 mg C/l, bromide 31.0 µg Br/l and forming potential of trihalomethanes (THMFP) 279.51 µg/l. Compared with UV-C photolysis and ozone alone, their combination (O<sub>3</sub>/UV) shows greater effectiveness in reduction of UV<sub>254</sub> absorbing organic matter (42-82%) and THM formation potential (23-50%) in water, and the best results were at the highest applied doses of ozone and UV radiation. Compared to ozonation, the hydroxyl radicals formation potential in O<sub>3</sub>/UV is much higher. Hydroxyl radicals are capable of oxidizing almost all reduced materials present in water and the combined O<sub>3</sub>/UV process has been observed to result in a significant mineralization of organic matter and reduction of THMFP.

HŽS P 15

### **Fotokatalitička degradacija dimetenamida-P u morskoj vodi**

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U ovom radu je proučavana fotokatalitička degradacija herbicida dimetenamida-P u morskoj vodi koristeći  $\text{TiO}_2$  kao fotokatalizator. Proces razgradnje je proučavan pod različitim eksperimentalnim uslovima kao što su koncentracija fotokatalizatora, vreme ozračivanja, početna koncentracija dimetenamida-P, pH reakcione suspenzije, efekat dodatnih soli, dodatak elektron akceptora, odnosno hvatača  $\cdot\text{OH}$  radikala. Rezultati su pokazali da je proces razgradnje snažno uslovljen ovim parametrima. Određeni su optimalni uslovi fotokatalize testiranog hloraacetamida i to koncentracija  $\text{TiO}_2$  od  $2 \text{ g/dm}^3$  i sadržaj dimetenamida-P od  $34,5 \text{ mol/dm}^3$ . Fotodegradaciona efikasnost raste sa porastom vremena ozračivanja. Alkalna sredina favorizuje fotokatalitičku degradaciju ovog herbicida. Degradacioni proces se neznatno ubrzava dodatkom male količine  $\text{H}_2\text{O}_2$ , dok dodatak etanola značajno usporava proces.

### **Photocatalytic degradation of dimethenamid-P in sea water**

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In this paper, photocatalytic degradation of herbicide dimethenamid-P was investigated in sea water by using  $\text{TiO}_2$  as a photocatalyst. The degradation was studied by varying the amount of the photocatalyst, illumination time, pH of the system, salt type, electron acceptors and scavengers of  $\cdot\text{OH}$  radicals. The results showed that the photocatalytic degradation of dimethenamid-P was strongly influenced by these parameters. The best conditions for the photocatalytic degradation of dimethenamid-P were obtained. The optimum amount of the photocatalyst used is  $2 \text{ g/dm}^3$  and the concentration of herbicide is  $34,5 \text{ mol/dm}^3$ . The photodegradation efficiency of dimethenamid-P increases with the increase of the illumination time. Alkaline media are favorable for the photocatalytic degradation of dimethenamid-P. The photodegradation efficiency of dimethenamid-P is accelerated by the addition of a small amount of  $\text{H}_2\text{O}_2$ , while the addition of ethanol significantly inhibits the process.

HŽS P 16

### **Anaerobna degradacija ugljovodonika u sedimentu u prisustvu kosupstrata**

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U ovom radu procenjena je mogućnost remedijacije sedimenta visoko opterećenog organskim materijama u anaerobnim uslovima. U cilju stimulacije i optimizovanja uslova za bioremedijaciju ispitivan je potencijal indogenih mikroorganizama, kao i dodatak kosupstrata (u vidu hleba). U toku anaerobnog tretmana praćena je promena hemijske potrošnje kiseonika (HPK), biološke potrošnje kiseonika (BPK), ukupnog organskog ugljenika (TOC) i policikličnih aromatičnih ugljovodonika (PAH). Vrednosti HPK i BPK u toku tretmana fluktuiraju. U probama sa kosupstratom dolazi do pada vrednosti HPK u toku prvih 60 dana tretman, nakon čega dolazi do povećanje vrednosti HPK do kraja eksperimenta kao posledica degradacije razgradnje kompleksnih organskih materija u manje organske molekule koji se mogu oksidovati dihromatom. BPK vrednosti osciluju u toku tretmana u većoj meri u zavisnosti od sadržaja biodegradabilnog supstrata. U tretmanu sa kosupstratom nakon 90 dana i posle 130 dana eksperimenta dolazi do naglog skoka BPK vrednosti. Sadržaj TOC opada u tretmanu sa nativnim sedimentom za oko 8%, dok u uzorcima sa kosupstratom za oko 12%. U pogledu degradacije policikličnih aromatičnih ugljovodonika (PAH), u tretmanu samo sa nativnim sedimentom razgrađeno je oko 61% PAH sumarno, dok je u tretmanu sa kosupstratom razgrađeno je 70,5%.

### **Anaerobic degradation of hydrocarbons from aquatic sediment in the presence of cosubstrate**

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In the present work the ability of indigenous microorganisms to anaerobically degrade organic pollutants in river sediments was investigated. In order to stimulate and optimize the conditions for the biodegradation, the potential of indigenous microorganisms and the addition of cosubstrates (as bread) were investigated. During the anaerobic treatment the change of the chemical oxygen demand (COD), biological oxygen demand (BOD), total organic carbon (TOC) and polycyclic aromatic hydrocarbons (PAH) were monitored. The values of COD and BOD during the treatment fluctuate. In the samples with cosubstrate values of COD led to a decrease during the first 60 days of treatment, but after this period in the sample COD values tended to increase to the end of the experiment, which may be due to decompositions of the complex organic substances to the less structures which can be oxidized after with dichromate. The BOD values oscillated to a greater extent, depending on the content of biodegradable substrate. In the treatment with bread after 90 days and after 130 days of the experiment the BOD values come to an abrupt jump. The content of TOC decreases for about 8% in the native sediment, whereas in the samples with the cosubstrate by about 12%. In terms of degradation of PAH, in the native treatment about 61 % was degraded, while in the treatment with the cosubstrates the degradation ws by about 70.5 %.

## Teorijska hemija / Theoretical Chemistry



TH O 1

**Uticaj proteinskog okruženja na redoks osobine kofaktora:  
Redoks potencijal sintetičkih citohroma b**

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U ovoj studiji je ispitivan uticaj proteinskog okruženja na pomeranje i fino podešavanje redoks potencijala hema, kao kofaktora, što otvara nove mogućnosti u dizajniranju sintetičkih proteina sa željenim osobinama za različite potrebe, kao na primer biosenzora u bioelektronici i bio-nanotehnologiji. Koristili smo kompjutersko "de novo" modelovanje da bi generisali atomske koordinate 25 sintetičkih proteina stukturno sličnih aktivnom centru citohroma b. Stanje protonovanja titratibilnih amino-kiselinskih ostataka i redoks potencijali hema kao kofaktora u tim proteinima su izračunati na osnovu elektrostatičkih energija, rešavanjem Poason-Bolcmanove jednačine. Rezultati izračunavanja se generalno dobro slažu sa eksperimentalnim merenjima. Analizirali smo zavisnost redoks potencijala hema od razlika u proteinskom okruženju. Generalno, na ukupnu vrednost redoks potencijala utiču niska dielektrična konstanta unutar proteina, veliki hidrofobni ostaci, polarni uticaj amidnih veza osnovnog proteinskog niza i jonski mostovi izmedju propionatnih grupa hema i arginina. Razlike u vrednostima redoks potencijala hema su uzrokovane ovim uticajima. Ova studija pokazuje da se savremene metode modelovanja biomolekula mogu uspešno primeniti kako bi se razumele protein-kofaktor interakcije na atomskom nivou. Takodje je pokazano kako se ovaj pristup može koristiti da bi se dizajnirali sintetički proteini sa željenim osobinama.

**Influence of protein environment on redox properties of cofactors:  
Redox potentials of artificial cytochrome b**

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Proteins that have tailored structural and functional properties are needed as central components of biosensors in bioelectronics and bio-nanotechnology. Model building from scratch with advanced modeling techniques is used to generate the atomic coordinates of 25 synthetic cytochrome b proteins. Protonation pattern and redox potentials of heme are evaluated from electrostatic energies by solving the Poisson-Boltzmann equations. The computed redox potentials generally agree well with experimental measurements. The factors of protein environment determining the shift in the redox potentials are elucidated. The dependence of the redox potential of the hemes on protein environment was analyzed. Generally, the total shift in the redox potential is mainly due to the low dielectric medium within the protein, bulky hydrophobic residues, the protein backbone charges and the salt bridges formed between arginines and heme propionate groups. The difference in the shift of the redox potentials is due to the interactions with hydrophilic and charged side chains and salt bridges formed with the heme propionates. Our results open new avenues in design of proteins with desired properties. The present study demonstrates that nowadays methods of modeling and computing of biomolecules can be successfully applied to understand protein-cofactor interactions in atomic detail. It also shows how one can use modeling and computation to design synthetic proteins with desired properties.



## Biohemija / Biochemistry

BH O 1

### **Anti-proliferativna i anti-mikrobna aktivnost ekstrakata kamilice dobijenih pregrejanom vodom**

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Ekstrakcija vodom u zatvorenim ekstrakcionim sistemima je relativno nova ekstrakciona tehnika koja je posebnu popularnost stekla sa aspekta zelene hemije. Temperatura ekstrakcije zavisi isključivo od analita koji se ekstrahuju, s obzirom da se polarnost vode menja sa njenim zagrevanjem u zatvorenom sistemu. Pri zagrevanju vode dolazi do značajnog pada njene polarnosti pri čemu nastaje rastvarač selektivan prema umerno-polarnim jedinjenjima, a takođe dolazi i do pada njenog viskoziteta, gustine i površinskog napona, što sve doprinosi povećanju efikasnosti ekstrakcije. Cilj ovog rada bio je da se ispita biološka aktivnost ekstrakata jezičastih cvetova kamilice dobijenih ekstrakcijom u zatvorenom sistemu koristeći vodu kao ekstragens. Proces je izvođen u trajanju od 20 min na temperaturi od 200°C i pritisku od 3 bar. Kombinovanom hromatografsko-spektrofotometrijskom metodom određen je sadržaj apigenina i apigenin-7-O- $\beta$ -glukozida u dobijenom ekstraktu. Ispitivanje biološke aktivnosti ekstrakata obuhvatalo je evaluaciju anti-proliferativne i anti-mikrobne aktivnosti. Anti-proliferativna aktivnost dobijenih ekstrakata ispitana je na osnovu rezultata njihovog delovanja na rast tri različite ćelijske linije. Anti-mikrobna aktivnost utvrđena je koristeći 8 različitih mikrobnih sojeva.

### **Anti-proliferative and anti-microbial activity of chamomile extracts obtained by superheated water**

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Extraction with water in closed systems is a relatively new extraction technique that has gained special popularity in terms of green chemistry. Extraction temperature depends on the analyte due to change of water polarity with heating. When water is heated there is a significant decrease in its polarity but also decline in its viscosity, density and surface tension which all contribute to the extraction efficiency. The purpose of this study was to investigate biological activity of chamomile ligulate flowers extracts obtained by extraction in a closed system using a water as a solvent. The process was carried out for 20 min at a temperature of 200°C and a pressure of 3 bars. Using a combined chromatographic-spectrophotometric method, the contents of apigenin and apigenin-7-O- $\beta$ -glucoside in chamomile extracts were determined. Determination of biological activity of obtained extracts included the evaluation of anti-proliferative and anti-microbial activity. Anti-proliferative activity of these extracts was investigated on three different cell lines that included. Anti-microbial activity was determined on eight different microbial strains.

BH P 1

### **Karamelizacija fruktoze indukovana ultrazvukom visokog intenziteta i antioksidativna aktivnost karamelizacionih proizvoda**

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Ultrazvuk visokog intenziteta je našao široku primenu u obradi hrane. Cilj ove studije je bio da se ispita karamelizacija indukovana ultrazvukom visokog intenziteta. Rastvor fruktoze je sonikovano na pH 12 i pH 4 tokom 5, 10, 20, 30, 45 i 60 min. Karamelizacija, praćena promenama u apsorbanca u UV oblasti, tamnjenju, pH i sadržaju redukujućih šećera, je rasla sa dužinom vremena sonikacije, i bila je dramatično veća u baznoj, u poređenju sa kiselom sredinom. Na pH 12, tokom prvih 20 minuta sonikacije došlo je do oštih promena u svim ispitivanim parametrima. U baznoj sredini došlo je do pada pH na oko 8, pri čemu se sadržaj redukujućih šećera smanjio na 50%. Moć uklanjanja DPPH (1,1-difenil -2-pikrilhidrazil) radikala, kao i redukujuća moć proizvoda karamelizacije su rasli paralelno sa tamnjenjem i formiranjem intermedijernih degradacionih produkata. Dobijeni rezultati sugerišu da se ultrazvuk visokog intenziteta može primeniti za dobijanje karamelnih aroma, boja i antioksidanata u proizvodnji prehrambenih proizvoda. U poređenju sa konvencionalnim zagrevanjem, ultrazvuk omogućava visoku efikasnost ranih faza karamelizacije, kao i uniforman transfer toplote, čime obezbeđuje mnogo lakšu kontrolu procesa karamelizacije.

### **Caramelization of fructose induced by high intensity ultrasound and antioxidative activity of caramel products**

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High intensity ultrasound is widely used for food processing. The aim of this study was to investigate caramelization induced by high-intensity ultrasound. Fructose was sonicated at pH 12 and pH 4 during 5, 10, 20, 30, 45 and 60 min. Caramelization, monitored by changes in UV absorbance, browning, pH and reducing sugar content, increased with sonication time, and was dramatically higher under alkaline conditions, compared to acidic. At pH 12, during the first 20 min of sonication sharp change of all measured parameters was observed. At basic conditions pH dropped to about 8 and reducing sugar content to 50% due to sonication. DPPH (1,1-diphenyl-2-picrylhydrazyl) scavenging activity and reducing power of caramelization products increased in parallel with browning and intermediate degradation products formation. These results suggest that high-intensity ultrasound can be used for generation of caramel flavours, colours and antioxidants in the food industry. In comparison with conventional heating, ultrasound enables high efficiency of early phases of caramelization, as well as uniform heat transfer providing more controllable caramelization process.



BH P 2

### **Toksičnost cipermetrina (sredstvo za zaštitu bilja Cipkord®) na acetilholin-esterazu i antioksidativne enzime medonosne pčele (*Apis mellifera* L.)**

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Laboratorijski testovi su neophodni kako bi se preciznije opisao uticaj pojedinih zagađujućih materija iz životne sredine na zdravlje pčelinjih kolonija. Cilj ovog istraživanja je ispitivanje efekata preparata Cipkord® (cipermetrin kao aktivna komponenta) na medonosne pčele kroz praćenje aktivnosti enzima antioksidativne zaštite kao i enzima acetilholin-esteraze, biomarkera intoksikacije pesticidima. Subletalni efekti različitih doza ovog pesticida na aktivnost navedenih enzima su ispitani oralnim testom toksičnosti. Statistički značajno povećanje aktivnosti glutation S-transferaze i superoksid dismutaze pri niskim koncentracijama insekticida ukazuju da cipermetrin izaziva indukciju enzimske antioksidativne zaštite kod medonosne pčele. Sa druge strane, visoke doze ovog insekticida smanjuju aktivnost antioksidativnih enzima, što može dovesti do oksidativnog stresa. Pored toga, najviša primenjena doza cipermetrina je inhibirala acetilholinesterazu, što vodi do poremećaja neurotransmisije. Važno je naglasiti da su ovi rezultati dobijeni u kontrolisanim laboratorijskim uslovima, dok u prirodnim populacijama na date parametre može da utiče veći broj faktora kao što su infekcije patogenima i parazitima, zagađujuće materije i drugi.

### **Toxic effects of cypermethrin (Cipkord®) on acetylcholinesterase and antioxidative enzymes activity in honey bee (*Apis mellifera* L.)**

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Laboratory tests are necessary in order to accurately describe the impact of certain pollutants from the environment on the honey bee colony health. The aim of this study was to investigate the effects of Cipkord® insecticide (cypermethrin as an active component) in honey bees by monitoring the activities of antioxidative enzymes and acetylcholinesterase, which is biomarker of pesticide intoxication. The sublethal effects of different doses of pesticide on enzyme activities were evaluated using oral toxicity test. Statistically significant increase in the activity of glutathione S-transferase and superoxide dismutase after exposure to low concentrations of insecticide indicates that cypermethrin causes induction of enzymatic antioxidant protection in honey bees. On the other hand, high doses of insecticides reduce the activity of antioxidant enzymes, which could lead to oxidative stress. In addition, the highest administered dose inhibited acetylcholinesterase leading to disruptions in neurotransmission. It is important to emphasize that these results are obtained in controlled laboratory conditions, while in natural populations they can be modified by multiple factors such as infection of colonies by pathogens or parasites, the impact of various pollutants or many others.

BH P 3

**Vezivanje fikocijanobilina za humani serum albumin**

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Humani serum albumin (HSA) je važan regulator farmakokinetičkih svojstava bioaktivnih jedinjenja. Fikocijanobilin (PCB) je plava tetrapirolna hromofora C-fikocijanina, glavnog proteina cijanobakterije Spiruline, sa dokazanim benefitima na zdravlje čoveka. U ovom radu je po prvi put okarakterisano vezivanje PCB za HSA i ispitana kompeticija sa strukturnim analogom bilirubinom (BR). Metode molekuskog modelovanja ukazuju na dva moguća vezujuća džepa (gotovo) identičnih energija za oba liganda (u neutralnoj, mono- i di-anjonskoj formi) na molekulu HSA (pod-domeni IIA i IB). Eksperimentalno određena stehiometrija vezivanja, na osnovu kvenčovanja ostatka Trp-214 (pod-domen IIA), pokazuje jedno visoko afinitetno ( $10^6 \text{ M}^{-1}$ ) vezivno mesto za PCB na HSA. Rezultati dobijeni na osnovu promena u fluorescenciji i CD spektrima smeša pigmenta i proteina, kao i u eksperimentima istiskivanja, potvrđuju stereo-selektivno vezivanje *M*-konformera PCB za HSA i njegovu kompeticiju sa BR. Fundamentalni biohemijski značaj i mogući praktični aspekti dobijenih rezultata biće ukratko diskutovani.

**Binding of phycocyanobilin to human serum albumin**

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Human serum albumin (HSA) is an important regulator of the pharmacokinetic properties of bioactive compounds. Phycocyanobilin (PCB) is blue tetrapyrrole chromophore of C-phycocyanin, a major protein of cyanobacteria Spirulina, with proven benefits for human health. For the first time, in this study we characterized the binding of PCB to the HSA and tested competition with structural analogues bilirubin (BR). Methods of molecular modeling point to two putative binding pockets of (virtually) identical energies for both ligands (in the neutral, mono- and di-anionic form) at HSA molecule (sub-domains IIA and IB). The experimentally determined stoichiometry of binding, on the basis of Trp-214 (sub-domain IIA) residue quenching, shows one high affinity ( $10^6 \text{ M}^{-1}$ ) binding site for PCB on the HSA. Results obtained on the basis of changes in fluorescence and CD spectra of pigment and protein mixtures, as well as displacement experiments, confirmed stereo-selective binding of PCB *M*-conformer to HSA and its competition with BR. The fundamental biochemical significance and possible practical aspects of the results will be briefly discussed.

## BHP 4

**Uticaj antipsihotika na reaktivnost tiolne grupe serum-albumina**

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Oksidativni stres se navodi kao jedan od uzroka nastanka šizofrenije. Postoji neslaganje o tome da li primena antipsihotika (AP) u lečenju šizofrenije dodatno doprinosi oksidativnom stresu. Humani serum albumin (HSA) ima jednu slobodnu tiolnu grupu (Cys34) i pošto je najzastupljeniji protein u krvi, predstavlja najvažniji ekstracelularni antioksidans. HSA vezuje različite ligande (masne kiseline, lekove, metale) koji mogu da izazovu promenu konformacije HSA molekula i time promenu reaktivnosti HSA-SH grupe. Cilj ovog rada bio je da se ispita uticaj vezivanja AP na redoks homeostazu (određivanjem sadržaja ukupnih tiola i HSA-SH grupa u plazmi pacova) i na reaktivnost HSA-SH grupe (određivanjem konstante brzine pseudo prvog reda za reakciju HSA-SH sa DTNB) u *in vivo* i *in vitro* eksperimentima. Kod tri grupe pacova, nakon tretmana od 4 nedelje sa sertindolom (S), klozapinom (C) i ziprasidonom (Z), sadržaj ukupnih tiola u plazmi bio je niži u odnosu na kontrolnu grupu ( $0,188 \pm 0,041$  mmol/l). Smanjenje je bilo statistički značajno ( $p < 0,05$ ) samo u grupi tretiranoj sa C. Sadržaj HSA-SH grupa u kontrolnoj grupi ( $0,345 \pm 0,065$  mol-SH/mol HSA) bio je statistički značajno ( $p < 0,05$ ) niži u odnosu na grupu sa C, i statistički značajno ( $p < 0,05$ ) viši u odnosu na grupe sa S i Z. Ispitivanja *in vitro* su pokazala da AP utiču na reaktivnost HSA-SH grupe u stepenu koji zavisi od mesta vezivanja leka na molekulu HSA u odnosu na položaj Cys34 grupe.

**The influence of antipsychotics on serum albumin thiol group reactivity**

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Oxidative stress is mentioned as one of the causes of schizophrenia. There is discrepancy whether the treatment of schizophrenia with antipsychotic (AP) additionally contributes to the oxidative stress. Human serum albumin (HSA) has one free thiol group (Cys34) and as the most abundant protein in the blood, it is major extracellular antioxidant. HSA binds various ligands (fatty acids, drugs, metals) which may cause change in the conformation of HSA molecules and therefore a change of HSA-SH group reactivity. The aim of this study was to investigate the influence of AP binding on redox homeostasis (determining of total thiol and HSA-SH group content in rat plasma) and reactivity of HSA-SH group (determining of the pseudo first order rate constant for the reaction of HSA-SH with DTNB) *in vivo* and *in vitro*. In three groups of rats, after 4 weeks treatment with sertindole (S), clozapine (C) and ziprasidone (Z), the total thiol content in plasma was lower than in the control group ( $0.188 \pm 0.041$  mmol/l). Decrease was statistically significant ( $p < 0,05$ ) only in the group treated with C. The content of HSA-SH group in the control group ( $0.345 \pm 0.065$  mol-SH/mol HSA) was statistically significant ( $p < 0,05$ ) lower than in the group with C, and statistically significant ( $p < 0,05$ ) higher compared to the groups with S and Z. Results of *in vitro* investigations shown that these AP affect HSA-SH group reactivity in the level depending on the distance between the positions of HSA-drug binding site and Cys34 thiol group.

**BH P 5**

**Kvantifikacija masnih kiselina vezanih za humani serum albumin**

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Broj molekula masnih kiselina (MK) vezanih po molekulu humanog serum-albumina (HSA) varira, zavisno od fiziološkog stanja i ishrane organizma. Tokom određivanja kinetičkih parametara reakcije slobodne HSA-Cys34 tiolne grupe sa Elmanovim reagensom utvrđeno je da vrsta i broj vezanih MK po molekulu HSA značajno utiče na kinetiku ove reakcije. Rezultati određivanja sadržaja MK vezanih za HSA mogu se primeniti za procenu reaktivnosti tiolne grupe, odnosno redukcionog kapaciteta HSA u cirkulaciji, što može imati klinički značaj u nekim patološkim stanjima (poput metaboličkog sindroma i dijabetesa) u kojima je metabolizam MK poremećen. Stoga je razvijena metoda kvantitativne tankoslojne hromatografije (qTLC) za određivanje MK vezanih za HSA. Kao standard upotrebljena je stearinska kiselina. Densitometrijska analiza rađena je ImageJ i statističkim programom, koji smo razvili u R jeziku. HSA je izolovan iz humanog seruma frakcionim taloženjem, MK su ekstrahovane smešom heptan-hloroforma i kiselog rastvora bakar(II)-sulfata, i njihov sadržaj je određen qTLC-om. Dobijeni rezultati se statistički ne razlikuju u odnosu na dobijene GC kvantifikacijom (sa internim C13 standardom). Razvijena metoda je tačna, reproduktivna, brza i jeftina, što je čini pogodnom za kliničku praksu.

**Quantification of fatty acids bound to human serum albumin**

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The number of fatty acids (FA) molecules bound per HSA molecule is variable, depending of the physiological state and diet. During the investigation of kinetics parameters of the reaction of free HSA-Cys34 thiol group and Ellman's reagent it was found that FA bound to HSA lead to the significant change of reaction kinetic. Thus, the results of determination of FA content bound to HSA could be used for the estimation of thiol group reactivity, i.e. of the redox capacity of HSA in circulation, making them useable in clinical studies of some pathologies (eg. diabetes and metabolic syndrome) with disturbed FA metabolism. Therefore, the quantitative thin-layer chromatography (qTLC) method for determination of FA bound to the HSA was developed. Stearic acid was used for preparation of the standard solution. For the FA quantification, a densitometric analysis with the ImageJ was performed, and a specialised program developed in the R language was used. The HSA was isolated from human serum by fractional precipitation, FA were extracted with heptane-chloroform and acidified copper(II)-supfate mixture, and their content was determined using the qTLC. The results obtained by qTLC and referent GC method (with internal C13 standard) were statistically comparable. Developed qTLC method is accurate, reproducible, low time-consuming and inexpensive, which makes it suitable for clinical practice.

BH P 6

### **Fenolni profil i antioksidantna aktivnost plodova vrste *Sorbus domestica* L.**

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Plodovi vrste *Sorbus domestica* L. (oskoruša) se koriste kao hrana velike nutritivne vrednosti, ali i kao tradicionalno adstringentno, antidijaroično i antidijabetičko sredstvo. Cilj prikazanih ispitivanja bio je određivanje fenolnog profila i antioksidantne aktivnosti vodenih i metanolnih ekstrakata suvog ploda. Prisustvo i kvantitativni sadržaj 44 odabrana fenolna jedinjenja i hinske kiseline ispitan je primenom LC-MS/MS tehnike. U cilju određivanja antioksidantnog potencijala sprovedena su tri *in vitro* testa: određivanje kapaciteta hvatanja DPPH i NO radikala, kao i određivanje redukcionog potencijala (FRAP). LC-MS/MS analizom identifikovano je prisustvo 23 od 45 odabrana fenolna jedinjenja. Hinska kiselina (prekursor fenola) je bila dominantna u oba ekstrakta, a zatim slede protokatehinska kiselina i hlorogenska kiselina. U korišćenim testovima metanolni ekstrakt je pokazao najveći antioksidanti potencijal. U poređenju sa standardnim antioksidansom, propil galatom (PG), svi antioksidantni testovi su pokazali umerenu antioksidantnu aktivnost. Dobijeni rezultati podržavaju upotrebu plodova *S.domestica* kao hrane i potencijalnih medicinskih preparata i impliciraju dalja istraživanja.

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### **Phenolic profile and antioxidant activity of fruits of *Sorbus domestica* L.**

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Fruits of *Sorbus domestica* L. (service tree) have been widely used as nutritious food and also as traditional adstringent, diarrheic and antidiabetic agents. The aim of this study was to define phenolic profile and antioxidant activity of water and methanolic extracts of dried fruits. The presence and content of 44 plant phenolics and quinic acid was determined using LC-MS/MS technique. In order to examine antioxidant potency, three *in vitro* assays were carried out: DPPH (diphenylpicrylhydrazyl) and NO (nitric oxide) scavenger capacity test and reducing power (FRAP) assay. LC-MS/MS analyses of selected phenols resulted in determination of 23 of 45 compounds. Among examined compounds, quinic acid (precursor of phenolics) was the most dominant in both samples, followed by protocatechuic acid and chlorogenic acid. In applied antioxidant tests, methanolic extract showed the highest antiradical activity. Compared to propyl galate, a well known synthetic antioxidant, the extracts exhibited a moderate antioxidant activity. Obtained results support the use of *S.domestica* fruits in food and potential medical preparations and encourage further studies.

*The Ministry of Education and Science RS (Grant No.172058) supported this research work.*

**BH P 7**

**Fenolni profil i antioksidantni potencijal šipka vrste *Rosa arvensis* Huds.**

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Plod vrsta *Rosa* (šipak) dugo se koristi u ishrani i narodnoj medicini kao izvor jedinjenja sa blagotvornim efektom na zdravlje. Cilj prikazanih ispitivanja bilo je određivanje fenolnog profila i evaluacija antioksidantne aktivnosti vodenog i metanolnog ekstrakta osušenog ploda do sada neispitane vrste *Rosa*, *Rosa arvensis* Huds. (poljska ruža). Prisustvo i kvantitativan sadržaj 44 odabrana fenolna jedinjenja i hinske kiseline ispitan je primenom LC-MS/MS tehnike. Detektovana su 25 jedinjenja, pri čemu je hinska kiselina, prekursor fenolnih jedinjenja, bila dominantna u oba ispitivana ekstrakta. Takođe, u vodenom ekstraktu određena je značajna količina fenolnih kiselina (protocatehinske i galne kiseline), dok su u metanolnom ekstraktu bili zastupljeniji flavonoidi, a posebno glikozidi kvercetina (kvercitrin, kvercetin-3-O-glukozid i hiperozid). Antioksidantna aktivnost procenjena je primenom tri *in vitro* testa: određivanjem kapaciteta „hvatanja“ azot monoksid (NO) i hidroksil (OH) radikala, kao i lipidne peroksidacije (LP). Oba ekstrakta pokazala su umeren antioksidantni potencijal u poređenju sa propil galatom (PG) i butilovanim hidroksitoluonom (BHT), dobro poznatim sintetičkim antioksidansima. Dobijeni rezultati predstavljaju šipak vrste *R. arvensis* kao dobar izvor fenolnih jedinjenja, umerene antioksidantne aktivnosti i podržavaju dalja ispitivanja.

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**Phenolic profile and antioxidant potential of *Rosa arvensis* Huds. rose hips**

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*Rosa* species fruit (rose hips) have been used for long time in diet and ethnomedicine, as a source of compounds with healthy properties. Therefore, the aim of this study was to examine phenolic profile and antioxidant activity of water and methanol extracts of air-dried rose hips of still unexplored *Rosa* species, *Rosa arvensis* Huds. (field rosa). The presence and content of 44 selected phenolics and quinic acid were studied using LC-MS/MS technique. Twenty five compounds were detected, where quinic acid, precursor of phenolics, was dominant compound in both extracts. In addition, water extract was more abundant with phenolic acids (protocatechuic and gallic acid), while in methanol extracts flavonoids, especially quercetin glycosides (quercitrin, quercetin 3-O-glucoside and hyperoside) were present in notable amount. Antioxidant activity was evaluated using three *in vitro* assays: nitric oxide (NO) and hydroxyl (OH) radical scavenger capacity test, as well as lipid peroxidation (LP). Both extracts showed moderate antioxidant activity in comparison with propyl gallate (PG) and butylated hydroxytoluene (BHT), well-known synthetic antioxidants. Obtained results present *R. arvensis* rose hips as a good source of phenolics, with moderate antioxidant potential and encourage further studies.

*The Ministry of Education, Sciences and Technological Development RS (OI 172050) supported this research work*



### Mogući mehanizam antioksidativnog delovanja antidepresiva

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Inhibitori ponovnog preuzimanja serotonina, norepinefrina i/ili dopamina su lekovi izbora u tretmanu depresije. U ovoj *in vitro* studiji su ispitani efekti antidepresiva na antioksidativni status u humanim eritrocitima i antioksidativnost lekova koristeći set standardnih antioksidativnih eseja (DPPH<sup>•</sup>, ABTS<sup>•+</sup>, ORAC, UAOK, "hvatanje" O<sub>2</sub><sup>-</sup> i OH<sup>•</sup>). Nakon inkubiranja pune krvi sa najvećom preporučenom dnevnom dozom sedam predhodno izolovanih aktivnih supstanci, statistički značajno smanjenje (p<0,05) aktivnosti Cu,Zn SOD nađeno je kod tretmana duloksetinom, escitalopramom i fluoksetinom. Nasuprot tome, aktivnost CAT, Se-GPx i GR nije bila statistički značajno različita od odgovarajuće kontrole. Niti jedan ispitani antidepresiv preinkubiran sa Cu,Zn SOD (mol/mol) nije menjao CD ili fluorescentne emisije spektre proteina, ukazujući da promene u aktivnosti nisu posledica direktnih interakcija leka sa ovim antioksidativnim enzimom. Samo tri gore navedena antidepresiva pokazuju izraženiji antioksidativni potencijal *per se*. S obzirom da je oksidativni stres uključen u etiologiju depresije, naši rezultati sugerišu da je zaštitni efekat nekih antidepresiva i uklanjanje slobodno-radikalnih vrsta, ukazujući na mehanizam njihovih plejotropnih efekata kao prateći deo ciljane kliničke namene.

### Possible mechanism of antidepressants antioxidant action

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Reuptake inhibitors of serotonin, norepinephrine and/or dopamine are the drugs of choice in the treatment of depression. This *in vitro* study tested the effects of antidepressants on antioxidant status in human erythrocytes, as well as antioxidant status of drugs using a set of standard antioxidant essays (DPPH<sup>•</sup>, ABTS<sup>•+</sup>, ORAC, TAOC, O<sub>2</sub><sup>-</sup> and OH<sup>•</sup> scavenging). After incubation of whole blood with a maximum recommended daily dose for seven previously isolated active substances, a statistically significant reduction (p<0.05) in activity of Cu,Zn SOD was found after the treatment with duloxetine, escitalopram and fluoxetine. In contrast, the activity of CAT, Se-GPx and GR was not statistically significantly different from the appropriate control. None of the tested antidepressant pre-incubated with Cu,Zn SOD (mol/mol) did not change the CD or fluorescence emission spectra of the protein, suggesting the change in activity was not the result of direct interaction of the drug with this antioxidant enzyme. Only three above mentioned antidepressants showed a significant antioxidant potential *per se*. Since oxidative stress is involved in the etiology of depression, our results suggest the protective effect of some antidepressants by removal of free-radical species, indicating the mechanism of their pleiotropic effects as the consequence of targeted clinical purpose.

**BH P 9**

**Biomedicinski potencijal novih estranskih saliciloiloksi derivata**

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Mnogi derivati estrogenih hormona se vezuju za estrogene receptore i deluju kao antiestrogeni, drugi inhibiraju enzime steroidogeneze, značajne za veliki broj životno važnih procesa, a neki deluju kao antioksidansi ili antiproliferativni agensi. Stoga je cilj ovog rada bilo ispitivanje biomedicinskog potencijala steroidnih jedinjenja u čijoj su strukturi kombinovane karakteristike estrogenih hormona i derivata salicilne kiseline, dve biološki veoma aktivne grupe jedinjenja. Ispitali smo inhibitorni potencijal sintetisanih estranskih saliciloiloksi derivata na enzime  $\Delta^5$ -3 $\beta$ HSD, 17 $\beta$ HSD1, 17 $\beta$ HSD2 i 17 $\beta$ HSD3, kao i antiproliferativnu i antioksidantnu aktivnost. Najpotentnijim se pokazao 3-hidroksiestra-1,3,5(10)-trien-17 $\beta$ -il salicilat, koji je vrlo efikasno inhibirao enzim 17 $\beta$ HSD2.

**Biomedical potential of novel salicyloyloxy estrane derivatives**

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Many estrogen hormone derivatives bind to estrogen receptors and act as antiestrogens, some inhibit steroidogenic enzymes, important for a number of vital processes, while others act as antioxidants or anti-proliferative agents. The aim of this study was to test biomedical potential of steroid compounds, whose structures possess characteristics of both estrogenic hormones and salicylic acid derivatives, two biologically very active groups. We examined the inhibitory potential of the synthesized salicyloyloxy estrane derivatives against enzymes  $\Delta^5$ -3 $\beta$ HSD, 17 $\beta$ HSD1, 17 $\beta$ HSD2 and 17 $\beta$ HSD3, as well as their antiproliferative and antioxidant activity. Among others, 3-hydroxyestra-1,3,5(10)-trien-17 $\beta$ -yl salicylate very efficiently inhibited 17 $\beta$ HSD2 enzyme.

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BH P 10

### **Antikancerska aktivnost 17 $\alpha$ -pikolil i 17(E)-pikolinilidenskih derivata androstana na MDA-MB-231 ćelije kancera dojke**

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U radu je ispitivan antiproliferativni i pro-apoptotski potencijal 17 $\alpha$ -pikolil i 17(E)-pikolinilidenskih derivata androstana. Svi steroidni derivati smanjili su proliferaciju MDA-MB-231 ćelija. Takođe su uticali na ćelijski ciklus i indukovali apoptozu i nekrozu iste linije ćelija. Većina jedinjenja povećavala je ekspresiju pro-apoptotskog Bax proteina. Ekspresija anti-apoptotskog Bcl-2 proteina bila je manja u većini tretiranih uzoraka u poređenju sa kontrolom. Raskidanje PARP proteina potvrđeno je u svim uzorcima. Izostanak aktivacije kaspaze-3 nekim jedinjenjima ukazuje na indukciju apoptoze kaspaza-nezavisnim putevima. Rezultati su pokazali da neka od ispitivanih jedinjenja imaju značajan biomedicinski potencijal i da bi mogla biti kandidati za razvoj antikancerske terapije.

### **Anticancer activity of 17 $\alpha$ -picolyl and 17(E)-picolinylidene androstane derivatives against MDA-MB-231 breast cancer cells**

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Antiproliferative and pro-apoptotic potential of 17 $\alpha$ -picolyl and 17(E)-picolinylidene androstane derivatives was studied. All tested steroid derivatives decreased proliferation of MDA-MB-231 cells. They also affected the cell cycle distribution and induced apoptosis and necrosis in the same cell line. Most derivatives induced higher expression of proapoptotic Bax protein. The expression of antiapoptotic Bcl-2 protein was lower in almost all treated samples compared to control. Cleavage of PARP protein was confirmed in all treated samples. The lack of Caspase 3 activation by some compounds, suggested that they induced apoptosis in Caspase-independent manner. Results showed that some of the investigated compounds have substantial biomedical potential and could be candidates for anticancer drug development.

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## Biotehnologija / Biotechnology

BT O 1

### **Hidroliza koncentrovane suspenzije sirovog skroba $\alpha$ -amilazom iz *Bacillus licheniformis* 9945a**

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$\alpha$ -Amilaza iz *Bacillus licheniformis* 9945a (BliAmy) je veoma efikasna u hidrolizi koncentrovanog sirovog kukuruznog skroba na temperaturi ispod temperature njegovog geliranja (60 °C). Kako na proces hidrolize koncentrovanog sirovog kukuruznog skroba utiče veći broj promenljivih, (sadržaj suve supstancije, temperatura, količina enzima, pH i vreme), metodom odzivne površine (Response surface methodology, RSM) određeni su reakcioni parametri hidrolize. Bez obzira na koncentracije sirovog skroba, BliAmy je bila veoma efikasna, postižući skoro kompletnu hidrolizu pri 30 %-noj suspenziji skroba posle 22 časa hidrolize. Način degradacije kukuruznih skrobnih granula i promene u strukturi skroba tokom hidrolize praćeni su TLC, SEM i XRD metodama.

### **Hydrolysis of concentrated raw corn starch by $\alpha$ -Amylase from *Bacillus licheniformis* 9945a**

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$\alpha$ -Amylase from *Bacillus licheniformis* 9945a (BliAmy) was found to be very efficient in hydrolysis of concentrated raw corn starch at temperatures below the starch gelatinization temperature (60 °C). The hydrolysis of raw corn starch is affected by many variables including solid content, temperature, enzyme loading, pH and time. Response surface methodology (RSM) was used to optimize the reaction parameters of concentrated raw corn starch hydrolysis. Regardless of raw starch concentration tested, BliAmy was very effective, achieving almost complete hydrolysis degree on 30 % starch suspension after 22 h of hydrolysis. The mode of degradation of native maize starch granules and the changes in the starch structure during the hydrolysis was monitored by TLC, SEM and XRD methods.



BT O 2

### **Stabilizacija lipaza iz *Candida rugosa* jednostavnom i efikasnom imobilizacijom na hidroksiapatitu**

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Razvili smo brz i efikasan metod imobilizacije industrijski veoma vrednih *Candida rugosa* lipaza (CRL) na ekonomičan, biokompatibilan nosač - hidroksiapatit, sa visokim prinosom imobilizacije (blizu 100 %) i prinosom aktivnosti od 50 %. Imobilizovane lipaze su pokazale značajno višu stabilnost nego slobodni enzim, nakon termalnog tretmana na 60 °C i u prisustvu različitih 95 % polarnih organskih rastvarača, pre svega kratkolančanih alifatičnih alkohola, značajnih polaznih sirovina u sintezi brojnih estara i drugih značajnih proizvoda. Predstavljeni rezultati ukazuju na veliki upotrebnii potencijal dobijenog preparata u različitim industrijskim procesima, koji iziskuju rad u nekonvencionalnim reakcionim uslovima.

### ***Candida rugosa* lipase stabilization by simple and effective immobilisation on hydroxyapatite**

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We have developed a simple and highly effective method for immobilising industrially very appreciated and valuable *Candida rugosa* lipases from commercial preparation on ecologically suitable, biodegradable and economical hydroxyapatite support. Our immobilisation protocol resulted in excellent immobilisation yield of nearly 100 % and activity yield of 50 %, which is significantly higher in comparison to other immobilisation protocols for different enzymes on the same support. Immobilised lipase formulation has proven to have superior stability, compared to free enzyme, at both high temperature (60 °C) and in the presence of different polar organic solvents, especially short-chain alcohols: methanol, ethanol and iso-propanol. Therefore, presented experimental data strongly support the great future potential of the prepared *Candida rugosa* immobilisate.

**BT P 1**

**Obezbojavanje antrahinonskih boja peroksidazom izolovanom  
iz svežeg ekstrakta rena**

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Peroksidaza iz rena se pokazala kao izuzetan biokatalizator za oksidaciju brojnih aromatičnih jedinjenja uključujući i antrahinonske boje. Poslednjih godina reakcije oksidacije antrahinonskih boja katalizovane peroksidazom iz rena sve više dobijaju na značaju zbog blagih reakcionih uslova, velike efikasnosti, malih utrošaka energije i ekološki prihvatljivih sporednih proizvoda. U cilju zaštite zdravlja čoveka i životne sredine, pre ispuštanja otpadnih voda u prirodne vodotokove neophodan je njihov pravilan tretman. U cilju ispitivanja obezbojavanja, kao model su izabrane dve antrahinonske boje: C.I. Acid Violet 109 (AV 109) i C.I. Acid Blue 225 (AB 225). U reakciji razgradnje izabranih antrahinonskih boja korišćena je peroksidaza iz svežeg ekstrakta rena bez daljeg prečišćavanja. U reakcijama katalizovanim sa 0,1 IU enzimske aktivnosti uklonjeno je 89 % AV 109 boje nakon 30 minuta (pH 4,0, koncentracija vodonik-peroksida 0,1 mM, koncentracija boje 30 mg dm<sup>-3</sup>) i 75 % AB 225 boje (pH 5,0, koncentracija vodonik-peroksida 0,2 mM, koncentracija boje 30 mg dm<sup>-3</sup>) nakon 40 minuta.

**Anthraquinone dyes decolorization using peroxidase  
from fresh horseradish extract**

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Horsradish peroxidase approved to be a great alternative for the detoxification of different organic compounds including anthraquinone dyes. Recently, horseradish peroxidase-mediated decolorization reactions have attracted much attention because of the mild reaction conditions, high efficiency, low energy consumption and eco-friendly by-products. Because of the human health and nature safety, proper treatment of polluted and colored wastewater is necessary before discharging in to the natural recipients. This paper deals with the decolorization of two model dyes: C.I. Acid Violet 109 (AV 109) and C.I. Acid Blue 225 (AB 225). For this purpose, fresh extract of horseradish peroxidase (from roots) without further purification was used. 0.1 IU of enzyme activity was sufficient for 89 % of AV 109 (pH 4.0, hydrogen peroxide 0.1 mM, dye concentration 30 mg dm<sup>-3</sup>), and 75 % of AB 225 dye decolorization (pH 5.0, hydrogen peroxide 0.2 mM, dye concentration 30 mg dm<sup>-3</sup>), within 30 and 40 min, respectively.



BT P 2

## **Procjena oksidativne stabilnosti biodizela – uticaj prirodnih antioksidanata**

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Biodizel je gorivo dobijeno iz biljnih ili životinjskih sirovina kroz proces esterifikacije. Zbog svoje hemijske strukture podložno je oksidacionim procesima. Sirovina (jestivo ulje, životinjska mast ili korišćeno otpadno ulje) sadrži značajnu količinu nezasićenih masnih kiselina i time je proces autoksidacije neizbježan, posebno pri dugom skladištenju.

U ovom radu, ispitan je antioksidativni potencijal 4 različite aromatične biljke iz porodice Lamiaceae kod biodizela dobijenog iz rafinisanog i korišćenog ulja uljane repice. Mjerenja oksidacione stabilnosti su vršena na Rancimat instrumentu na temperature od 110 °C.

Rezultati su pokazali da ružmarin i posebno žalfija imaju visok antioksidativni potencijal u poređenju sa sintetičkim antioksidantom BHT, posebno kod biodizela dobijenog iz otpadnog ulja uljane repice.

## **Evaluation of oxidative stability of biodiesel – impact of natural antioxidants**

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Biodiesel is a fuel component made from plant or animal feedstock through an esterification process. Due to the chemical structure, the biodiesel is susceptible to oxidation processes. Its raw material (vegetable oils, animal fats and used frying oils) contains a significant amount of fatty acids with double bonds, so, its oxidative stability is a reason for concern, especially when storing it over an extended period of time.

In this work, the potential of 4 different aromatic plants from Lamiaceae family to improve the oxidation stability of refined and waste rapeseed oil based biodiesel was investigated. Measurements of oxidation stability were carried out according to the European biodiesel specifications with a Rancimat instrument at 110 °C.

Rosemary and especially sage showed very high antioxidant protection factor compared to commercial antioxidant BHT, especially in waste rapeseed oil based biodiesel.

**BT P 3**

**Aflatoksin u kukuruзу iz žetve 2013. godine u Republici Srbiji**

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Tokom jeseni 2013. i prvog polovini 2014. godine, sa različitih farmi u Srbiji, prikupljen je 221 uzorak kukuruza poreklom iz proizvodne 2013. godine. Uzorci su zatim analizirani na prisustvo aflatoksina korišćenjem imunohemijske (ELISA) metode. Prosečan sadržaj aflatoksina u ispitivanim uzorcima kukuruza je iznosio 33,6 µg/kg, u opsegu od 2 do 93 µg/kg. Prisustvo aflatoksina u uzorcima je iznosilo 16,3 %, a 6,8 % uzoraka je sadržavalo aflatoksin iznad maksimalno dozvoljene granice prema važećoj regulativi Republike Srbije, odnosno 18,1 % prema regulativi Evropske Unije. Uzimajući u obzir vremenske uslove, može se zaključiti da su oni u 2013. godini bili znatno nepovoljniji za rast plesni iz roda *Aspergillus* u odnosu na 2012. godinu kada je utvrđeno izuzetno visoko prisustvo i sadržaj aflatoksina. To su potvrdili i rezultati predstavljeni u ovom radu, gde je pored smanjenog prisustvu aflatoksina u uzorcima kukuruza poreklom iz žetve 2013. godine, registrovan i smanjen njegov sadržaj.

**Aflatoxin in Serbian maize from 2013 harvest**

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Two hundred and twenty one samples of maize from 2013 harvest were randomly collected from different farms in Serbia during autumn 2013 and the first half of 2014 year. Samples were then analyzed for the presence of aflatoxin using enzyme linked immunosorbent assay (ELISA) method. The average aflatoxin content was 33.6 µg/kg within range from 2 to 93 µg/kg. Established overall presence was 16.3 %. Regarding legislation, 6.8 % of samples were above the maximum limit permitted by Serbian and 18.1 % above EU regulations. Bearing in mind the weather conditions, it can be concluded that these in 2013 were considerably less favorable for the growth of *Aspergillus* mold relative to 2012 when remarkably high aflatoxin presence and content were found. This was confirmed by results presented in this paper, where besides lower presence of aflatoxin, its content in maize was meaningfully decreased.

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## Hemija i tehnologija hrane / Chemistry and Technology of Food

HTH O 1

### Optimizacija uslova ultrazvučne ekstrakcije za određivanje sadržaja fenola i antioksidativni kapacitet divljih jabuka

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Cilj ovog rada bio je optimizovati uslove ultrazvučne ekstrakcije za fenolna jedinjenja i odrediti antioksidativni kapacitet divljih jabuka (kora i pulpa) korišćenjem metodologije odgovora površine. Statistički softver Design Expert je korišćen za određivanje uticaja koncentracije metanola (v/v %), odnosa čvrsto/tečno (g/mL), temperature (°C) i vremena ekstrakcije (min) na sadržaj fenolnih jedinjenja. HPLC analiza ekstrakata dobijenih pod optimalnim uslovima je pokazala da oba ekstrakta sadrže hlorogensku kiselinu, epikatehin i floridzin, dok ekstrakt kore sadrži još i katehin, hiperozid i kvercitrin. Ekstrakt kore ima veći sadržaj ispitivanih fenolnih jedinjenja, kao i flavonoida i fenolnih kiselina kvantifikovanih HPLC metodom. Ekstrakt kore je takođe pokazao bolju antioksidativnu aktivnost koja je ispitana DPPH, ABTS<sup>+</sup>, NO, OH, O<sub>2</sub><sup>-</sup> metodama i metodom reduktivne aktivnosti. Dobijeni optimalni uslovi mogu biti iskorišćeni u daljim istražovanjima za izolovanje flavonoida i fenolnih kiselina, koji su prisutni u divljoj jabuci, a koja je zapostavljena kao prirodni resurs ovih važnih bioaktivnih jedinjenja.

### Optimization of ultrasound-assisted extraction conditions for phenolic content and antioxidant capacity of wild apple fruits

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The objective of this study was to optimize ultrasound - assisted extraction conditions for phenolic compounds and antioxidant capacity in wild apples (peel and pulp) using response surface methodology. Design Expert statistical software was applied to determine the effects of methanol concentration (v/v %), solid : liquid ratio (g/mL), temperature (°C) and extraction time (min) on phenolic content. HPLC analysis of extracts obtained by extraction under optimal conditions showed that both of extracts contains chlorogenic acid, epicatechin and phloridzin, while peel contains catechin, hyperozide and quercitrin except these three compounds. The peel extract contains higher concentrations of phenolic compounds, as well as flavonoids and phenolic acid quantified using HPLC. Peel extract also showed higher antioxidant activity, measured by DPPH, ABTS<sup>+</sup>, NO, OH, O<sub>2</sub><sup>-</sup> scavenging activity methods and reducing activity method. Obtained optimal conditions could be used in further research for the isolation of flavonoids and phenolic acids that are present in wild apple, which has been underestimated as a natural resource of these important bioactive compounds.

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HTH P 1

**Antioksidantni potencijal šire i vina „Frankovka“**

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Sorta vinove loze *Frankovka* se koristi širom Evrope za proizvodnju vina. Postoji malo podataka o antioksidantnom potencijalu različitih proizvoda od ove sorte grožđa, te je cilj ovog rada bilo određivanje antioksidantne aktivnosti i sadržaja ukupnih fenola i flavonoida, glavnih nosioca ove biološke aktivnosti, u širi i vinima starim 14 dana (mlado vino), odnosno 6 meseci. Sadržaj ukupnih fenola i flavonoida, kao i antioksidantni potencijal određeni su primenom standardnih spektrofotometrijskih testova. Antioksidantna aktivnost je ispitana testovima zasnovanim na sposobnosti neutralizacije slobodnih radikala (kapacitet „hvatanja“ hidroksil radikala) i transferu elektrona (neutralizacija difenilpikrilhidrazil radikala i određivanje redukcione sposobnosti-FRAP test). Najveći sadržaj ukupnih fenola i flavonoida detektovan je u širi i opada tokom starenja vina, najverovatnije usled degradacije ovih jedinjenja tokom sazrevanja vina. U svim primenjenim testovima, šira je ispoljila najbolju antioksidantnu aktivnost, koje korelira sa ukupnim sadržajem fenola i flavonoida. U poređenju sa propil-galatom, poznatim sintetičkim antioksidantom, uzorci su ispoljili sličan antioksidantni efekat. Rezultati potvrđuju da su ispitani uzorci potentan izvor prirodnih antioksidanata (fenola i flavonoida) i nedvosmisleno potvrđuju njihovu vezu sa „francuskim paradoksom“, jer se smatra da su ovi prirodni proizvodi odgovorni za smanjenu učestalost kardiovaskularnih bolesti među francuskom populacijom.

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**Antioxidant potential of must and wine "Blaufränkisch"**

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Grape variety *Blaufränkisch* is used all over Europe for producing wine. There is little data on antioxidant potential of various products of this grape variety. Therefore, the goal of this study was to determine antioxidant activity and the content of total phenolics and flavonoids, as the main carrier of biological activities in must and wines 14 days (young wine) and 6 months old. Content of total phenolics and flavonoids, as well as the antioxidant potential were determined using standard spectrophotometric assays. Antioxidant potential was evaluated using antioxidant tests, based on measuring the radical scavenging effect on diphenylpicrylhydrazyl radical and hydroxyl radical, and reducing power (FRAP) assay. The highest content of total phenols and flavonoids was detected in the must and decreased during the aging of wine, probably due to the degradation of these compounds during wine ripening. In all tests, the must had the best antioxidant activity, correlating with total phenol and flavonoid content. Compared with propyl gallate, a well known synthetic antioxidant, the samples expressed similar antioxidant effect. The results confirmed that the must and wine are a potent source of natural antioxidants (phenols and flavonoids), and undoubtedly connecting them to the "French paradox" -responsability of this natural products for reduce cardiovascular disease among the French population.

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## HTH P 2

**Superkritična ekstrakcija korijandera – Uticaj srednjeg prečnika čestica**

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Korijander (*Coriandrum sativum* L.) je aromatična i medicinska biljka iz porodice *Apiaceae*. Seme korijandera sadrži različita lipofilna (nezasićene masne kiseline i etarsko ulje) i hidrofилna (fenolne kiseline i flavonoide) bioaktivna jedinjenja. Prema tome, različita farmakološka delovanja kao što su antimikrobno, antioksidativno, antidijabetičko, antiinflamatorno i neuroprotektivno delovanje, su pripisana semenu korijandera. Cilj ovog rada je bilo ispitivanje uticaja srednjeg prečnika čestica usitnjenog semena korijandera na prinos i hemijski sastav ekstrakata dobijenih superkritičnom ekstrakcijom (SFE). Ekstrakti dobijeni pomoću SFE su upoređeni sa lipidnim ekstraktima i etarskim uljem dobijenim Soxhlet ekstrakcijom i destilacijom vodom, a njihov hemijski sastav je određen GC-MS i GC-FID analizom. Dominantno jedinjenje u etarskom ulju, kao i u ekstraktima dobijenim pomoću SFE, je linalool, čiji je prinos rastao sa smanjenjem srednjeg prečnika čestica biljnog materijala. Pored linaloola, dominantna jedinjenja u ekstraktima korijandera su monoterpenski ugljovodoni ( $\gamma$ -terpinen i (+)-limonen) i oksidovani monoterpini (kamfor i geraniol).

**Supercritical fluid extraction of coriander – Influence of mean particle size**

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Coriander (*Coriandrum sativum* L.) is aromatic and medicinal plant, belonging to *Apiaceae* family. Coriander seeds contain various lipophilic (unsaturated fatty acids and essential oil) and hydrophilic (phenolic acids and flavonoids) bioactive compounds. Therefore, various pharmacological activities such as antimicrobial, antioxidant, antidiabetic, antiinflammatory and neuroprotective, have been ascribed to coriander seeds, due to its interesting chemical composition. The aim of this work was to investigate influence of mean particle size of coriander seeds on yield and chemical profile of extracts obtained by supercritical fluid extraction (SFE). Extracts obtained by SFE were compared with lipid extracts and essential oil obtained by Soxhlet extraction and hydrodistillation, and their chemical profile was determined by GC-MS and GC-FID analysis. Dominant compound in essential oil, as well as in extracts obtained by SFE, was linalool, which yield increased with reduction of mean particle size of plant material. Besides linalool, dominant compounds in coriander extracts were monoterpene hydrocarbons ( $\gamma$ -terpinene and (+)-limonene) and oxygenated monoterpenes (camphor and geraniol).

HTH P 3

**Superkritična ekstrakcija i antioksidativna aktivnost  
etarskog ulja korijandera**

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Korijander (*Coriandrum sativum* L.; *Apiaceae* botanička familija) je jednogodišnja biljka koja potiče sa Mediterana i Bliskoistočne regije. Biljka cveta u junu i julu, a daje okrugle plodove sa dva perikarpa. Ova biljka se uzgaja radi dobijanja lišća i semena. Seme korijandera sadrži oko 18% masnih kiselina/triglicerida, i oko 0,84% etarskog ulja. Plod korijandera, takođe, sadrži više masnih kiselina/triglicerida nego etarskog ulja. Etarsko ulje je bezbojna do svetlo žuta tečnost koja poseduje karakterističan miris i ukus. Glavna komponenta etarskog ulja je linalool (do 70%). Etarsko ulje ove biljke se u prehrambenoj industriji upotrebljava kao začim, a poznata je i njegova upotreba u tradicionalnoj medicini (protiv reumatizma i bolova u zglobovima). Cilj ovog istraživanja bio je izolovanje etarskog ulja iz semena korijandera primenom superkritične ekstrakcije, kao i ispitivanje antioksidativne aktivnosti dobijenih ekstrakata. U cilju dobijanja ekstrakata izvršene su ekstrakcije na pritiscima od 100, 150 i 200 bar, temperaturama 40, 55 i 70°C i protocima ekstragensa od 0,2; 0,3 i 0,4 kg CO<sub>2</sub>/h. Određeni su prinosi ekstrakcije i dobijeni ekstrakti su testirani na antioksidativnu aktivnost primenom DPPH testa i metode inhibicije lipidne peroksidacije.

**Supercritical fluid extraction (SFE) and antioxidant activity  
of coriander essential oil**

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Coriander (*Coriandrum sativum* L., *Apiaceae* botanical family) is an annual, herbaceous plant originally from the Mediterranean and Middle Eastern regions. The plant flowers from June to July, and yields round fruits consisting of two pericarps. The plant is cultivated for its aromatic leaves and seeds. The seeds contain up to 18% of oil (fatty acids/triglycerides); however, the essential oil content in seeds is approximately 0.84%. Coriander extract from the fruits is also very rich in fat, and relatively poor in essential oil. Essential oil is a colorless or pale yellow liquid with a characteristic odor and mild, sweet, warm and aromatic flavor; linalool is the major constituent (up to 70%). In the food industry, coriander oil is used as a flavoring agent and adjuvant, but it also has a long history in the traditional medicine (against rheumatism and pain in the joints). Aim of this research was to isolate essential oil from coriander fruit and test the obtained extracts for antioxidant activity. Extraction processes were conducted at three different pressures (100, 150, and 200 bar), temperatures (40, 55, and 70°C) and solvent flows (0,2; 0,3, and 0,4 kg CO<sub>2</sub>/h). After determination of extraction yield, obtained extracts were tested for antioxidant activity using DPPH assay and lipid peroxidation test.

HTH P 4

### **Uticaj sistema gajenja na sadržaj skroba u krtolama krompira**

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Hemijski sastav i nutritivnu vrednost krompira određuju različiti faktori: sortiment, oblast gajenja, zemljište, klima i primenjena poljoprivredna praksa. U cilju određivanja uticaja sorte i načina gajenja na sadržaj skroba u krtolama krompira ispitivani su uzorci četiri sorte iz dve godine proizvodnje u tri različita sistema gajenja. Na osnovu određene ukupne specifične rotacije ( $\alpha_1$ ) i specifične rotacije komponenti rastvornih u etanolu ( $\alpha_2$ ) izračunat je sadržaj skroba u kori i jezgri krtola. Dobijeni rezultati pokazuju da je jezgro bogatije skrobom u odnosu na koru. U uzorcima krompira iz dve godine proizvodnje (2013. i 2014.), nije utvrđena statistički značajna razlika između srednjih vrednosti sadržaja skroba. Jednofaktorska analiza varijanse je pokazala da se ispitivane sorte ne razlikuju značajno po sadržaju skroba. Međutim, utvrđena je statistički značajna razlika u sadržaju skroba u uzorcima dobijenim organskim, konvencionalnim i integralnim sistemom zemljoradnje. Uzorci gajeni konvencionalnim sistemom zemljoradnje imaju najveći sadržaj skroba.

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### **The influence of growing system on the content of starch in potato tubers**

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Chemical composition and nutritional value of potatoes is determined by various factors: cultivar, growing area, soil, climate and agricultural practice. In order to determine the impact of the cultivar and cultural practice on starch content, the samples of four cultivars from two production years in three different agronomic systems were tested. Based on total specific rotation ( $\alpha_1$ ) and specific rotation of components dissolved in ethanol ( $\alpha_2$ ) the content of starch in the core and peel of tubers was calculated. Results showed that the core contained more starch in comparison to peel. There was no statistically significant difference between the mean values of starch content in samples from two production years (2013 and 2014). One-way analysis of variance showed that the investigated cultivars were not differ in starch content. However, the statistically significant difference was established regarding to organic, conventional or integral systems of agriculture. The highest starch content was found in potato samples from conventional farming system.

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HTH P 5

**Primena binarnog sistema u multivarijantnoj analizi heksanskih ekstrakata cerealija i pseudocerealija**

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U ovom radu analizirani su heksanski ekstrakti brašna različitih sorti heljde, amarantusa, spelte, kukuruza i strnih žita (pšenice, ječma, ovsa, raži i tritikalea). Heksanom ekstrahovane lipidne komponente derivatizovane su reagensom za transesterifikaciju, trimetilsulfonijum-hidroksidom (TMSH) u 0,2M metanolu. Primenom gasne hromatografije sa masenom spektrometrijom u heksanskim ekstraktima cerealija i pseudocerealija identifikovani su metil-estri dominantnih i minornih masnih kiselina, kao i neosapunjive materije: fitosteroli, tokoferol i skvalen. Eliminacijom metil-estara dominantnih masnih kiselina prisutnih u heksanskim ekstraktima svih ispitanih sorti i primenom binarnog sistema u multivarijantnoj analizi automatski integrisanih površina ekstrahovanih komponenata, ispitana je mogućnost diferencijacije brašna cerealija i pseudocerealija. Rezultati pokazuju da se heljda, amarantus, kukuruz i spelta jasno i nedvosmisleno klasifikuju i diferenciraju u grupe uzoraka brašna svake vrste. S obzirom na visok stepen botaničke srodnosti sortimenta strnih žita, uzorci brašna svih vrsta pšenice, ječma, ovsa, raži i tritikalea čine jednu zasebnu grupaciju.

**Binary system application in multivariate analysis of hexane extracts of cereal and pseudocereal flour**

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In this work hexane flour extracts of different varieties of buckwheat, amaranth, spelt, corn and small grains (wheat, barley, oats, rye and triticale) were analyzed. Lipid components extracted with hexane were derivatized using transesterification reagent, trimethylsulfonium hydroxide (TMSH) in 0.2M methanol. The system of gas chromatography with mass spectrometry was used to identify dominant and minor fatty acid methyl esters and unsaponifiable compounds: phytosterols, tocopherol and squalene, present in hexane extracts of cereals and pseudocereals. The dominant fatty acid methyl esters, present in hexane extracts of all investigated varieties, were eliminated. Applying the binary system and the multivariate analysis to automatically integrated areas of extracted components we examined the possibility of differentiation of cereal and pseudocereal flour samples. The results show that buckwheat, amarantus, corn and spelt could be clearly and unambiguously classified and differentiated into groups of samples of each type of flour. Because of high botanical similarity of small grains assortment, flour samples of all types of wheat, barley, oats, rye and triticale were classified into a separate group.

## **Hemija i tehnologija makromolekula** Chemistry and Technology of Macromolecules

HTM P 1

### **Epoksidni nanokompoziti na bazi organoglina**

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Mehanička, reološka i antikoroziivna svojstva polimera se mogu značajno poboljšati dodatkom malih količina nanoglina. Svojstva nanokompozita polimer/nanoglina prvenstveno zavise od stepena dispergovanosti nanogline u polimernoj matrici. U ovom radu, pripremljena je serija nanokompozita na bazi epoksidne smole (diglicidil-etar bisfenola A), poliamido-amina kao umreživača i 1 mas.% hemijski modifikovanih glina. Korišćene su dve komercijalne organogline (Cloisite 30B; Cloisite 15A) i jedna sintetisana u našoj laboratoriji sa heksadecilaminom kao modifikatorom. Nanokompoziti su pripremani direktnim mešanjem gline i epoksidne smole (DM) i prethodnim dispergovanjem gline u smeši organskih rastvarača (SM). Na osnovu oglada bubrenja i reoloških merenja konstatovano je da polarnost modifikatora gline određuje stepen bubrenja organoglina u smeši rastvarača, kao i elastična svojstva formirane fizičke mreže čestica gline. Optička mikroskopija i reološka merenja su potvrdila veći stepen dispergovanosti gline i bolja reološka svojstva neumreženih nanokompozita pripremljenih SM metodom u odnosu na DM metodu. Dinamičko-mehanička analiza je pokazala da dodatak gline sa modifikatorom koji sadrži hidroksilne grupe, koristeći SM metodu, dovodi do najvećeg poboljšanja mehaničkih svojstava umrežene epoksidne smole u staklastom stanju. Postupak pripreme i tip modifikatora gline nisu bitno uticali na temperaturu ostakljivanja umreženih nanokompozita.

### **Epoxy nanocomposites based on organoclays**

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The mechanical, rheological and anticorrosion properties of polymers can be significantly improved by incorporation of small amounts of nanoclays. The properties of polymer/clay nanocomposites (NCs) are primarily dependent on the dispersion degree of nanoclays. In this work, the series of epoxy NCs based on diglycidyl ether of bisphenol A epoxy resin, polyamidoamine curing agent and 1 wt% of chemically modified clays was prepared. Two commercial organoclays (Cloisite 30B; Cloisite 15A) and one synthesized in our laboratory with hexadecylamine as clay modifier were used. Clays were incorporated by either direct mixing with epoxy resin (DM) or by previous swelling of clay in a thinner (SM). Based on the "free swelling" test and rheological analysis, the swelling capacity of clays in the thinner and elastic properties of formed physical network of clay were dependent on the type of clay modifier. The optical microscopy confirmed higher dispersion degree of clays in the uncured NCs prepared with the SM in comparison to the DM method, which also had more improved rheological properties. The dynamic-mechanical analyses showed that incorporation of clay with modifier that contained hydroxyl groups, using SM method, led to the highest improvement of mechanical properties of cured epoxy resin in the glassy state. The preparation method and the type of clay modifier slightly affect the glass transition temperature of cured NCs.

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HTM P 2

**Nanokompoziti biodegradabilnog poliestra sa glinama modifikovanim  
heksadecilaminom i poli(etilen-oksidom)**

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Biodegradabilni polimeri, a posebno alifatski poliestri, kao ekološki pogodni materijali postali su aktuelno polje istraživanja i jedno od rešenja rastućih problema zaštite životne sredine. Mehanička, barijerna i termička svojstva alifatskih poliestara se mogu značajno poboljšati dodavanjem punioca nano-dimenzija, kao što je prirodni montmorilonit (glina). Da bi se poboljšala kompatibilnost sa polimernom matricom i obezbedila dobra disperzija punioca, neophodno je modifikovati površinu gline pogodnim organskim modifikatorima. U okviru ovog radu urađena je modifikacija gline (Cloisite®Na<sup>+</sup>) organskim modifikatorima: heksadecilaminom, HDA, i poli(etilen-oksidom) sa jednom završnom amino grupom, PEO-NH<sub>2</sub>. Uspješnost modifikacije gline potvrđena je WAXS analizom (povećanje *d*-rastojanja) i TG analizom. Modifikovane gline su korišćene za pripremu nanokompozita na bazi biodegradabilnog poliestra, metodom iz rastvora u hloroformu. Kao polimerna matrica korišćen je diblok kopolimer poli( $\epsilon$ -kapolakton)-poli(etilen-oksid), PCL/PEO, kod koga je PEO blok bio molarne mase 750 g/mol, a PCL blok 22000 g/mol. Sadržaj nanogline je bio konstantan i iznosio je 3,2 mas.%. WAXS analizom i reološkim merenjima u stanju rastopa utvrđena je bolja disperzija nanogline modifikovanih hidrofobnim HDA, u odnosu na nanogline bez modifikatora ili modifikovane sa PEO-NH<sub>2</sub>. Termička svojstva nanokompozita su ispitana DSC i TG analizom.

**Nanocomposites of biodegradable polyester with clays modified with  
hexadecylamine and poly(ethylene oxide)**

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Biodegradable polymers, especially aliphatic polyesters are in the focus of current research as a potential solution for the growing environmental pollution. Mechanical, thermal or barrier properties of aliphatic polyesters can be greatly improved with the addition of small amount of nano-filler, such as natural montmorillonite (clay). In order to achieve good dispersion of the filler in the polymer matrix, the surface of clay is modified with suitable organic modifiers. Within the frame of this work, surface modification of the natural clay (Cloisite®Na<sup>+</sup>) was performed, by using hexadecylamine, HDA, and poly(ethylene oxide) with amino end group, PEO-NH<sub>2</sub>, as clay modifiers. The efficiency of the modification was confirmed by WAXS and TG measurements. The obtained modified clays were used for the preparation of nanocomposites with biodegradable polyester, by solution casting. The polymer matrix was diblock copolymer poly( $\epsilon$ -caprolactone)-poly(ethylene oxide), PCL/PEO, with PCL of 22000 g/mol and PEO of 750 g/mol. The amount of the nanoclays was fixed at 3.2 wt.%. WAXS and melt rheology measurements confirmed better dispersion for the clay modified with hydrophobic HDA, in comparison to the unmodified or the clays modified with PEO-NH<sub>2</sub>. Thermal properties of the nanocomposites were evaluated by using DSC and TG analysis.

*Rad je finansiran od strane Ministarstva prosvete i nauke Republike Srbije: Projekat 172062*



## HTM P 3

**Triblok i diblok PCL kopolimeri sa malim sadržajem PEO segmenta: termička, površinska i morfološka svojstva**

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Dve serije, jedna triblok (PCL/PEO/PCL) i druga diblok (PCL/PEO) kopolimera su sintetisane polimerizacijom otvaranja prstena  $\epsilon$ -kaprolaktona, upotrebom dihidroksi i monohidroksi poli(etilen-oksida) kao makroinicijatora i kalaj(II)-oktoata kao katalizatora. Dužina PEO bloka je bila fiksirana dok je dužina PCL bloka podešavana promenom molskog odnosa  $\epsilon$ -CL/PEO. Struktura kopolimera je potvrđena  $^1\text{H}$  NMR spektroskopijom, a molarna masa je određena GPC analizom. Termička svojstva i stepen kristaliničnosti kopolimera su ispitivani pomoću DSC i WAXS. Kopolimeri su semikristalinični sa ortorombičnom kristalnom rešetkom PCL-a. Morfologija površine kopolimernih filmova je ispitana optičkom mikroskopijom i AFM analizom, dok su apsorpcija vode i apsorpcija vlage testirani u rastvoru fosfatnog pufera i izlaganjem atmosferi vlažnosti 97 %. Rezultati ukazuju da mali sadržaj PEO segmenta utiče na termičku degradaciju, kristaliničnost i morfologiju kopolimernih uzoraka. Optička mikroskopija i AFM analiza su potvrdile lamelarnu strukturu sferulita. Hrapavost kopolimernih filmova je uslovljena sadržajem PEO i prečnikom sferulita.

**Triblock and diblock PCL copolymers with a low content of PEO segment: the thermal, surface and morphological properties**

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Two series, one of triblock (PCL/PEO/PCL) and the other of diblock (PCL/PEO) copolymers were synthesized by ring-opening polymerization of  $\epsilon$ -caprolactone, by using dihydroxy or monohydroxy poly(ethylene oxide) as the macroinitiators and tin(II) octoate as the catalyst. The PEO block length was fixed while the PCL block lengths were tailored by changing molar ratio of  $\epsilon$ -CL/PEO. The copolymers' structure was confirmed by  $^1\text{HNMR}$  spectroscopy while molecular weights were determined by GPC analysis. The thermal properties and the degree of crystallinity of the copolymers were investigated by DSC and WAXS. Copolymers were semicrystalline with the orthorhombic PCL crystal lattice. The surface morphology of the copolymer films was investigated by using optical microscopy and AFM analysis, while water absorption and moisture uptake properties were tested in a phosphate buffer solution and by exposure to an atmosphere of 97 % relative humidity. Data indicated that a low content of PEO segment had an influence on thermal degradation behavior, crystallinity and morphology of copolymer samples. Optical microscopy and AFM analysis confirmed the spherulitic lamellar structure. Roughness of copolymer films were affected by the content of PEO and by the spherulites' diameter.

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НТМ Р 4

**Могућност примене полимерног емулгатора Cithrol DPHS  
за стабилизацију В/У емулзија**

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Емулзије типа вода у уљу (В/У) су дисперзни системи, који се као готови производи, или носачи активних материја, често користе у фармацеутској и прехранбеној индустрији. Познато је да се ради о изузетно нестабилним системима, код којих често долази до сепарације фаза и других облика нестабилности, тако да избор емулгатора и особине уљне фазе представљају веома важне факторе који дефинишу њихову стабилност.

Циљ овог рада био је испитивање могућности примене липофилног, полимерног емулгатора, Cithrol DPHS-а, за стабилизацију емулзија В/У погодних за примену у фармацеутској индустрији. Спроведена испитивања обухватила су дефинисање међуфазног понашања емулгатора применом тензиометрије, као и испитивање особина 20% (м/м) емулзија воде у парафинском и маслиновом уљу, стабилованих са 1% (м/м) Cithrol DPHS. На основу резултата испитивања особина добијених емулзија (величина и расподела величина капи и седиментациона стабилност у току 30 дана) изведени су одговарајући закључци.

*Раг је проистекао из испитивања на пројекту бр. III 46010/3, који финансира Министарство просвете, науке и технолошког развоја Републике Србије.*

**The possibility of using polymeric emulsifier Cithrol DPHS for stabilization  
of W/O emulsions**

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Water-in-oil (W/O) emulsions are dispersed systems that are often used in the pharmaceutical and food industries as products, or as carriers of active substances. It is well known that they are very unstable systems, so that the selection of emulsifier and the properties of the oil and water phase are the main factors affecting their stability.

The aim of this paper was to examine the possibility of application the lipophilic, polymeric emulsifier, Cithrol DPHS, for stabilization of W/O emulsions that are suitable for use in pharmaceuticals. The behavior of the emulsifier at W/O interphases was determined by means of tensiometry. The characteristics of 20% (w/w) emulsions of water in paraffin oil and in olive oil (droplet size and size distribution and stability during 30 days of storage), which were stabilized with 1% (w/w) Cithrol DPHS, were investigated too. On the basis of obtained results appropriate conclusions were made.

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## Organska hemija / Organic Chemistry

OH O 1

**Toksičnost i anti-acetilholinesterazna aktivnost novih hibridnih jedinjenja ferocena i indola**

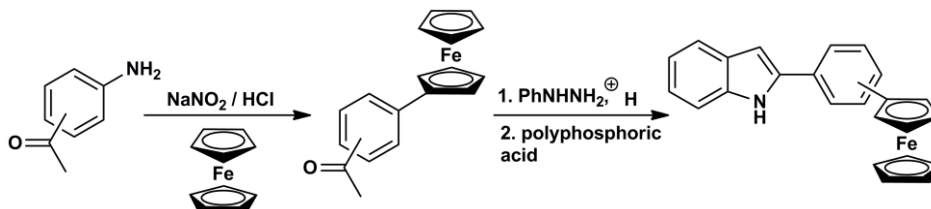
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Nedavno je utvrđeno da se citotoksičnost supstituisanih 2-fenilindola drastično povećava uvođenjem ferocenskog jezgra u molekul. U potrazi za novim citotoksičnim agensima sintetisali smo dva nova hibridna jedinjenja ferocena i indola, 2-(3-ferocenilfenil)-1H-indol i 2-(4-ferocenilfenil)-1H-indol, koristeći Fišerovu sintezu indola kao ključni korak sinteze. Oba jedinjenja pokazala su slabu anti-acetilholinesteraznu aktivnost, ali i jaku citotoksičnu aktivnost na makrofage peritoneuma pacova i slanovodne račice *Artemia salina*. Takođe, oba jedinjenja su u značajnoj meri inhibirala mijeloperoksidaznu aktivnost što sugeriše njihovu potencijalnu primenu za lečenje upalnih procesa. Dobijeni rezultati bioloških testova vrlo su ohrabrujući i ukazuju na moguću citotoksičnu aktivnost ovih jedinjenja i na ljudske ćelije raka.

**Toxicity and acetylcholinesterase inhibiting activity of novel ferrocene-indole hybrids**

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It has been recently reported, that an incorporated ferrocene unit into the 2-phenylindole scaffold can drastically improve the cytotoxic activity of the parent compounds. In our search for new promising cytotoxic agents we have designed and prepared two novel ferrocene-indole hybrids, 2-(3-ferrocenylphenyl)-1H-indole and 2-(4-ferrocenylphenyl)-1H-indole, utilizing the Fischer indole synthesis as the key step. Both compounds have showed weak anticholinesterase activity but high cytotoxicity against rat peritoneal macrophages and crustacean *Artemia salina*. Also, both compounds have showed significant myeloperoxidase inhibiting activity, thus suggesting a potential use in inflammatory disorders. The results of these tests are very encouraging as they also suggest possible cytotoxic activities of these compounds against human cancer cell lines.



OH O 2

**Интеракције 5-арил-1H-пиразол-3-карбоксилних киселина  
са четири изоформе хумане карбо-анхидразе из перспективе  
молекулног моделовања**

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Укратко су описане вероватне интеракције конгенерне серије 23 5-арил-1H-пиразол-3-карбоксилне киселине са четири изоформе хумане карбо-анхидразе. Инхибиторна активност једињења према четири изоформе хумане карбо-анхидразе (I, II, IX и XII) је експериментално одређена. Једињења инхибирају изоформе IX и XII хумане карбо-анхидразе у микромоларном опсегу концентрација, док су према изоформама I и II неактивна у концентрацијама до 50 микромола ( $K_i$  вредности). Различите групе једињења у оквиру испитиваног сета инхибирају изоформу IX или изоформу XII хумане карбо-анхидразе. Разматрајући само структуру испитиваних једињења немогуће је објаснити селективност. Студије молекулног докинга, користећи 3Д структуре *целих* ензима као мете, су успешно објасниле селективност испитиваних једињења према изоформама IX и XII хумане карбо-анхидразе, као и селективност различитих група једињења према изоформи IX или XII.

**Interactions of 5-aryl-1H-pyrazole-3-carboxylic acids with four  
human carbonic anhydrase isoforms - a molecular modelling perspective**

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We describe *in silico* modelled interactions between 23 5-aryl-1H-pyrazole-3-carboxylic acids and four isozymes (I, II, IX and XII) of human carbonic anhydrase (hCA). Inhibitory activity of compounds were experimentally determined. Compounds inhibited hCA IX and XII in micromolar range of concentrations. Compounds were proved as inactive toward hCA I and II in concentrations up to 50  $\mu$ M ( $K_i$  values). Different subsets of compounds inhibited hCA IX or hCA XII. It appears as impossible to provide rationale for experimentally determined selectivity by considering structure of compounds only. Molecular docking on whole isozyme structures explained strong inhibitory preferences of compounds toward hCA IX and XII over hCA I and II, as well as preferences of different subsets toward hCA IX or hCA XII.

## OHP 1

**Sinteza i biološka aktivnost 5-supstutuisanih derivata benzimidazola**

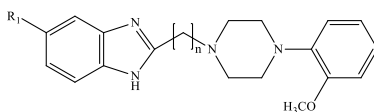
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 Slađana V. Kostić-Rajačić

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U cilju proučavanja interakcija između liganda i vezivnog mesta dopaminskog D<sub>2</sub> receptora, sintetisana je serija 5-supstutuisanih derivata benzimidazola. Zahvaljujuci različitoj dužini i fleksibilnosti mosta između glave (benzimidazolski deo molekula) i repa (arilpiperazinski deo molekula), novosintetisani ligandi se različito orijentišu u vezivnom mestu receptora.

Za sve novosintetisane ligande ispitan je afinitet vezivanja za dopaminski (D<sub>2</sub>) receptor u *in vitro* eksperimentima kompeticije sa [<sup>3</sup>H]-spiperonom kao radioaktivnim ligandom. Sinaptosomalne membrane izolovane su iz strijatuma mozga pacova.



R	H	H	H	H	H	H	Cl	Cl	Cl
n	1	2	3	4	5	6	4	5	6

**Synthesis and biological activity of 5-substituted benzimidazole derivatives**

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 Slađana V. Kostić-Rajačić

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A series of 5-substituted benzimidazole derivatives were synthesized in order to further examine the interaction between the ligand and the binding site of the dopamine (D<sub>2</sub>) receptor. Due to the different length and flexibility of the bridge between the head (the benzimidazole moiety) and the tail (the arylpiperazine part of the molecule), newly synthesized ligands will be oriented differently in the binding site of the receptor.

Dopaminergic activities of newly synthesized ligands were estimated by *in vitro* competition binding experiments using [<sup>3</sup>H]-spiperone as the radioligand.

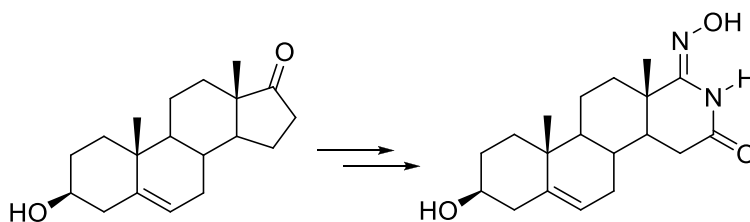
*This work was supported by the Ministry of Education and Science of the Republic of Serbia ( project No. 172032)*

## OHP 2

**Sinteza novog steroidnog heterocikličnog derivata**Aleksandar M. Oklješa, Andrea R. Nikolić, Dušan Đ. Škorić, Marija N. Sakač,  
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Steroidni oksimi<sup>1</sup> i D-homo heterociklični derivati<sup>2,3</sup> ispoljavaju antiproliferativnu aktivnost prema ćelijskim linijama različitih humanih karcinoma. Cilj ovog rada bio je sinteza novog 17-aza-D-homo derivata androstanske serije, potencijalnog antitumorskog agensa. Polazno jedinjenje u ovoj višefaznoj sintezi bio je dehidroepiandrosteron. Struktura novosintetizovanog jedinjenja utvrđena je NMR spektroskopijom i masenom spektrometrijom visoke rezolucije (TOF). Pomoću HMBC NMR eksperimenta utvrđeno je da se u položaju C-16 nalazi okso grupa, a da se u položaju C-17a nalazi hidroksiimino grupa.

Realizacija ovog rada finansirana je iz sredstava projekata ON172021 i 114-451-1104/2014-02

**Synthesis of a new steroidal heterocyclic derivative**Aleksandar M. Oklješa, Andrea R. Nikolić, Dušan Đ. Škorić, Marija N. Sakač, Evgenija A.  
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Steroidal oximes<sup>1</sup> and D-homo heterocyclic derivatives<sup>2,3</sup> display antiproliferative activity against various human carcinoma cell lines. The aim of this work was synthesis of a new 17-aza-D-homo androstane derivative, as a potential antitumor agent. The starting compound in this multistep synthesis was dehydroepiandrosterone. The structure of the newly synthesised compound was determined by NMR and HRMS (TOF). Positions of oxo and oximino groups were confirmed by 2D HMBC- NMR experiment.

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## OHP 3

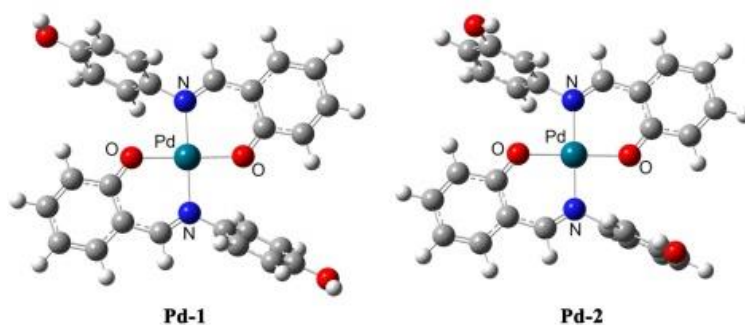
## Sinteza i biološka aktivnost Pd(II)-kompleksa salicilaldehidno-anilinskih Šifovih baza

Dušica Simijonović, Zorica D. Petrović, Vladimir P. Petrović, Marko N. Živanović\*,  
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Sintetizovane su salicilaldehidne Šifove baze, 2-(((4-hidroksifenil)imino)metil)fenol (**1**) i 2-(((3-hidroksifenil)imino)metil)fenol (**2**) i njihovi odgovarajući Pd(II)-kompleksi (**Pd-1** i **Pd-2**), slika 1. Za razliku od liganada, oba kompleksa ispoljavaju veliku citotoksičnost prema ispitivanim ćelijskim linijama raka debelog creva (HCT-116) i raka dojke (MDA-MB-231).



Slika 1. Strukture kompleksa  
Figure 1. Structures of the complexes

Tabela 1. Citotoksični efekti ispitivanih uzoraka  
Table 1. Cytotoxic effects of examined compound

IC <sub>50</sub> , μM	HCT-116		MDA-MB-231	
	24 h	72 h	24 h	72 h
<b>1</b>	142.3	368.0	440.2	133.6
<b>Pd-1</b>	11.8	17.1	276.9	7.2
<b>2</b>	>500	277.6	>500	>500
<b>Pd-2</b>	5.8	0.6	55.6	40.7

## Synthesis and biological activity of Pd(II)-complexes derived from salicylaldehyde-aniline Schiff bases

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Salicylaldehyde Schiff bases, 2-(((4-hydroxyphenyl)imino)methyl)phenol (**1**) and 2-(((3-hydroxyphenyl)imino)methyl)phenol (**2**) and their corresponding Pd(II)-complexes (**Pd-1** and **Pd-2**) were synthesized, Fig. 1. Both complexes exert outstanding cytotoxic character, while ligands are much less cytotoxic on the human colon cancer cell line, HCT-116 and breast cancer cell line, MDA-MB-231.

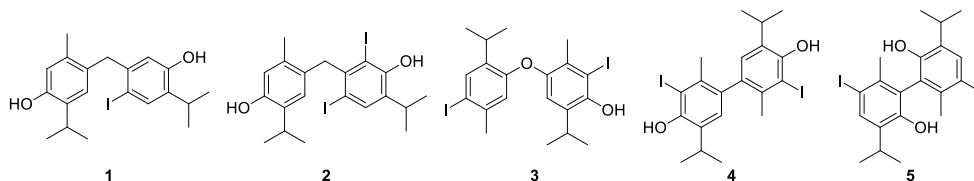
## OHP 4

**Revizija strukture aristola: novo lateralno oksidativno kuplovanje timola**

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Aristol je oficinalni antiseptik, prisutan na tržištu oko 130 godina, ali je njegova struktura i dalje nedovoljno proučena. Većina farmakopeja i komercijanih izvora ga vodi kao 4,4'-bis(jodoksi)-2,2'-dimetil-5,5'-bis(1-metiletil)-1,1'-bifenil, iako ne postoje dokazi da je ova struktura ispravna. Ovo nas je motivisalo da pripremimo aristol po proceduri iz *Annales de Chimie analytique*, Vol. 14, 1909, p. 228, i pokušamo da odredimo njegovu tačnu strukturu. Hromatografijom reakcione smeše dobijene tretiranjem timola jodom u alkalnom rastvoru, pored čistih mono- i diiodotimola, dobijeno je nekoliko proizvoda oksidativnog kuplovanja (**1-5**). Njihova struktura je nedvosmisleno određena na osnovu spektralnih podataka (MS, IR, UV-Vis, 1D- i 2D-NMR). Jedinjenja **2-5** imala su molekulska masu (550) kao i komercijalno dostupan aristol. Može se zaključiti da je aristol najverovatnije smeša više jedinjenja, čime se mogu objasniti teškoće u utvrđivanju samo jedne strukture koja bi objasnila sve osobine aristola. Među proizvodima kuplovanja, jedinjenja **1** i **2** predstavljaju dosada nezabeležene proizvode oksidativnog lateralnog *meta*-kuplovanja (u odnosu na fenoksi-radikal) bilo kog fenola. Mehanizam kojim nastaju ovi proizvodi verovatno uključuje benzilne radikale za koje se nije znalo da nastaju pod ovim uslovima.

**Structural revision of aristol: a new lateral oxidative coupling of thymol**

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Aristol is an antiseptic that has been on the market for about 130 years, but its structure has never been investigated in full detail. Most pharmacopeias and commercial sources list it as 4,4'-bis(iodooxy)-2,2'-dimethyl-5,5'-bis(1-methylethyl)-1,1'-biphenyl although there is no conclusive proof that supports this structure. This motivated us to synthesize aristol in a traditional way, according to *Annales de Chimie analytique*, Vol. 14, 1909, p. 228, and try to determine its structure. Chromatography of the reaction mixture obtained by treatment of thymol with iodine in alkaline solution yielded, along with mono- and diiodothymols, several pure products of oxidative coupling (**1-5**). The structure of these compounds was unequivocally established based on extensive spectral data. Compounds **2-5** have the same molecular weight (550) as commercial aristol. One can conclude that aristol most probably represents a mixture of several compounds, which in turn explains the difficulties encountered during its structural elucidation. Among the identified compounds, **1** and **2** represent unreported products of oxidative lateral *meta*-coupling (with respect to the phenoxy radical) of any phenol. The mechanism of formation of **1** and **2** probably includes benzyl radicals that were not known to be formed under these conditions.

**Acknowledgement:** This work was funded by the Ministry of Education, Science, and Technological Development of Serbia (Project 172061).



OH P 5

**Skринing antibakterijske aktivnosti D-modifikovanih derivata androстана**

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Siniša L. Markov\*, Evgenija A. Đurendić, Marija N. Sakač, Katarina M. Penov-Gaši  
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Modifikacija steroidnih jedinjenja uvođenjem novih funkcionalnih grupa, može rezultirati u promeni farmakoloških svojstava polaznog molekula. Cilj rada je bio ispitivanje antibakterijske aktivnosti petnaest D-modifikovanih derivata androстана. Test mikroorganizmi bili su referentni sojevi Gram-pozitivnih (*Staphylococcus aureus* ATCC 25923, *Bacillus cereus* ATCC 10876 i *Listeria monocytogenes* ATCC 13932) i Gram-negativnih (*Escherichia coli* ATCC 8739 i *Pseudomonas aeruginosa* ATCC 27853) bakterija. Antibakterijska aktivnost je ispitana disk difuzionom metodom koristeći 15 µl derivata koncentracije 10 mg/ml. Najveću otpornost pokazale su Gram-negativne bakterije prema kojima ni jedan od ispitanih derivata nije ispoljio aktivnost. Od Gram-pozitivnih bakterija najosetljivija je bila *S. aureus* na koju je deset derivata delovalo baktericidno, a najotpornija *L. monocytogenes* prema kojoj su samo četiri derivata ispoljila bakteriostatsku aktivnost.

**Screening of antibacterial activity of D-modified androstane derivatives**

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Siniša L. Markov\*, Evgenija A. Đurendić, Marija N. Sakač, Katarina M. Penov-Gaši  
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Modification of steroidal compounds by connecting a special functionality to the core of steroid can result in altered pharmacological properties. The aim of this study was to screen antibacterial activity of fifteen D-modified androstane derivatives. Tested microorganisms were reference strains of Gram-positive (*Staphylococcus aureus* ATCC 25923, *Bacillus cereus* ATCC 10876 and *Listeria monocytogenes* ATCC 13932) and Gram-negative (*Escherichia coli* ATCC 8739 and *Pseudomonas aeruginosa* ATCC 27853) bacteria. Antibacterial activity was tested in concentration of 10 mg/ml by disc diffusion method using 15 µl of derivatives. Gram-negative bacteria were the most resistant strains, because there was no activity of any tested derivatives. Ten derivatives showed bactericidal activity towards *S. aureus*, which was the most susceptible among Gram-positive bacteria, while *L. monocytogenes* was the most resistant. Only four derivatives showed bacteriostatic activity to this strain.

*This work was supported by a grant from the Ministry of Education, Science and Technological Development of Republic of Serbia (Project No. ON172021) and Provincial Secretariat for Science and Technological Development (Project No. 114-451-1104/2014-02).*

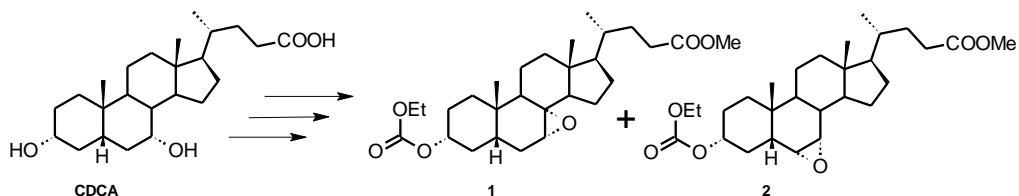
## OH P 6

**Sinteza epoksida iz henodezoksiholne kiseline**

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Polazeći od henodezoksiholne kiseline (CDCA) izvršena je sinteza metil-3 $\alpha$ -[(etoksikarbonil)oksi]-7 $\alpha$ ,8 $\alpha$ -epoksi-5 $\beta$ -holan-24-oata (**1**), a kao sporedni proizvod dobijen je metil-3 $\alpha$ -[(etoksikarbonil)oksi]-6 $\alpha$ ,7 $\alpha$ -epoksi-5 $\beta$ -holan-24-oat (**2**). Najpre je izvršena selektivna zaštita OH grupe u položaju 3, a zatim je izvršena eliminacija slobodne OH grupe u položaju 7 koja je dala pretežno  $\Delta^7$  olefin dok se  $\Delta^6$  olefin javlja kao sporedni proizvod. Ključni korak u sintezi je reakcija epoksidacije sa MCPBA (*m*-hlorperbenzoeva kiselina) u kojoj nastaju pomenuti epoksidi **1** i **2**. Stereochemija sintetisanih epoksida potvrđena je 1D i 2D NOESY NMR eksperimentima. Dobijeni epoksidni derivati mogu biti korisni intermedijeri u sintezi više derivata žučnih kiselina reakcijama otvaranja epoksida.



*Autori se zahvaljuju Pokrajinskom sekretarijatu za nauku i tehnološki razvoj AP Vojvodine (ugovor broj 114-451-999/2014-2) na finansijskoj pomoći u izradi ovog rada.*

**Synthesis of some epoxides from chenodeoxycholic acid**

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Starting from chenodeoxycholic acid (CDCA) the synthesis of methyl 3 $\alpha$ -[(ethoxycarbonyl)oxy]-7 $\alpha$ ,8 $\alpha$ -epoxy-5 $\beta$ -cholan-24-oate **1** was accomplished, while methyl 3 $\alpha$ -[(ethoxycarbonyl)oxy]-6 $\alpha$ ,7 $\alpha$ -epoxy-5 $\beta$ -cholan-24-oate **2** was obtained as a side product. First, selective protection of OH group in position 3 was performed, then the  $\Delta^7$  double bond was introduced by elimination of free OH group in position 7. A smaller quantity of  $\Delta^6$  olefin also was identified. The key step in this synthesis was an epoxydation reaction with MCPBA, which afforded epoxides **1** and **2**. Stereochemistry of the synthesized epoxides was elucidated with the aid of 1D and 2D NOESY NMR experiments. The obtained epoxy-derivatives could be good starting materials in the synthesis of some new bile acid derivatives through epoxide opening reactions.

*Authors would like to thank the Provincial Secretariat for Science and Technological Development of AP Vojvodina (Contract No. 114-451-999/2014-2) for the financial support of this work.*

## Funkcionalizacija 2-tio-1,2,3,4-tetrahidropirimidina i sinteza novih halkona bez prisustva rastvarača

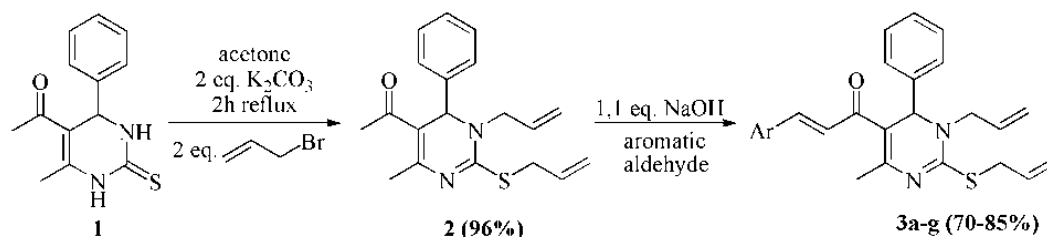
Nenad Janković, Vesna Stanjlović, Jelena Petronijević, Nenad Joksimović, Zorica Bugarić  
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2-tio-1,2,3,4-tetrahidropirimidin (**1**) i njegovi analozi predstavljaju klasu heterocikličnih molekula koji su privukli veliko interesovanje medicinske hemije.<sup>1,2</sup> Uprkos činjenici da 2-tio-1,2,3,4-tetrahidropirimidini imaju širok spektar bioloških aktivnosti poznato je samo nekoliko metoda za njihovu funkcionalizaciju. U ovom radu, predstavljamo reakciju dvostrukog alkilovanja jedinjenja **1** pomoću alil-bromida (Shema 1). Zatim, opisujemo sintezu novih halkona (**3a-g**) preko funkcionalizacije jedinjenja **2** sa odabranim aromatičnim aldehidima bez prisustva rastvarača.

## Functionalization of 2-thio-1,2,3,4-tetrahydropyrimidine and synthesis of novel chalcones under solvent-free conditions

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The 2-thio-1,2,3,4-tetrahydropyrimidine (**1**) and their analogs, which represent a class of heterocyclic molecules, have attracted a considerable interest in medicinal chemistry.<sup>1,2</sup> Despite the fact that 2-thio-1,2,3,4-tetrahydropyrimidines have a wide range of biologically activities, there are only few known methods for their functionalization. Here, we present a reaction for double alkylation of compound **1** with allyl-bromide (Scheme 1). Then, we describe a reaction for the syntheses of novel chalcones (**3a-g**) via functionalization of **2** with some aromatic aldehydes under solvent-free conditions.



**aromatic aldehyde** = benzaldehyde (**a**), 4-nitrobenzaldehyde (**b**), 4-(benzyloxy)benzaldehyde (**c**), furfural (**d**), vanillin (**e**), 3,4,5-trimethoxybenzaldehyde (**f**), cinnamaldehyde (**g**)

**Scheme 1.** Solvent-free synthesis of chalcones

**Acknowledgements:** The authors are grateful to the Ministry of Education, Science and Technological Development of the Republic of Serbia for financial support (Grant 172011)

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## OH P 8

### Sinteza i antitumorska aktivnost novog pseudo-C-nukleozida sa thiazolkarboksamidnim aglikonom

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Ostvarena je višefazna sinteza novih pseudo C-nukleozida (**8** i **10**, Shema 1) polazeći od D-glukoze nizom selektivnih hemijskih transformacija. Ispitana je *in vitro* citotoksična aktivnost novosintetizovanih jedinjenja prema više humanih malignih ćelijskih linija, kao i prema ćelijama normalnih fibroblasta pluća (MRC-5).

### Synthesis and antitumour activity of new pseudo-C-nucleosides bearing thiazolcarboxamide aglicone

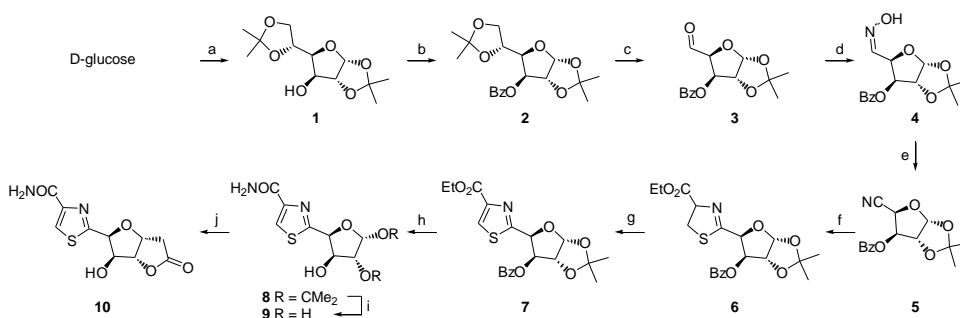
Miloš M. Svirčev, Mirjana M. Popsavin, Ivana M. Kovačević, Saša B. Spaić, Bojana M. Srećo Zelenović, Vesna V. Kojić\*, Milka B. Jadranin\*\*, Marko V. Rodić, Velimir J. Popsavin

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A multistep synthesis of two novel pseudo C-nucleosides (**8** and **10**, Scheme 1) has been achieved starting from D-glucose, via a number of selective chemical transformations. Both targets **8** and **10** were evaluated for their *in vitro* cytotoxicity against several human malignant cell lines, as well as against normal foetal lung fibroblasts (MRC-5).



**Scheme 1.** Reagents and conditions: (a) conc. H<sub>2</sub>SO<sub>4</sub>, dry Me<sub>2</sub>CO; (b) BzCl, Py, CH<sub>2</sub>Cl<sub>2</sub>; (c) H<sub>5</sub>IO<sub>6</sub>, dry EtOAc; (d) NH<sub>2</sub>OH × HCl, AcONa, EtOH; (e) MsCl, Py; (f) HSCH<sub>2</sub>CH(NH<sub>2</sub>)CO<sub>2</sub>Et × HCl, Et<sub>3</sub>N, MeOH; (g) CBrCl<sub>3</sub>, DBU, CH<sub>2</sub>Cl<sub>2</sub>; (h) NH<sub>3</sub>, MeOH; (i) TFA/H<sub>2</sub>O (9:1); (j) Meldrum's acid, DMF, Et<sub>3</sub>N.

**Acknowledgement:** The work was supported by a grant from the Ministry of Education, Science and Technological Development (Project 172006).

## OHP 9

**Sinteza i antitumorska aktivnost kardiobutanolida i njegovog (6S,7S)-izomera**

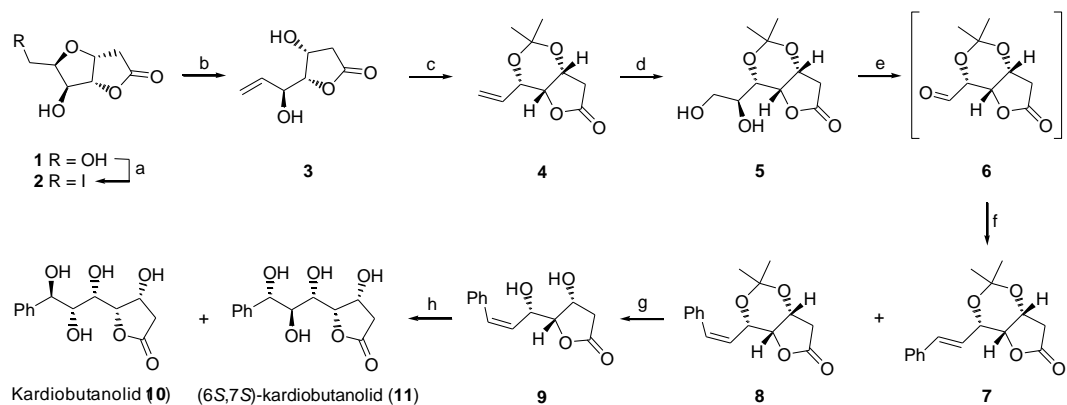
Ivana M. Kovačević, Mirjana Popsavin, Marko Rodić, Goran Benedeković, Vesna Kojić\*,  
Gordana Bogdanović\*, Velimir Popsavin  
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Kardiobutanolid (**10**) je prirodni stiril-lakton izolovan iz subtropske biljke *Goniothalamus cardiopetalus*<sup>1</sup>. Ovom prilikom želimo da saopštimo novu totalnu sintezu kardiobutanolida (**10**), kao i prvu sintezu njegovog (6S,7S)-stereoizomera **11** polazeći iz furanolaktona **1**, koji je lako pristupačan iz D-kсилоze. Pored toga, detaljno će biti prikazani i diskutovani rezultati ispitivanja antitumorske aktivnosti sintetizovanih jedinjenja prema odabranim humanim malignim ćelijskim linijama

**Synthesis and antitumour activity of cardiobutanolide and its (6S,7S)-isomer**

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Cardiobutanolide (**10**) is a naturally occurring styryl lactone isolated from the stem bark of *Goniothalamus cardiopetalus*<sup>1</sup>. Herein we report a new total synthesis of cardiobutanolide (**10**) as well as the first synthesis of its (6S,7S)-stereoisomer **11** starting from lactone **1**, which is readily available from D-xylose. Additionally, the results related to evaluation of antiproliferative activity of both final products against certain human tumour cell lines, will be presented and discussed in details.



**Shema 1.** (a)  $I_2$ ,  $Im$ ,  $THF$ ; (b) aktivirani  $Zn$ ,  $THF/H_2O$  (4:1); (c) 2,2-DMP,  $TsOH \cdot H_2O$ ,  $Me_2CO$ ; (d) 2.5 wt%  $OsO_4$  u  $^tBuOH$ ,  $NMO$ ,  $H_2O/Me_2CO$ ; (e)  $NaIO_4$ ,  $MeOH/H_2O$  (2:1); (f)  $Ph_3PCH_2PhBr$ ,  $LiHMDS$ , *anh.*  $THF$ ; (g)  $TFA/H_2O$  (1:1); (h) 2.5 wt%  $OsO_4$  u  $^tBuOH$ ,  $Me_2CO/H_2O$  (10:1).

**Acknowledgement:** The work was supported by a grant from the Ministry of Education, Science and Technological Development (Project 172006).

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## OHP 10

**Totalna sinteza (–)-kleistenolida iz diacetonida D-glukoze**

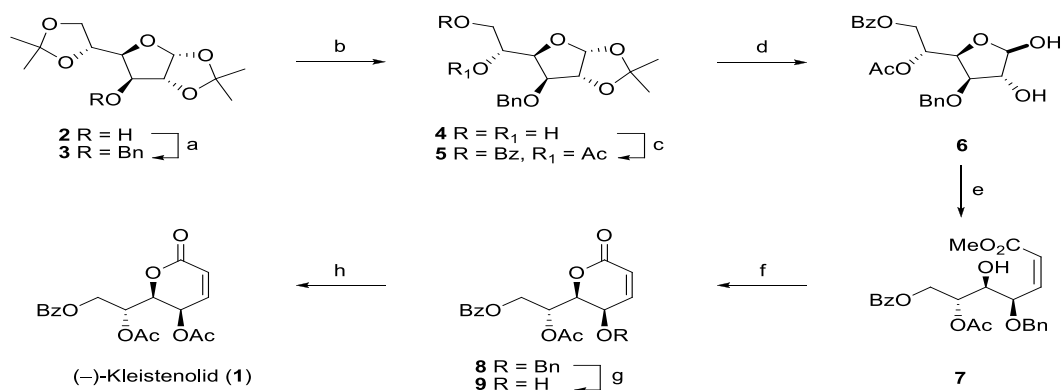
Goran Benedeković, Mirjana Popsavin, Jovana Francuz, Velimir Popsavin  
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(–)-Kleistenolid (**1**) je prirodni proizvod izolovan iz biljke *Cleistochlamys kirkii* poreklom iz Tanzanije i Mozambika. Jedinjenje **1** ima 5,6-dihidro-2H-piran-2-onsku strukturu i pokazuje antibakterijsku (*Staphylococcus aureus* i *Bacillus anthracis*) i antigljivičnu aktivnost (*Candida albicans*). U ovom radu želimo da saopštimo novu totalnu sintezu (–)-kleistenolida (**1**). Kao polazno jedinjenje primenjen je diol **4** koji dobijen iz diacetonida D-glukoze u dve sintetičke faze. Molekul **4** je preveden u intermedijerni Z-olefin **7** primenom višefazne sintetičke sekvencije koja je prikazana na reakcionoj shemi. Prirodni proizvod **1** se dobija nakon ciklizacije–debenzilovanja olefina **7** i naknadnog acetilovanja alkohola **9**.

**Total synthesis of (–)-cleistenolide from diacetone-D-glucose**

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(–)-Cleistenolide (**1**) is a natural product isolated from the plant *Cleistochlamys kirkii* originating from Tanzania and Mozambique. (–)-Cleistenolide has a 5,6-dihydro-2H-pyran-2-one structure and shows antibacterial activity against *Staphylococcus aureus* and *Bacillus anthracis*, as well as antifungal activity against *Candida albicans*. Herein we wish to report a new total synthesis of natural product **1**. Diol **4**, readily available from diacetonide D-glucose (**2**) over two steps, has served as a convenient starting material in this work. Molecule **4** was converted to the intermediary Z-olefine **7** through a multi-step sequence outlined in the reaction scheme. The natural products **1** can be accessed after cyclization-debenzylation of **7**, followed by final acetylation of alcohol **9**.



Reagents and conditions: (a) BnBr, NaH, DMF, 0°C, rt; (b) 60% AcOH, CH<sub>2</sub>Cl<sub>2</sub>, rt; (c) BzCl, DMAP, AcCl, CH<sub>2</sub>Cl<sub>2</sub>, rt; (d) 9:1 TFA/H<sub>2</sub>O, CH<sub>2</sub>Cl<sub>2</sub>, 0°C, rt; (e) (i) H<sub>3</sub>IO<sub>6</sub>, EtOAc/H<sub>2</sub>O; (ii) MeOH, MCMP, rt; (f) TsOH, CH<sub>2</sub>Cl<sub>2</sub>, rt; (g) TiCl<sub>4</sub>, CH<sub>2</sub>Cl<sub>2</sub>, rt; (h) TsOH, Ac<sub>2</sub>O, rt.

**Acknowledgement:** The work was supported by a grant from the Ministry of Education, Science and Technological Development (Project 172006).

## OHP 11

**Sinteza novih 4-ferocenil-1,2,3,4-tetrahidrohinolina**

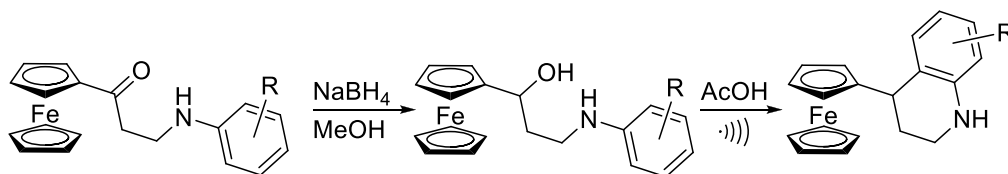
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Tetrahidrohinolinski prsten ulazi u sastav brojnih biološki aktivnih prirodnih proizvoda i važnih farmakoloških agenasa, zbog čega je razvoj metoda za dobijanje derivata ovog heterocikla privukla značajnu pažnju organskih hemičara.<sup>1</sup> U ovom radu biće opisana sinteza serije novih 4-ferocenil-1,2,3,4-tetrahidrohinolina. Ta sinteza ostvarena je polazeći od 3-(arilamino)-1-ferocenilpropan-1-ona (čija sinteza je ranije razvijena u našim laboratorijama),<sup>2</sup> koji su redukovani (NaBH<sub>4</sub>) do odgovarajućih 3-(arilamino)-1-ferocenilpropan-1-ola. Ovi poslednji tretirani su sirćetnom kiselinom u ultrazvučnom kupatilu dajući ciljna jedinjenja (79-99%) preko stabilnog α-ferocenil-karbokatjona.



R = H, CH<sub>3</sub>, F, Cl, OCH<sub>3</sub>,

**Synthesis of novel 4-ferrocenyl-1,2,3,4-tetrahydroquinolines**

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The tetrahydroquinoline motif makes up the core structure of numerous biologically active natural products and pharmacologically relevant therapeutic agents. Due to these facts the development of new methodologies for the synthesis of tetrahydroquinoline derivatives attracted nowadays enviable attention of organic chemists.<sup>1</sup> In the present report the synthesis and spectral characterization of a small series of novel 4-ferrocenyl-1,2,3,4-tetrahydroquinolines will be described. This synthesis was achieved starting from 3-(arylamino)-1-ferrocenylpropan-1-ones (recently described by us),<sup>2</sup> which were reduced (NaBH<sub>4</sub>) to the corresponding 3-(arylamino)-1-ferrocenylpropan-1-ols. The later were treated with acetic acid and irradiated in an ultrasonic bath to give the target molecules (79-99%) via a stable α-ferrocenyl carbocation.

1. V. Sridharan *et al.*, *Chem. Rev.* **111** (2011) 7157.
2. a) I. Damljanović *et al.*, *J. Organomet. Chem.* **696** (2011) 3703; b) A. Pejović *et al.*, *Helv. Chim. Acta*, **95** (2012) 1425.

## OHP 12

**Novi adamantanski derivati 4-aminohinolina:  
sinteza, ispitivanje *in vitro* i *in vivo* antimalarijske aktivnosti**

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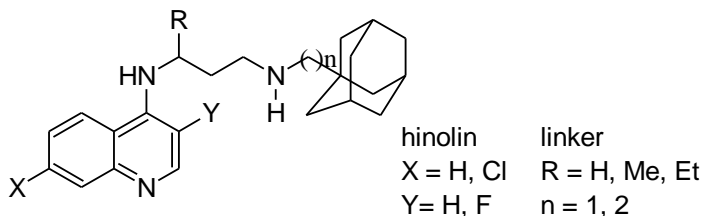
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Malaria je jedna od najsmrtonosnijih bolesti uzrokovana protozoičnim parazitima roda *Plasmodium*. Prema procenama Svetske zdravstvene organizacije (WHO), svake godine od malarije oboli preko 500 miliona ljudi od kojih umire preko milion. Nagli porast rezistentnosti parazita, pogotovo soja *P. falciparum* (malarija prouzrokovana ovim parazitom u 80% slučajeva dovodi do smrtnog ishoda) prema odobrenim terapijama stvara neprestanu potrebu za razvojem novih i efikasnijih antimalarika. Sintetisana je serija derivata u okviru koje su vršene modifikacije kako na linkeru (promena dužine) tako i na hinolinskom jezgru (uvođenje F u poziciju C3). Dobijenim jedinjenjima određena je *in vitro* antimalarijska aktivnost prema CQ-osetljivom (D6) i CQ-rezistentnim (W2 i TM91C235) sojevima *P. falciparum*-a i *in vivo* oralna aktivnost prema *P. berghei*.



**New adamantane 4-aminoquinoline derivatives:  
synthesis, *in vitro* and *in vivo* antimalarial activity**

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Malaria is one of the deadliest diseases caused by protozoan parasite *Plasmodium*. According to the World Health Organization (WHO) more than 500 million people are diagnosed with malaria every year and over one million dies from it. The sharp rise in parasite resistance, especially of strain *P. falciparum* towards the approved therapies creates a constant need for development of new and more effective antimalarials. Herein is presented the synthesis and the activity of a series of derivatives with adamantane carrier, in which the linker length and quinoline substitution (introduction of F in position C3) were varied. The obtained compounds were screened *in vitro* against CQ sensitive (D6) and CQ resistant (W2 and TM91C235) strains of *P. falciparum* and *in vivo* oral activity was obtained against *P. berghei*.

**Acknowledgement:** This research was supported by the Ministry of Education, Science and Technological Development of Serbia (grant no. 172008).



OHP 13

### **Sinteze hidroksamskih derivata individualnih i prirodnih naftnih kiselina**

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Hidroksamska funkcionalna grupa ulazi u sastav mnogih biološki aktivnih jedinjenja koja ispoljavaju širok spektar farmakoloških aktivnosti kao što su antiinflamatorna, antiastmatička, antimetastatička, antibiotska i psihotropna aktivnost. U ovom radu sintetisani su hidroksamski derivati individualnih i prirodnih naftnih kiselina „Velebit“ sa ciljem ispitivanja njihovog efekta na porast broja mikroorganizama. Hidroksamske kiseline su dobijene iz metil-estara naftnih kiselina koje su reagovale sa hidroksilaminom hidrohloridom i kalijum-hidroksidom u molskom odnosu 1:3:6. Sinteze su rađene u metanolnom rastvoru na temperaturi od 110 °C, primenom zatvorenog sistema mikro-talasnog reaktora. Optimizacija reakcione temperature i reakcionog vremena rađena je na metil-estru benzoeve kiseline kao model supstanci, praćenjem toka reakcije HPLC analizom. Svi dobijeni hidroksamski derivati prirodnih i individualnih naftnih kiselina su okarakterisani NMR i IR spektrima. Ostvareni prinosi se kreću u opsegu od 61-93% za reakciono vreme od jedne minute.

### **The syntheses of hydroxamic derivatives of individual and natural naphthenic acids**

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The hydroxamic functional group is part of many biologically active compounds that exhibit a wide range of pharmacological activities such as anti-inflammatory, antiasthmatic, antimetastatic, antibiotic and psychotropic. The aim of this study was synthesis of new hydroxamic derivatives from individual and natural naphthenic acids and evaluation of their effects on a number of microorganisms. The hydroxamic acids were obtained from methyl esters of naphthenic acids which have reacted with hydroxylamine hydrochloride and potassium hydroxide in a molar ratio of 1: 3: 6. The syntheses were carried out in methanol solution at a temperature of 110 ° C, under *sealed*-vessel conditions in a microwave reactor. Optimization of the reaction temperature and reaction time was done on the methyl ester of benzoic acid as a model substance, and the progress of reactions were monitored by HPLC analysis. All obtained hidroksamic derivatives of individual and natural naphthenic acids were characterized by NMR and IR spectra. The yields are reported in the range of 61-93% for a reaction time of one minute.

## OHP 14

**Sinteza i antitumorska aktivnost 5-O-metil derivata goniofufurona**

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U ovom radu želimo da saopštimo prvu sintezu dva 5-O-metil derivata (jedinjenje **1** i **2**) prirodnih stiril-laktona tipa goniofufurona, kao i rezultate ispitivanja njihovog uticaja na proliferaciju odabranih humanih neoplastičnih ćelija. Ključni benzilni alkoholi **5** i **6**, koji su lako pristupačni iz D-glukoze, prevedeni su u derivate **1** i **2** sekvencom koja je prikazana na reakcionoj shemi.

**Synthesis and antitumour activity of 5-O-methyl goniofufurone derivatives**

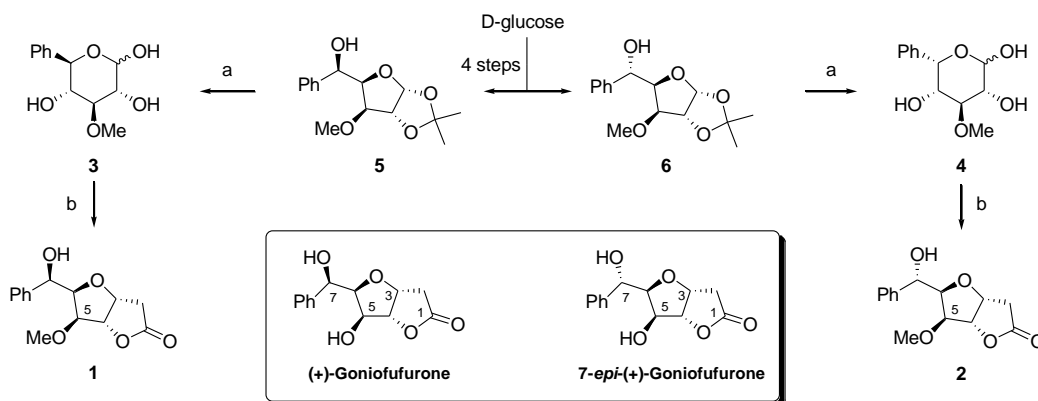
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Herein we want to report the first synthesis of two 5-O-methyl derivatives (compound **1** and **2**) of natural styryl lactones related to goniofufurone, along with their effects on the proliferation of selected human neoplastic cell lines. The key benzylic alcohols **5** and **6**, which were readily available from D-glucose, were converted to derivatives **1** and **2** through sequence outlined in reaction scheme.



Reagents and conditions: (a) 90% aq TFA, rt, 0.5 h, **3** 99%; 0.5 h, **4** 81%; (b) Meldrum's acid, Et<sub>3</sub>N, DMF, 46 °C, 74 h, **1** 41%; 70 h, **2** 46%.

**Acknowledgement:** The work was supported by a grant from the Ministry of Education, Science and Technological Development (Project 172006).

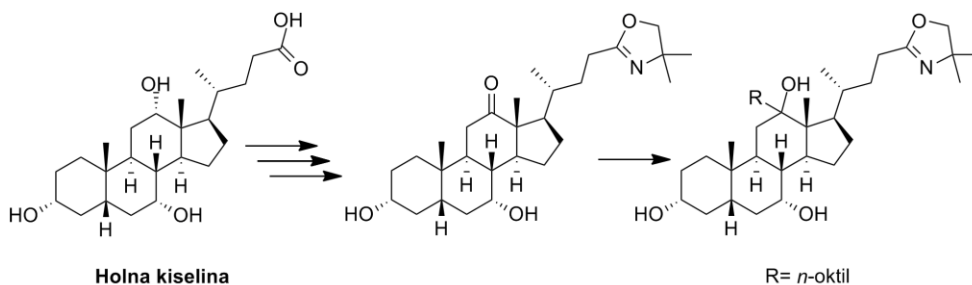
## OH P 15

**Alkilovanje sterno zahtevnog 12-keto oksazolinskog derivata holne kiseline**

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Soli žučnih kiselina mogu da grade micelle koje su sposobne da vežu i prenose hidrofobne lekove. Povećanje hidrofobne površine žučne kiseline može poboljšati sposobnost za vezivanje hidrofobnih molekula. Mi smo modifikovali hidrofobnost žučne kiseline uvođenjem oktil grupe u položaj 12 steroidnog jezgra upotrebom Grignard-ove reakcije uz neophodnu zaštitu karboksilne grupe oksazolinskim prstenom. Nukleofilna adicija odgovarajućeg Grignard-ovog reagensa na C-12 keto grupu se dešava stereoselektivno, a apsolutna konfiguracija na novonastalom hirlnom centru će biti naknadno određena. Adicioni proizvod, pored potencijalnog farmakološkog značaja, je i stereochemijski zanimljiv, s obzirom na to da je 12-keto grupa relativno zaklonjena.<sup>1</sup>

**Alkylation of sterically demanding 12-keto cholic acid oxazoline derivative**

Srđan I. Bjedov, Marija Sakač

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Bile acid salts are amphiphilic molecules able to form micelles that can bind and transport lipophilic drugs. Increase in hydrophobic area of bile acid can improve binding of hydrophobic drugs. Hydrophobicity of molecule has been modified by introduction of octyl alkyl chain in steroid skeleton using Grignard reaction. Carboxylic group has been protected with oxazoline ring. Nucleophilic addition occurs stereoselectively, and absolute configuration will be determined. The synthesis is also stereochemically interesting due to lack of reactivity of keto group in olefination reactions.<sup>1</sup>

1. M. Poša, S. Bjedov, A. Sebenji, M. Sakač, *Steroids* **86** (2014) 16.

**Zahvalnica:** Ovaj rad je finansiran od strane Ministarstva prosvete, nauke i tehnološkog razvoja (Projekat ON172021).

## OH P 16

**Spektralna i kvantnohemijska proučavanja  $N,N'$ -diarilmalonamida**

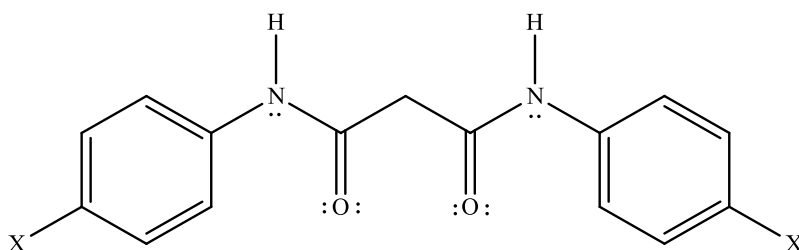
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 Željko J. Vitnik\*\*, Gordana S. Ušćumlić\*, Slobodan S. Petrović\*, Dušan Ž. Mijin\*

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$N,N'$ -Diarilmalonamidi predstavljaju jedinjenja koja se koriste u medicini i farmaciji zbog svoje biološke i hemijske aktivnosti. U cilju detaljnijeg izučavanja strukture ovih jedinjenja, ispitan je uticaj elektronske prirode supstituenata na FT-IR,  $^1\text{H}$  i  $^{13}\text{C}$  NMR spektre, pomoću modela linearne korelacije slobodnih energija, korišćenjem proste i proširene Hametove jednačine i Svejn–Laptonove jednačine. Dodatno, eksperimentalni podaci su korelisani sa DFT teorijskim podacima i dobijene su odlične linearne zavisnosti.



X: OH, MeO, Me, H, Cl, Br, COOH, CH<sub>3</sub>CO, CN, NO<sub>2</sub>

*Slika. Struktura ispitivanih  $N,N'$ -diarilmalonamida*

*Figure. Structure of the investigated  $N,N'$ -diarylmalonamides*

**Spectral and quantum chemical study of  $N,N'$ -diarylmalonamides**

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 Željko J. Vitnik\*\*, Gordana S. Ušćumlić\*, Slobodan S. Petrović\*, Dušan Ž. Mijin\*

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$N,N'$ -Diarylmalonamides are used in medicinal and pharmaceutical chemistry due to their biological and chemical activity. In order to get more insight into the structure of these compounds, the influence of the electronic nature of substituents of  $N,N'$ -diarylmalonamides on FT-IR,  $^1\text{H}$  and  $^{13}\text{C}$  NMR chemical shifts has been investigated by LFER models using the simple and the extended Hammett equations, as well as by the Swain–Lupton equation. Additionally, experimental data have been correlated with DFT results and excellent linear dependence has been obtained.

OH P 17

**Eksperimentalna i kvantnohemijska proučavanja strukture  
3-(4-supstituisanih benzil)-1,3-diazaspiro[4.4]nonan-2,4-diona**

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Gordana S. Ušćumlić

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U radu je sintetizovana serija 3-(4-supstituisanih benzil)-1,3-diazaspiro[4.4]nonan-2,4-diona čija je struktura određena temperaturama topljenja, FT-IR,  $^1\text{H}$  i  $^{13}\text{C}$  NMR i UV/Vis spektroskopijom. Uticaj supstituenata na apsorpcione spektre spirohidantoina, u različitim rastvaračima, proučavan je Hametovom jednačinom. Eksperimentalni rezultati su pokazali zadovoljavajuću saglasnost sa rezultatima kvantnohemijskih proračuna dobijenih primenom DFT metode. Pokazano je da supstituenti značajno utiču na intramolekulski transfer naelektrisanja (ICT) kao i na reaktivnost proučavanih hidantoina razmatranu na osnovu molekuskog elektrostatičkog potencijala (MEP).

**Experimental and quantum chemical studies on the structure of  
3-(4-substituted benzyl)-1,3-diazaspiro[4.4]nonane-2,4-dione**

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In this work, a series of 3(4-substituted benzyl)-1,3-diazaspiro[4.4]nonane-2,4-dione was synthesized and fully characterized by melting points, FT-IR,  $^1\text{H}$ , and  $^{13}\text{C}$  NMR and UV/Vis spectroscopy. The Hammett equation was used to quantitatively evaluate the effects of substituents on the absorption frequencies in different solvents. The experimental results showed very good agreement with quantum chemical calculations obtained using DFT method. It was shown that substituents change the extent of conjugation, and affect intramolecular charge transfer (ICT) character. To estimate chemical reactivity of the molecules, the molecular electrostatic potential (MEP) surface maps were calculated for the optimized geometries of the investigated molecules.

**Acknowledgment.** Authors are grateful to the Ministry of Education, Science and Technological Development of the Republic of Serbia (Project 172013) for the financial support of this work.

**Eksperimentalna i kvantnohemijska UV-Vis apsorpciona analiza  
5-(supstituisanih fenilazo)-3-cijano-1-etil-6-hidroksi-4-metil-2-piridona u  
etanolu i *N,N*-dimetilformamidu**

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Gordana S. Ušćumlić, Dušan Ž. Mijin  
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*\*IHTM, Univerzitet u Beogradu, Studentski trg 12-16, Beograd, Srbija*

UV-Vis apsorpcioni spektri i tautomerija deset piridonskih azo boja detaljno su analizirani u etanolu i *N,N*-dimetilformamidu. Jedinjenja u okviru ove serije boja razlikuju se prema prirodi (OMe, NO<sub>2</sub>, Cl i H) i položaju (*ortho*-, *meta*- i *para*-) supstituenata u fenilnom jezgru. U etanolu ispitivane boje postoje u hidrazonskom obliku, dok se u *N,N*-dimetilformamidu pojavljuje hidrazon-azo anjon tautomerija. UV-Vis apsorpcione energije za jedinjenja dobijene su pomoću vremenski zavisne DFT metode (TD-DFT). Položaj apsorpcionog maksimuma anjonskog oblika zavisi od prirode i položaja supstituenta i može se naći na višim ili nižim talasnim dužinama u odnosu na hidrazonski oblik u *N,N*-dimetilformamidu. Dobijeno je dobro slaganje između eksperimentalnih i teorijskih rezultata.

**Experimental and quantum chemical UV-Vis study of  
5-(substituted phenylazo)-3-cyano-1-ethyl-6-hydroxy-4-methyl-2-pyridones in  
ethanol and *N,N*-dimethylformamide**

Jelena M. Mirković, Bojan Đ. Božić, Vesna D. Vitnik\*, Željko J. Vitnik\*,  
Gordana S. Ušćumlić, Dušan Ž. Mijin  
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In this work, UV-Vis absorption spectra, as well as tautomerism of ten arylazo pyridone dyes have been thoroughly analysed in ethanol and *N,N*-dimethylformamide. The dyes have different substituents (OMe, NO<sub>2</sub>, Cl and H) in different positions (*ortho*-, *meta*- and *para*-) of the phenyl moiety. In ethanol, investigated dyes exist in hydrazone form, while in *N,N*-dimethylformamide there is hydrazone-azo anion tautomerism. UV-Vis absorption energies for these compounds have been calculated using TD-DFT M06-2X model. The position of the absorption maxima of the anion forms in *N,N*-dimethylformamide depend on the nature and position of the substituents and as a result of that, absorption maxima are either at lower or higher wavelengths with respect to hydrazone form. A good agreement between experimental and theoretical data was obtained.

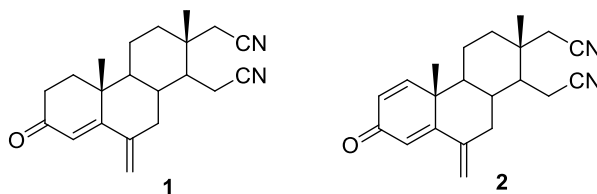
## OHP 19

**Sinteza novih androstanskih 16,17-seko-16,17a-dinitrila**

Andrea R. Nikolić, Jovana J. Ajduković, Marina P. Savić, Aleksandar M. Oklješa, Ivana Z. Kuzminac, Katarina M. Penov-Gaši, Evgenija A. Đurendić, Marija N. Sakač  
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Eksemestan (6-metilenandrosta-1,4-dien-3,17-dion) je najpotentniji oralni steroidni inhibitor enzima aromataze koji je pokazao impresivno farmakološko i kliničko dejstvo u lečenju hormon-zavisnih karcinoma dojke kod žena u postmenopauzi. Takođe, veliku efikasnost u lečenju ove bolesti pokazuju i nesteroidni inhibitori aromataze, anastrozol i letrozol. Imajući u vidu strukturne elemente anastrozola i letrozola (dve nitrilne grupe) i eksemestana, cilj ovog radu bila je sinteza novih steroidnih derivata koji u svojoj strukturi poseduju funkcionalne grupe prisutne u pomenutim lekovima. U skladu sa tim, polazeći od dehidroepiandrosterona, sintetizovani su novi steroidni 16,17-seko-16,17a-dinitrili sa 6-metilen-4-en-3-on (1) i 6-metilen-1,4-dien-3-on sistemom (2), čija će anti-aromatazna aktivnost biti ispitana u kasnijem radu.

*Autori se zahvaljuju Ministarstvu prosvete, nauke i tehnološkog razvoja Republike Srbije (br. projekta 172021).*

**Synthesis of new androstane 16,17-seco-16,17a-dinitriles**

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Exemestane (6-methyleneandrosta-1,4-diene-3,17-dione) is selective, orally active aromatase inhibitor, which has exhibited impressive pharmacologic and clinical properties in treatment of *hormone-dependent breast cancer in postmenopausal women*. *Non-steroidal aromatase inhibitors anastrozole and letrozole (contain two nitrile groups) showed high efficiency in the treatment of breast cancer*. In order to get new compounds which have substructures of the above-mentioned drugs, starting from dehydroepiandrosterone, new androstane 16,17-seco-16,17a-dinitriles with 6-methylene-4-en-3-one (1) and 6-methylene-1,4-dien-3-one system (2) were synthesised.

*Authors would like to thank the Ministry of Education, Science and Technological Development of the Republic of Serbia (Grant No. 172021).*

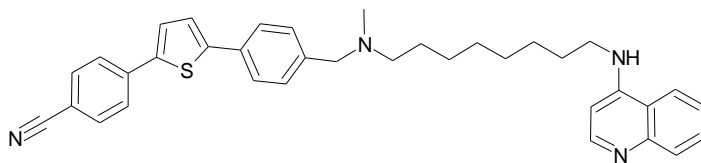
**Nova generacija 4-aminohinolina:  
dizajn, sinteza i antimalarijska aktivnost**

Milica Đ. Videnović, Bogdan A. Šolaja\*

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Malaria je infektivna bolest od koje je samo 2013. godine obolelo 198 miliona ljudi, sa smrtnim ishodom kod oko pola miliona pacijenata. Malariju izaziva parazit iz roda *Plasmodium*, koji na ljude prenosi ubodom ženka *Anopheles* komarca. Usled razvoja rezistencije parazita prema postojećim lekovima, postoji stalna potreba za pronalaženjem novih antimalarika. U nastavku naših istraživanja u oblasti dizajna i sinteze potencijalnih antimalarika sa različitim aromatičnim grupama kao nosačima aminohinolinske farmakofore,<sup>1a,b</sup> sintetisani su novi derivati 4-aminohinolina i ispitana njihova biološka aktivnost prema različitim sojevima *P. falciparuma*. Pokazana je zavisnost antimalarijske aktivnosti ispitanih jedinjenja od dužine alkil-niza i supstituenata na heterocikličnim jezgrima.



*P. falciparum* IC50 (nM)

W2	D6	TM91C235
5	7	13

**New generation of 4-aminoquinolines:  
design, synthesis and antimalarial activity**

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Innovation center of the Faculty of Chemistry, Belgrade

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Malaria is an infectious disease resulting in 198 million estimated cases and over half a million deaths in 2013 alone. It is caused by the *Plasmodium* parasite transmitted among humans by female mosquitoes of the genus *Anopheles*. According to the development of parasite's resistance to standard antimalarial drugs, there is a constant need for identification of new effective therapeutics. Following our previous results in the field of design and synthesis of potent antimalarials with an aromatic group as a carrier of aminoquinoline pharmacophore, we have synthesized new 4-aminoquinoline derivatives and discussed their *in vitro* antimalarial activities against three *P. falciparum* strains. We have shown that antimalarial activity depends on the length of alkyl linker and substitution pattern of heterocyclic cores.

**Acknowledgment:** This research was supported by the Ministry of Education, Science and Technological Development of Serbia (grant no. 172008)

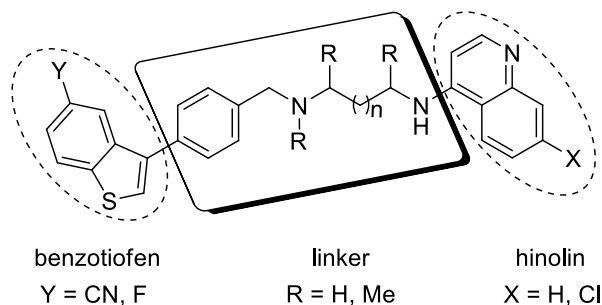
1. a) Opsenica, I. M.; Tot, M.; Gomba, L.; Nuss, J. E.; Sciotti, R. J.; Bavari, S.; Šolaja, B. A.; *J. Med. Chem.* **2013**, *56*, 5860-5871; b) Opsenica, I. M.; Verbić, T. Ž.; Tot, M.; Sciotti, R. J.; Pybus, B. S.; Đurković-Đaković, O.; Slavić, K.; Šolaja, B. A.; *Bioorg. Med. Chem.* **2015**, *23*, 2176-2186



## Primena benzotiofenskih derivata u inhibiciji BoNT/A

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Botulinum neurotoksini (BoNTs) su proteini koje proizvodi bakterija *Clostridium botulinum*. Pripadaju klasi najjačih neurotoksina i izazivaju botulizam, smrtonosnu bolest kod ljudi i životinja. Iz tog razloga, težnja za sintezom malih molekula kao inhibitora je sve veća. U cilju određivanja odnosa između strukture i aktivnosti (SAR) izvršene su modifikacije na benzotiofenskom i hinolinskom delu strukture, zajedno sa promenom dužine linkera. Sintetisani molekuli su se pokazali kao izuzetno dobri inhibitori BoNT/A LC, sa procentom inhibicije do 84%. U cilju ispitivanja mehanizma delovanja, ispitana je aktivnost novih derivata prema holotoksinu BoNT/A, u eksperimentu kojim se određuje procenat razloženog SNAP-25 proteina. Pokazano je da jedinjenja testirana u primarnim neuronima štite SNAP-25 i do 68% (pri niskim  $\mu\text{M}$ ), što je vodilo daljem ispitivanju inhibicije pri različitim koncentracijama.



## Application of benzothiophene derivatives in BoNT/A inhibition

Jelena M. Konstantinović, Katarina B. Bogojević, Mario V. Zlatović, Bogdan A. Šolaja  
Faculty of Chemistry, University of Belgrade, Belgrade

Botulinum neurotoxins (BoNTs) are proteins produced by the bacterium *Clostridium botulinum*. They are among the most potent toxins known and can cause botulism, a serious and life-threatening illness in humans and animals. That is why small-molecule pharmacological intervention is highly desirable. In order to determine structure-activity relationship, structural modifications at the benzothiophene and quinoline scaffolds were performed, combined with different length of the linker. The newly synthesized molecules are potent inhibitors of BoNT/A light chain, with percent of inhibition up to 84%. Encouraged by these findings, in order to explore the mechanism of action, we examined the efficacy of these analogs against full length BoNT/A in SNAP-25 gel cleavage assay. Compounds tested during BoNT/A challenge in primary neurons were found to protect SNAP-25 by up to 68% at low  $\mu\text{M}$  concentrations. This led us to the investigation of dose-dependent inhibition of SNAP-25 cleavage.

**Acknowledgement:** This research was supported by the Ministry of Education, Science and Technology Development of Serbia (grant no. 172008) and US National Institute of Health (1U01AI082051-01).

OH P 22

**Uticaj supstituenata i rastvarača na UV-apsorpcione maksimume  
supstituisanih 1,4-dihidropiridina**

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*Katedra za organsku hemiju, Tehnološko-metalurški fakultet, Karnegijeva 4, 11000 Beograd*

Snimljeni su UV spektri za seriju 1,4-dihidropiridinskih jedinjenja u različitim rastvaračima. Na osnovu dobijenih spektralnih podataka analizirani su uticaji prisutnih supstituenata i rastvarača na položaj apsorpcionog maksimuma karakterističnog za ispitivana jedinjenja. Kako bi se rezultati mogli kvantitativno vrednovati, relevantni apsorpcioni maksimumi su korelisani Hammetovom jednačinom radi ispitivanja efekata supstituentata i Kamlet-Taftovom jednačinom radi ispitivanja efekata rastvarača.

**Substituent and solvent effect on the UV-absorption maxima of  
substituted 1,4-dihydropyridines**

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*Department of Organic Chemistry, Faculty of Technology and Metallurgy, University of  
Belgrade, Karnegijeva 4, 11000 Belgrade*

The UV spectra of a series of substituted 1,4-dihydropyridines were recorded in a set of various solvents. Based on the obtained spectral data the substituent and solvent influence on the characteristic UV-absorption maxima was analyzed. In order to present the results quantitatively the absorption maxima were correlated with the Hammett equation for the investigation of substituent effect, and with the Kamlet-Taft equation for the investigation of solvent effect.

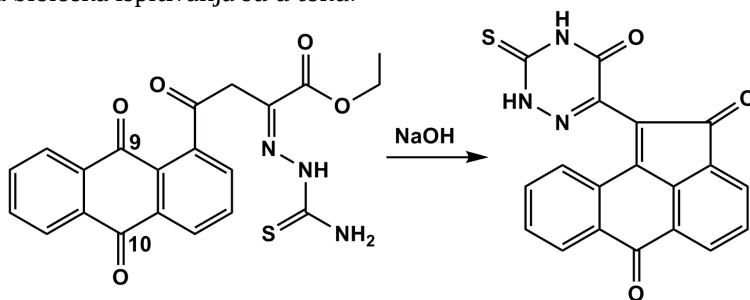
OH P 23

## Sinteza novog derivata antrona sa 6-azatiouracilskim prstenom

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Izvršena je sinteza novog antronskog derivata koji je vezan za 6-azatiouracil. Odgovarajući prekursor, antrahinonski tiosemikarbazon, dobijen je polazeći od antracena.<sup>1</sup> U rastvoru ovo jedinjenje postoji isključivo u keto-iminskoj formi. Iz njega u prisustvu NaOH na sobnoj temperaturi nastaje karbanjon koji vrši intramolekulski nukleofilni napad na C-9 ugljenikov atom karbonilne grupe antrahinonskog dela molekula. Tačna struktura finalnog jedinjenja određena je pomoću elementalne analize kao i 1D i 2D NMR spektroskopije. Dobijeni antronski derivat poseduje potencijalno zanimljive antibakterijske osobine i u vezi s tim odgovarajuća biološka ispitivanja su u toku.



Rad je finansiran sredstvima Ministarstva prosvete, nauke i tehnološkog razvoja Republike Srbije u okviru projekta OI 172016.

## Synthesis of a novel anthrone derivative containing 6-azathiouracyl moiety

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The synthesis of a novel anthrone based compound bearing 6-azathiouracyl ring was performed. The corresponding precursor anthraquinone-thiosemicarbazone was obtained starting from anthracene.<sup>1</sup> In the solution it exists exclusively in keto-imine tautomeric form which in the presence of NaOH aqueous solution at room temperature allows carbanion formation and intramolecular nucleophilic attack on C-9 carbonyl group of the anthraquinone moiety. The exact structure of the final compound was determined using elemental analysis and 1D and 2D NMR spectroscopy. The synthesized anthrone derivative possesses potentially interesting antibacterial properties and biological examinations are in progress.

This work has been funded by the Ministry of Education, Science and Technological Development of the Republic of Serbia (Project No. 172016).

1. V. Marković et al. *Eur. J. Med. Chem.* **64** (2013) 228.

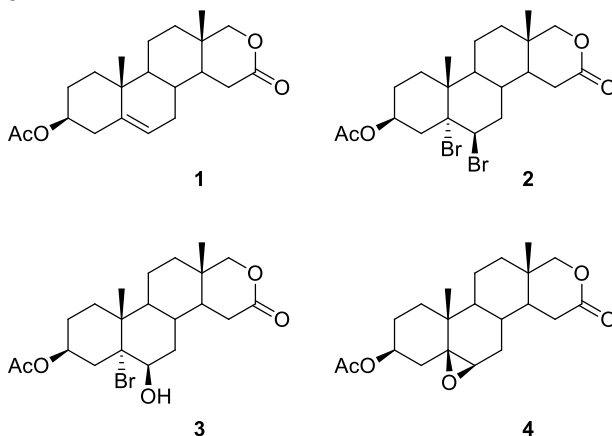
### Strukturalna analiza proizvoda dobijenih u reakciji D-homo laktonskog derivata androst-5-ena sa N-bromacetamidom

Ivana Z. Kuzminac, Dušan Đ. Škorić, Olivera R. Klisurić\*, Andrea R. Nikolić, Srđan I. Bjedov, Marija N. Sakač

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U cilju dobijanja potencijalnih antitumorskih agenasa, izvedena je reakcija 3 $\beta$ -acetoksi-17-oksad-homoandrost-5-en-16-ona (**1**) sa N-bromacetamidom. Dobijena su tri proizvoda: 5 $\alpha$ ,6 $\beta$ -dibromid **2**, 5 $\alpha$ -brom-6 $\beta$ -ol **3** i 5 $\beta$ ,6 $\beta$ -epoksid **4**, čije su strukture određene detaljnom analizom NMR spektara (<sup>1</sup>H, <sup>13</sup>C, 2D HSQC, 2D HMBC, NOE-eksperimenti), kao i rendgenostrukturalnom analizom.



*Autori se zahvaljuju Ministarstvu prosvete, nauke i tehnološkog razvoja Republike Srbije (br. projekta 172021) na finansijskoj podršci.*

### Structural analysis of the products obtained in the reaction of a D-homo lactone androst-5-ene derivative with N-bromoacetamide

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In order to obtain potential antitumor agents, the reaction of 3 $\beta$ -acetoxy-17-oxad-homoandrost-5-en-16-one (**1**) with N-bromoacetamide was carried out and resulted in three products: 5 $\alpha$ ,6 $\beta$ -dibromide **2**, 5 $\alpha$ -bromo-6 $\beta$ -ol **3** and 5 $\beta$ ,6 $\beta$ -epoxide **4**. Molecular and crystal structures were confirmed by detailed NMR (<sup>1</sup>H, <sup>13</sup>C, HSQC 2D, 2D HMBC, NOE-experiments) and X-ray analysis.

*Authors would like to thank the Ministry of Education, Science and Technological Development of the Republic of Serbia (Grant No. 172021) for financial support.*

## Uticaj rastvarača i supstituenata na solvatohromizam 5-ariliden-3-metil-2,4-tiazolidindiona

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Thiazolidin-2,4-dion (TZD) predstavlja interesantnu strukturnu jedinicu u medicinskoj hemiji koja je odgovorna za brojna farmakološka svojstva i biološke aktivnosti. TZD derivati privlače značajnu pažnju istraživača zbog različitih sintetičkih mogućnosti i terapijskog značaja, posebno od uvođenja nekoliko glitazona u kliničku upotrebu za tretman dijabetesa tipa 2 (dijabetes melitus) krajem devedesitih godina. Da bi se proučavao solvatohromno ponašanje 5-ariliden-3-metil-2,4-tiazolidindiona, sintetisano je devet derivata sa različitim supstituentima u arilidenskom jezgru i njihovi UV spektri su snimljeni u rastvaračima različite polarosti u opsegu od 200-600 nm. Uticaj intermolekulskih interakcija sa rastvaračem na pomeranje apsorpcionih maksimuma je analiziran primenom Catalán-ovog modela linearne solvatacione energije (LSER). Takođe, principi korelacija linearne slobodne energije (LFER) korišćenjem SSP i DSP modela su primenjeni na apsorpcione maksimume u cilju proučavanja uticaja supstituenata na solvatohromizam ovih derivata.

## Solvent and substituent effect on solvatochromism of 5-arylidene-3-methyl-2,4-thiazolidinediones

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Thiazolidine-2,4-dione (TZD) represents an interesting scaffold in medicinal chemistry responsible for numerous pharmacological properties and biological activities. TZD derivatives have attracted a significant research interest because of their synthetic diversity and therapeutic relevance, especially since the introduction of several glitazones into clinical use for the treatment of diabetes mellitus in the late 1990s. In order to investigate solvatochromic behaviour of 5-arylidene-3-methyl-2,4-thiazolidinediones, nine derivatives were synthesized with different substituent's at aryl moiety, and their UV absorption spectra have been recorded over the range of 200-600 nm in organic solvents of different polarity. The influence of the intermolecular interactions of these molecules with solvent on the shifts of absorption maxima has been analyzed by means of the linear solvation energy relationship (LSER) model proposed by Catalán. Linear free energy relationship (LFER) has been further applied to absorption maxima. The correlation analysis has been performed using SSP (single substituent parameter) and DSP (dual substituent parameter) model in order to study substituent effect on solvatochromism of these derivatives.

**Funkcionalizovani izokumarini kao antifungalni agensi:  
sinteza i biološka studija**

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Seriya novih 3-supstituisanih izokumarina sintetisana je primenom Pd-katalizovanih reakcija i ispitivano je njihovo *in vitro* antifungalno dejstvo na *C. albicans*. Studija je pokazala da izokumarini supstituisani azolima pokazuju antifungalno dejstvo, pri čemu je aktivnost nekih derivata uporediva sa vorikonazolom. Neki od dobijenih supstituisanih izokumarina su pokazali značajnu aktivnost prema vorikonazol-rezistentnim *Candida* sojevima, *C. krusei* 6258 i kliničkom izolatu *C. parapsilosis* CA-27.

**Functionalised isocoumarines as antifungal compounds:  
Synthesis and biological study**

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A series of novel 3-substituted isocoumarines was prepared via Pd-catalysed coupling processes and screened *in vitro* for antifungal activity against *C.albicans*. The study revealed antifungal potential of isocoumarines possessing the azole substituents, which, in some cases, showed biological properties comparable to those of clinically used voriconazole. Importantly, selected isocoumarines showed significant activity against voriconazole resistant *Candida* species, *C. krusei* 6258 and a clinical isolate of *C. parapsilosis* CA-27.

## **Nastava i istorija hemije / Education in and History of Chemistry**

NIH O 1

### **Istorija hemije u nastavi hemije – stavovi nastavnika hemije**

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Ideja o uključivanju istorije i filozofije nauke u kurikulume prirodnih nauka promovise se decenijama. Primena istorije i filozofije nauke u nastavi/učenju prirodnih nauka okarakterisana je kao dobar način za poboljšanje naučne pismenosti učenika. Neke od prepreka za takav pristup u nastavnoj praksi su: perspektive nastavnika, njihova uverenja, razumevanje glavnih ideja i ciljeva nastave/učenja, kao i epistemološka shvatanja.<sup>1</sup> U istraživanju, u kome je učestvovalo 272 nastavnika hemije osnovnih škola u Srbiji, putem upitnika ispitani su: stavovi nastavnika o sadržajima iz istorije hemije i efektima primene tih sadržaja u nastavi na učeničko razumevanje hemije, načini kako nastavnici prezentuju nauku učenicima, učestalost primenjivanja metoda nastave/učenja i aktivnosti koje podstiču razvoj naučnog rezonovanja kod učenika. Ispitano je i da li su nastavnici tokom inicijalnog obrazovanja učili sadržaje iz istorije i filozofije nauke i da li imaju uzore među naučnicima. Na osnovu dobijenih rezultata može se zaključiti da nastavnici uviđaju potencijal sadržaja iz istorije hemije za bolje razumevanje hemije, ali da takve sadržaje u praksi nedovoljno primenjuju. Rezultati istraživanja ukazuju i na nedostatak kurseva iz istorije i filozofije nauke u toku inicijalnog obrazovanja nastavnika.

*Rad je podržan od strane Ministarstva prosvete, nauke i tehnološkog razvoja Republike Srbije (Projekat br. 179048).*

### **History of chemistry in chemistry education – chemistry teachers attitudes**

Vesna D. Milanović, Dragica D. Trivić

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The idea of involving the history and philosophy of science in the science curricula has been promoting for decades. The involving of the history and philosophy of science in the science classes is characterized as a good way to improve scientific literacy of students. Some of obstacles for this approach in teaching practice are: the perspectives of teachers, their beliefs, understanding of the main ideas and goals of teaching/learning and epistemological understanding.<sup>1</sup> In the research with 272 chemistry teachers from primary schools in Serbia, questionnaire was used to examine: the attitudes of teachers about the contents of the history of chemistry and about the effects of application these contents in teaching practice on student's understanding of chemistry, the ways in which teachers present science to students, the frequency of application teaching/learning methods and activities that encourage the development of scientific reasoning of students. Also, we examined whether the teachers learned the contents of the history of chemistry and philosophy of science during their initial education and their personal preferences about scientists. Based on the results it can be concluded that teachers recognize the potential of the history of chemistry content for better student's understanding of chemistry. However, they don't apply these contents in practice enough. The results indicate the lack of courses in the history and philosophy of science during initial education of chemistry teachers.

*This work was supported by the Ministry of Education, Science and Tehnological Development of the Republic of Serbia (Project No. 179048).*

NIH O 2

## **Unapređivanje naučne pismenosti učenika kroz interdisciplinarni pristup u nastavi prirodnih nauka**

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Naučna pismenost obuhvata osposobljenost učenika da znanja iz različitih naučnih disciplina povežu i primene prilikom rešavanja problema iz realnog života. Da bismo utvrdili da li primena interdisciplinarnog pristupa koji potencira povezanost gradiva različitih nastavnih predmeta može da unapredi naučnu pismenost, sprovedli smo pedagoški eksperiment s paralelnim grupama. Eksperiment je izveden u okviru obrade nastavne teme *Varenje*. Učestvovalo je 97 učenika četvrtog razreda gimnazije prirodno-matematičkog smera (51 učenik u eksperimentalnoj i 46 učenika u kontrolnoj grupi). Ustanovili smo da su učenici iz eksperimentalne grupe na završnom testiranju postigli statistički značajno bolji ukupni procenat tačnih odgovora ( $t=2,3$ ; razlika je statistički značajna na nivou  $p<0,05$ ), što ukazuje da interdisciplinarni pristup nastavi ima potencijal da doprinese razvoju naučne pismenosti učenika.

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## **Improvement of students' scientific literacy through interdisciplinary approach to science teaching**

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Scientific literacy encompasses students' ability to connect and apply knowledge from various scientific disciplines in solving real-life problems. In order to determine the effects of interdisciplinary approach that emphasises connections between content knowledge of various school subjects to the improvement of scientific literacy, we conducted the pedagogical experiment with parallel groups. The experiment was conducted within the framework of dealing with the teaching topic *Digestion* and it encompassed 97 fourth-year grammar school students (51 students in the experimental and 46 in the control group). We found statistically significant difference in the overall percentage of correct answers on the post-test in favour of the students from the experimental group ( $t=2.3$ ; the difference is statistically significant at the level of  $p<0.05$ ), which implies that the interdisciplinary teaching approach has the potential to improve scientific literacy of our students.

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NIH P 1

### **Dimenzionisanje kognitivne kompleksnosti stehiometrijskih zadataka**

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Kognitivna kompleksnost je jedna od najvažnijih karakteristika nastavne aktivnosti koja je kritična za procenu merljivih komponenata kognitivnog opterećenja - mentalnog napora i performansi. Cilj ovog rada je formiranje Rubrike za dimenzionisanje kognitivne kompleksnosti u stehiometrijskim zadacima. Kombinovanjem ove Rubrike sa Rubrikom za procenu kognitivne kompleksnosti<sup>1</sup> dobijen je validan postupak kojim se može proceniti kognitivna kompleksnost stehiometrijskih zadataka. Osnova na kojoj se ovaj instrument zasniva je sabiranje težina koncepata ili veština potrebnih za rešavanje zadatka, uz razmatranje međusobne interaktivnosti radi dobijanja numeričke vrednosti rejtinga kognitivne kompleksnosti radi dalje statističke obrade. Validnost Rubrike potvrđena je Testom stehiometrijskih zadataka na uzorku od 82 učenika sa osnovnim statističkim parametrima i međusobnom linearnom zavisnošću.

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### **Dimensioning of complexity of stoichiometric tasks**

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Cognitive complexity is one of the most important characteristics of teaching activities which is crucial for the assessment of measurable components of cognitive load - performances and mental effort. The aim of this study was the creation of Rubric for dimensioning of complexity of stoichiometric tasks. Combining created Rubric with Cognitive complexity rating rubric<sup>1</sup> a valid instrument for dimensioning cognitive complexity of stoichiometric tasks has been provided. This Rubric is based on summation of concepts' difficulty or skills needed to solve the task by considering their mutual interactions in order to obtain numerical values of cognitive complexity rating for further statistical analysis. The validity of this Rubrics was confirmed with Test of stoichiometric tasks on a sample of 82 students by basic statistical parameters and with mutual linear dependence.

*The results presented are part of the research conducted within the project Grant Number 179010 of the Ministry of Education, Science and Technological Development of the Republic of Serbia.*

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NIH P 2

## **Razvoj veština sistemskog mišljenja primenom sistemskih zadataka u domenu organske hemije**

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Cilj ovog rada bio je ispitivanje razlika u veštinama sistemskog mišljenja kod učenika obučavanih primenom sistemskog pristupa (E grupa) i kod učenika obučavanih tradicionalnom nastavom (K grupa). Uzorak ispitanika činilo je 65 učenika trećeg razreda gimnazije „Jovan Jovanović Zmaj” iz Novog Sada. Veštine sistemskog mišljenja kod učenika E grupe (34 učenika) i K grupe (31 učenik) u domenu organske hemije ispitane su primenom sistemskih zadataka. Da bi se pomenute veštine dovele u vezu sa postignućem učenika u sistemskim zadacima, praćena je shema vrednovanja ueničkih odgovora kroz nekoliko nivoa: identifikovanje pojedinačnih koncepata (1 bod), parcijalno povezivanje koncepata (2 boda), višestruko povezivanje (3 boda) i kompleksno povezivanje (4 boda). Nakon izvršenog bodovanja, statistički značajne razlike među grupama pojavile su se u nivoima višestrukog i kompleksnog povezivanja, u korist E grupe kod oba pola ispitanika. Međutim, dok razlike među ispitanicima različitog pola u okviru K grupe nisu bile značajne, učenice iz E grupe su bile uspešnije od učenika razmatrajući najviši nivo sistemskog mišljenja.

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## **The development of systems thinking skills ussing systemic questions in organic chemistry domain**

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The aim of this study was to investigate differences in students' systems thinking skills when they were taught ussing systemic approach (E group) and traditional method (C group). The study included 65 third-grade students from high school „Jovan Jovanović Zmaj” in Novi Sad. E group (34 students) and C group (31 students) students' systems thinking skills in organic chemistry domain were evaluated by applying systemic questions. In order to correlate mentioned skills with students' achievement in systemic questions, specific assessment schema was developed and followed throught several levels: identifying individual concepts (1 point), partial linking between concepts (2 points), multiple linking (3 points) and complex linking (4 points). After scoring of students' performance, statistically significant differences between groups were observed in multiple and complex linking levels, in favor of E group by each gender. Additionally, while differences between males and females in C group were not significant, females in the E group were better at the highest level of systems thinking than males from the same group.

*Acknowledgement: This work has been supported by Grant No. 179010 from the Ministry of Education, Science and Technological Development of the Republic of Serbia.*



NIH P 3

### **Imamo li dovoljno hemijskog znanja o kozmetičkim proizvodima?**

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Svako od nas, čime god da se bavi, svakodnevno se susreće s proizvodima hemijske industrije. Za pravi izbor neophodna je osnovna građanska hemijska pismenost, koja se stiče ne samo u školi, već i iz iskustva, iz medija ili knjiga. Cilj ovog rada bio je utvrditi nivo hemijske pismenosti odraslog stanovništva Vojvodine u oblasti hemijskih sadržaja vezanih za kozmetičke proizvode. Istraživanje je sprovedeno na 103 ispitanika sa teritorije Vojvodine, oba pola, starosti 19-63 godine, srednje stručne spreme različitih obrazovnih profila. Dobijeni rezultati ukazuju da odraslo stanovništvo ne poseduje dovoljno hemijskog znanja o kozmetičkim proizvodima koje svakodnevno koristi. Uprkos tvrdnji da najviše znanja imaju na osnovu sadržaja izučavanih u školi, na njihove stavove i mišljenja značajno utiču i TV reklame koje svakodnevno gledaju, a koje su često izvor dezinformacija i miskoncepcija. Postoji razlika između muškaraca i žena, što se može objasniti većim učešćem žena u odabiru kozmetičkih preparata i proizvoda kućne hemije. Obrazovni profil srednjoškolskog obrazovanja ne utiče na informisanost i znanje ispitanika.

### **Have we got sufficient chemical knowledge on cosmetic products?**

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All of us, no matter what our profession is, come into contact with chemical products on a daily basis. For making right choices it is important to possess the basic civic chemical literacy, which is gained not only through schooling, but also through experience, books, media... The aim of this research was to establish the level of chemical literacy of the adult population of Vojvodina in the field of cosmetic products. Research was conducted on the sample of 103 persons of both genders, aged 19-63, with secondary education of various profiles. Obtained results indicate insufficient chemical knowledge of cosmetic products in everyday use among the adult population. In spite of statement that the main source of their knowledge is school, their attitudes and opinions are greatly influenced by TV commercials which they watch every day, and which are often the source of disinformation and misconceptions. It was established that there is a difference between male and female population, which can be explained with the fact that women are more involved in choosing and applying cosmetic preparations. Educational profile does not significantly affect the knowledge of the participants.

NIH P 4

**Miskonceptije studenata prve godine smera Diplomirani hemičar  
u oblasti opšte hemije**

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Ova studija je fokusirana na identifikaciju i opisivanje miskonceptija kod studenata prve godine Prirodno-matematičkog fakulteta u Novom Sadu, smera Diplomirani hemičar. Za utvrđivanje miskonceptija primenjena je metoda računanja frekvencija biranja distraktora i tačnih odgovora u zadacima<sup>1</sup>. Istraživanjem je obuhvaćeno 60 studenata, a testiranje je sprovedeno na samom početku prvog semestra školske 2014/15. godine. Kao instrument za identifikaciju miskonceptija korišćen je test, koji je konstruisan u formi dvoslojnog dijagnostičkog testa. Svaki zadatak čine dva sloja, tako da prvi sloj čini pitanje koje se odnosi na problem zadatka, dok drugi nivo čini set objašnjenja za odgovor dat u prvom sloju. Odabrana objašnjenja u drugom sloju pružaju informaciju o postojanju miskonceptija. Rezultati testa dali su uvid u postojanje ukupno 8 miskonceptija kod ispitivane grupe studenata.

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**Misconceptions of the first year students majoring in chemistry  
in the field of general chemistry**

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This study is focused on the identification and description of misconceptions among the first year students of the Faculty of Sciences, Novi Sad majoring in chemistry. For determination of misconceptions, a frequency of choosing distractors and correct options has been applied<sup>1</sup>. The study included 60 students, and the testing was conducted at the very beginning of the first semester of the 2014-15 school year. A test was used as a tool for identifying misconceptions and it was constructed in a form of a two-tier diagnostic test. Each task consist of two tiers, so that the first tier relates to a problem statement, while a second tier offers a set of explanations for the answers from the first tier. The second tier provides information about the existence of misconceptions. Test results gave an insight into the existence of a total of 8 misconceptions among tested students.

*Acknowledgement.* This work has been supported by Grant No. 179010 from the Ministry of Education, Science and Technological Development of the Republic of Serbia.

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**Lav Aleksandrovič Čugajev prvi moderni ruski naučnik organske hemije**

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U ovom radu je prikazan plodonosan život i rad ruskog hemičara i bakteriologa Čugajeva. Svojim delima je utemeljio modernu organsku hemiju. Njegov naučni rad je bio vezan za organsku hemiju i hemiju kompleksnih jedinjenja. Najpoznatija ostvarenja su mu: Čugajeva so, Čugajeva reakcija, Teorija koordinacione strukture kompleksnih jedinjenja, Kvantitativna metoda za određivanje mobilnih atoma vodonika u organskim jedinjenjima, Primena organskih reagenasa u analitičkoj hemiji, Primena reagensa za kvalitativno i kvantitativno određivanje nikla. Čugajev je sarađivao sa poznatim hemičarima: Zelinskim, Gabričevskim, Cerevitinovim i Nobelovcem Vernerom. Njegova izreka: Sve teorije su u redu, dok se ne pojave nove, a onda stare lete naglavačke, aktuelna je i danas.

**Lev Aleksandrovich Chugaev first modern Russian scientist  
in organic chemistry**

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In this paper, the life and work of Russian chemist and bacteriologist Chugai is shown. His works has provided the basis modern organic chemistry. His scientific work was cohere to organic chemistry and chemistry of complex compounds. Chugai best-known achievements are: Chugai salt, Chugai reaction, Theory of coordination structure of complex compounds, Quantitative method for the determination of mobile hydrogen atoms in organic compounds, Application of organic reagents in analytical chemistry, Application of reagents for the qualitative and quantitative determination of nickel. Chugai cooperated with renowned chemists: Zelinsky, Gabrichevsky, Tserevitinov and Nobel laureate Werner. His saying: All theories are fine, until the appearance of new ones, and then the olds fly upside down, is still heard today.

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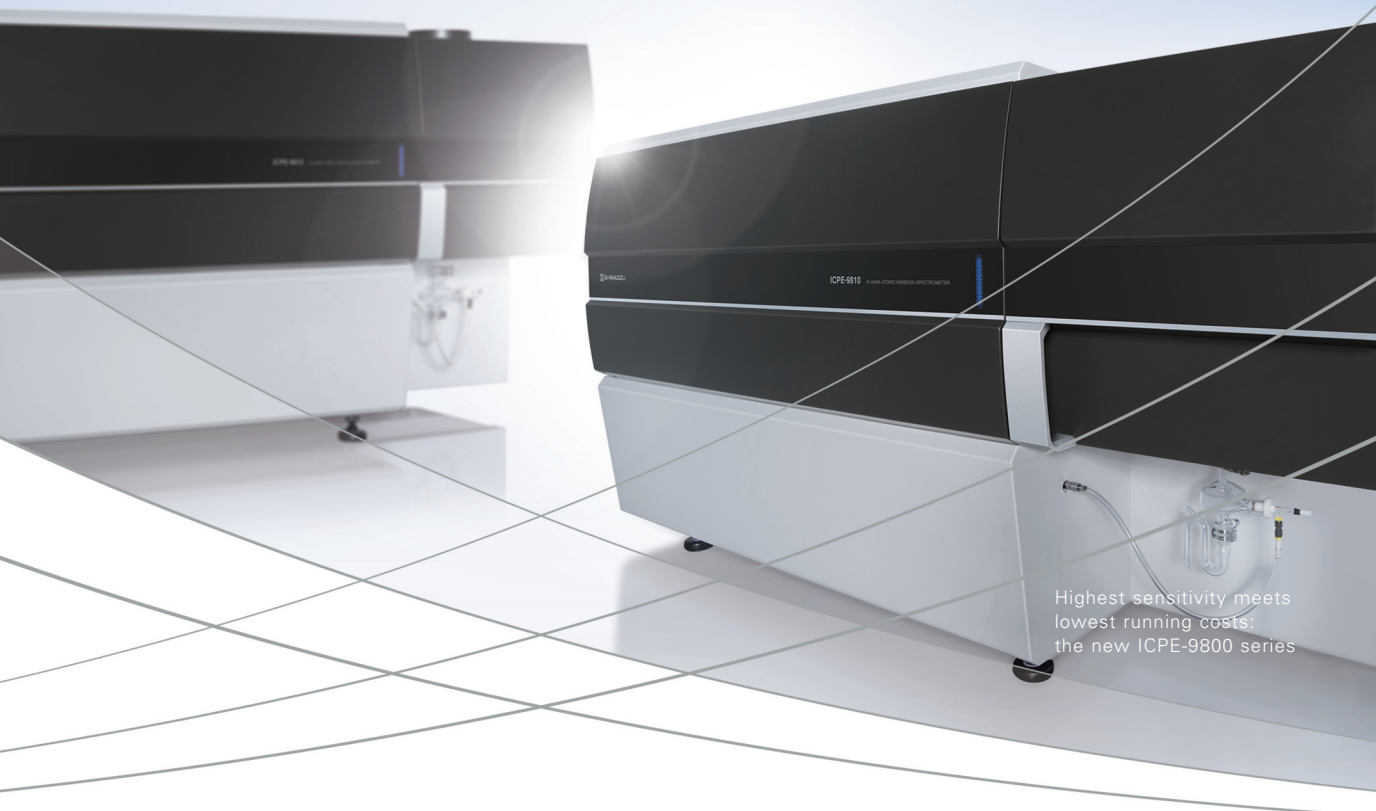
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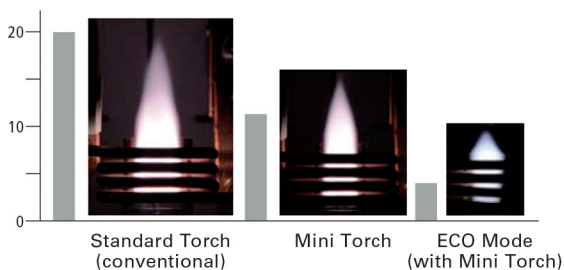


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Za manje od jedne decenije svog postojanja, **SUPERLAB** je uspeo da zauzme lidersku poziciju na našem tržištu. Danas, **SUPERLAB** reprezentuje i prodaje proizvode više od 100 najrenomiranijih svetskih kompanija iz oblasti laboratorijuma, i to sa eskuzivnim pravom od 22 ino-kompanije.

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Da bi se maksimalno približili korisničkim zahtevima i želji da na njih odgovorimo, organizovali smo rad **SUPERLAB** po najnovijim principima savremenog poslovanja.

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Poštujući tradiciju i rukovodeći se aktuelnim trendovima i zahtevima tržišta, putokaz za budućnost biće nam zahtevi i potrebe naših poslovnih partnera!

## MISIJA

**SUPERLAB** treba da obezbedi najsavremeniju laboratorijsku opremu, pribor i potrošni materijal svim zainteresovanim klijentima / kupcima po pristupačnim cenama, poštujući visoke standarde u pogledu kvaliteta proizvoda i usluga.

