



*J. Serb. Chem. Soc.* 77 (1) 83–94 (2012)  
JSCS–4251

## Influence of the sodium dodecyl sulphate (SDS) concentration on the disperse and rheological characteristics of oil-in-water emulsions stabilized by octenyl succinic anhydride modified starch–SDS mixtures

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(Received 30 June 2010, revised 7 February 2011)

**Abstract:** Stability of oil-in-water emulsions can be achieved by chemically modified starch, such as octenyl succinic anhydride (OSA) starch, as an emulsifier. In order to analyse the disperse and rheological characteristics of emulsions containing two kinds of emulsifiers, part of the OSA starch can be substituted with an adequate concentration of sodium dodecyl sulphate (SDS), which is a small surfactant with the same charge as OSA starch. The oil contents of the examined emulsions were 5, 20 and 50 %. The selected OSA starch concentration was 10 % and replacements of a part of the OSA starch were realized with SDS concentrations of 1, 3 and 5 %. Dispersed droplets of emulsions were defined by determination of the Sauter mean diameter  $d_{32}$  and particle size distribution. Flow curves were used to describe the rheological properties of the emulsions. In addition, the stability of the emulsion samples was observed and expressed by the creaming index. The obtained results indicated a decrease in the Sauter mean diameter of the droplets, the standard deviation and the apparent viscosity of the emulsions with increasing amounts of SDS within the emulsifier mixture OSA starch–SDS. According to creaming rate, the emulsions with OSA starch were more stable than the emulsions stabilized by the OSA starch and SDS combinations.

**Keywords:** oil/water emulsions; octenyl succinic anhydride starch; sodium dodecyl sulphate, rheology; disperse characteristics.

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doi: 10.2298/JSC100630150K

## INTRODUCTION

An emulsion is a dispersed system that contains at least two immiscible liquids and a third component that ensures system stability, known as an emulsifier. An emulsifier adsorbs at the oil–water interface, where it decreases the surface tension and coats the dispersed droplets, thereby protecting them from coalescence.

The classification of emulsifiers includes two groups of widely applied compounds for emulsion stability: small-molecule surfactants and macromolecular emulsifiers, known as biopolymers. Sodium dodecyl sulphate (SDS) belongs to the first group and octenyl succinic anhydride (OSA) starch to the second.

Modified starches are native starches that have been chemically or physically changed in order to improve their properties and to satisfy specific demands of industrial application.<sup>1</sup> Chemically modified starches are, generally, obtained by introducing a chemical group or molecule part that reacts with hydroxyl groups of the starch molecules. Accordingly, the properties of native starch are more or less changed.<sup>2</sup>

OSA starch is formed by the modification of native starch by octenyl succinic anhydrides. One of the three accessible carbon atoms of the glucose molecule, at position 2, 3 and 6, are substituted by octenyl succinic anhydride. The substitution largely occurs at branched amylopectin chains, and the degree of substitution (*DS* value) for food application is in the range 0.01–0.03.<sup>3,4</sup> The hydrophobic OSA group incorporated into the hydrophilic structure of native starch provides surface active properties to the macromolecule. In addition, the hydrophobic OSA substituent contains a carboxylic group that can be negatively charged, thus considering the low degree of substitution, this modified starch has properties of a weakly charged polyelectrolyte.<sup>5</sup>

In this manner, OSA starch has dual properties; the amphiphilic nature of the molecules provides stronger surface activity compared to native starch molecules,<sup>6</sup> and the macromolecule characteristic increases the viscosity of the continuous phase. These characteristics make it suitable for stabilisation of dispersed systems, especially oil-in-water emulsions. The macromolecules are brought to the oil–water interface by short, hydrophobic, octenyl succinic chains that are in the oil phase, while long amylopectin chains stay at the water phase and protect the droplets against flocculation by the steric stabilization mechanism.<sup>7,8</sup>

Synthetic surfactants, such as SDS, are widely used components for different industrial formulations. The specific molecular structure of SDS, represented by long aliphatic chains with a sulphate ester group, confers amphiphilic properties and significant surface activity to the molecule.<sup>9</sup> The dispersed droplets of an emulsion are stabilized by electrostatic repulsion, unlike OSA starch that accomplishes stabilization by the steric mechanism.<sup>10</sup> The adsorption of negatively charged SDS molecules to the surface of the oil droplets increases the electro-

static repulsion between droplets.<sup>11</sup> Dickinson and Ritzoulis<sup>12</sup> examined creaming and rheology of an oil-in-water emulsion containing SDS and sodium caseinate. They concluded that an excess of SDS promoted destabilization through fast creaming and explained that non-adsorbed surfactant micelles in the aqueous phase of the emulsions caused depletion flocculation.

The aim of this work was to examine the properties of emulsions stabilized by OSA starch and mixtures of OSA starch and SDS. SDS is chosen because of its same electric charge as OSA starch, but different chemical structure and different stabilizing mechanism. Knowledge about properties of the final emulsion product, such as droplet size, stability and rheology, when those two surfactants are used together, is important in order to improve quality and the range of their application. Due to that part of the OSA starch, which are used as emulsifier, will be substituted with adequate concentrations of SDS and the characteristics of emulsions will be analyzed.

## EXPERIMENTAL

### *Material*

Sunflower oil "Olivko", acquired from the local oil production plant "Banat" Nova Crnja, Serbia, has a high content of oleic acid as a monounsaturated acid and is highly resistant to oil hydrolysis and oxidation. Its physico-chemical characteristics at 25 °C were: density,  $\rho = 0.9145 \text{ g cm}^{-3}$ , viscosity coefficient,  $\eta = 56 \text{ mPa s}$ , and total acid number,  $TAN = 0.353 \text{ mg KOH g}^{-1}$  oil. The fatty acid composition of the "Olivko" oil presented in Table I was determined by gas chromatography-mass spectrometry analysis.<sup>13</sup>

As an emulsion stabilizer, OSA starch with the trade name Purity Gum 2000, which is a waxy maize derivative produced by the National Starch and Chemicals GmbH, Germany, was used. It is recommended as good natural emulsifier for food and pharmaceutical applications. The SDS was obtained from Centrohem, Beograd, Serbia. Double-distilled water was used. All chemicals were of reagent grade and were used as such.

### *Preparation of the emulsions*

The continuous phase was formed by dissolving an appropriate amount of OSA starch or OSA starch-SDS mixtures in water and heating at 50 °C. The selected OSA starch concentration was 10 %. Part of the OSA starch was replaced with adequate SDS concentrations (1, 3 and 5 %). Hence, emulsions stabilized with: 10 % OSA starch; 9 % OSA starch and 1 % SDS; 7 % OSA starch and 3 % SDS; and 5 % OSA starch and 5 % SDS. The chosen amount of oil, the dispersed phase, based on the mass of emulsion, was added to the continuous phase in order to form an emulsion. The dispersed phase concentrations were 5, 20 and 50 %. In order to avoid microbial contamination, 0.01% of sodium azide was added. Homogenization was performed in an Ultra-Turrax T25 basic homogenizer, equipped with an S 25 N-18 G dispersing tool, at a constant temperature of  $25 \pm 0.1 \text{ }^\circ\text{C}$  at  $9500 \text{ min}^{-1}$ . The energy density for the preparation of the emulsions<sup>14</sup> was  $6.0 \times 10^6 \text{ W m}^{-3}$ . The total homogenization time was 20 min. Prior to the measurements, the prepared emulsions were kept at room temperature for 24 h.

### *Determination of the particle size and particle size distribution*

The size distribution of the droplets was determined using microphotography. The emulsion samples required pre-arrangements before taking the microphotographs.<sup>15</sup>

The microphotographs were taken at few optical fields using an optical microscope (TP-1001C TOPICA CCD CAMERA (Kruss)). They were adjusted by adequate software and expanded 1:1000; hence, a droplet with an actual size of 1  $\mu\text{m}$  corresponded to 1 mm on the photograph. Due to the diameter frequency, the Sauter mean diameter  $d_{32}$ ,  $\mu\text{m}$ , (Eq. (1)) was determined in order to define the droplet size distribution.

$$d_{32} = \frac{\sum n_i d_i^3}{\sum n_i d_i^2} \quad (1)$$

The standard deviation was calculated according to Eq. (2):

$$\sigma^2 = \frac{\sum n_i (d_i - d_n)^2}{\sum n_i} \quad (2)$$

where  $n_i$  is number of droplets in each size class,  $d_i$  is the droplet diameter,  $\sum n_i$  is total number of droplets, and  $d_n$  is the average droplet diameter:

$$d_n = \frac{\sum n_i d_i}{\sum n_i} \quad (3)$$

#### *Rheological measurement*

The determination of rheological behaviour was performed using a HAAKE Rheostress RS600 rotational viscometer ("Thermo Electron Corporation", Karlsruhe, Germany) with a cone-plate C60/1Ti sensor (the cone and plate gap was 0.052 mm). The measurements were realized at a constant temperature of  $25 \pm 0.1$  °C. The rheological method included hysteresis loop tests. The samples were exposed for the first 180 s to an increasing shear rate from 0 to  $500 \text{ s}^{-1}$ , the shear rate was held constant at  $500 \text{ s}^{-1}$  for 120 s and finally, the shear rate was decreased to  $0 \text{ s}^{-1}$  in 180 s.

#### *Creaming rate determination*

Creaming of the emulsion samples was observed at room temperature. The volume of the transparent serum layer formed at the bottom of the cylinder was registered visually and expressed by  $H_S$ . The total volume of the emulsion sample was  $H_E$ . The extent of creaming was characterized by the creaming index  $H / \%$  (Eq. (4)):

$$H = 100 \frac{H_S}{H_E} \quad (4)$$

#### *Statistical analysis*

Statistical analysis of the data and significant differences at the significant level 0.05 for several variables, based on three individual measurements, were determined by the ANOVA procedure and Duncan multiple range tests. The calculations were performed using the statistical software SPSS 15.0 (SPSS Inc, Chicago, Illinois, USA).

## RESULTS AND DISCUSSION

### *Disperse characteristics*

A comparison of the Sauter mean diameter  $d_{32}$  of the emulsions stabilized with OSA starch and suitable concentrations of SDS is given in Fig. 1A.

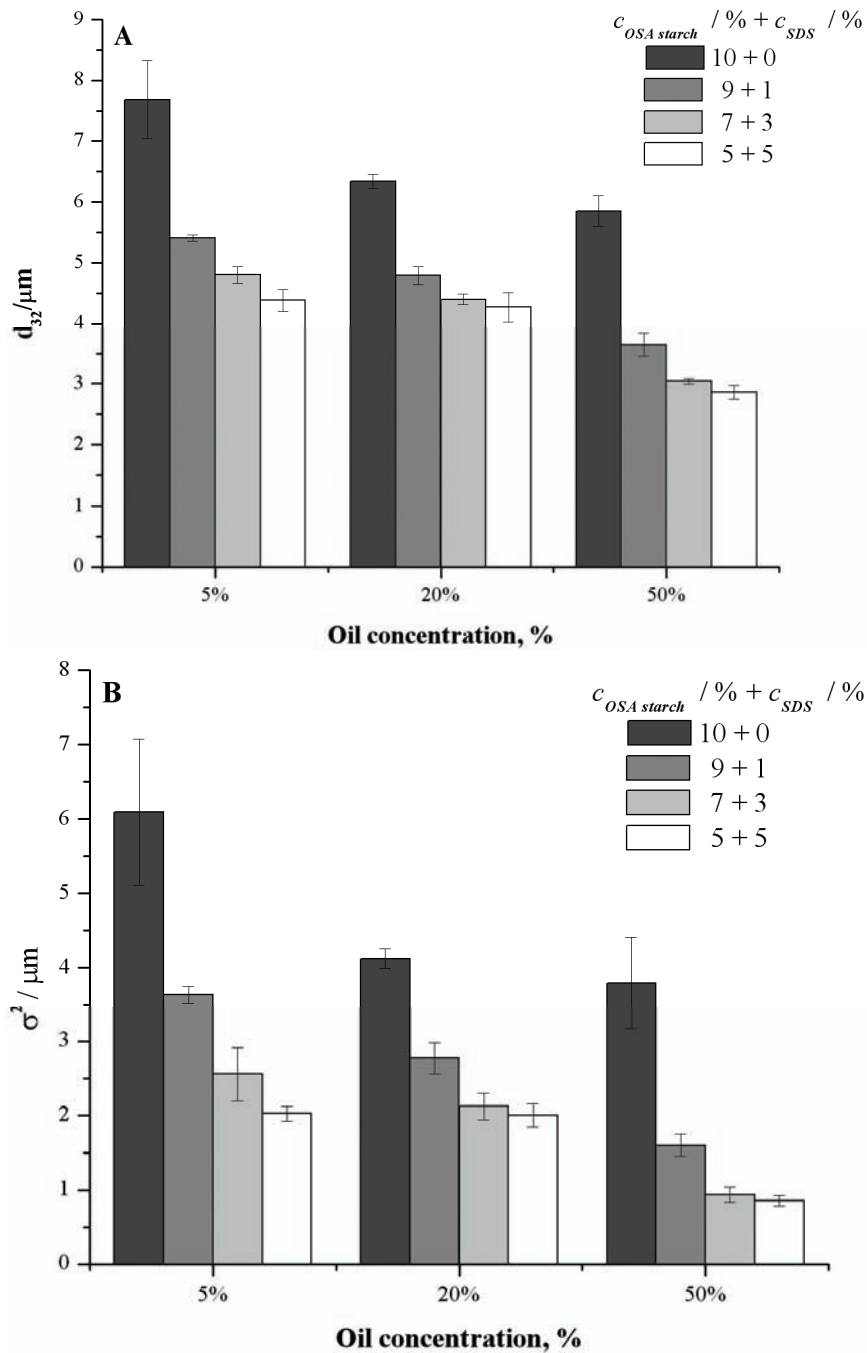


Fig. 1. The influences of OSA starch–SDS ratio on A) the Sauter mean diameter  $d_{32}$  of the dispersed droplets and B) the standard deviations of the investigated emulsions.

Increasing the amount of SDS in the OSA starch–SDS mixtures led to a decrease in the average droplet diameter of the examined emulsions. The same effect occurred for all the examined oil concentrations.

SDS molecules are able to adsorb at the oil–water interface more rapidly than biopolymers, such as OSA starch.<sup>18,22</sup> The slow adsorption of OSA starch is caused by the crowding of its hydrophobic and hydrophilic groups that slows down the diffusion process and delays adsorption.<sup>16–18,22</sup> Several biopolymer molecules can be adsorbed at a newly created interface at the early stage of the emulsification process, but presence of adsorbed small surfactant molecules cause biopolymer substitution from interface.<sup>17,19,20</sup>

The standard deviation is a value of the width of the size distribution of the droplets in a polydisperse emulsion.<sup>21</sup> The changes in the standard deviation (Fig. 1B) were similar to those for  $d_{32}$ . It is obvious that emulsions without SDS had the largest standard deviation values, irrespective of the oil content, which means that they were highly polydisperse.

Jafari *et al.*<sup>18</sup> announced that addition of Tween 20 to oil-in-water emulsions stabilized with OSA starch caused a considerable reduction of the average droplet diameter. This occurrence is a repercussion of the ability of the small Tween 20 molecules to adsorb at the interface before the OSA starch and to disable coalescence during the emulsification process. SDS is also a small molecule like Tween 20 and they have similar mobility and adsorption properties in comparison to OSA starch; hence, the premise by Jafari *et al.* could also be valid for the composite emulsifiers analyzed in this work. The results presented in Fig 1A were confirmed by Tesch *et al.*,<sup>22</sup> who reported that the average droplets diameters of emulsions stabilized by OSA starch were higher than those for emulsions stabilized by SDS. This was caused by the faster adsorption of small SDS molecules at the oil–water interface.

The observation is also in accordance to the results presented by Tangsu-phoom *et al.*<sup>23</sup> They added SDS to coconut milk before the homogenization step and noticed a reduction in the average diameter of the droplets.

#### *Rheological characteristics*

To determine flow parameters of the emulsions, a power law equation (Eq. (5)) was suitable because the systems did not show yield stress:

$$\tau = K \dot{\gamma}^n \quad (5)$$

where  $\tau$  is the shear stress,  $\dot{\gamma}$  is the shear rate,  $K$  is a consistency index and  $n$  is the flow behaviour index.

The consistency index  $K$  is a measure of the system consistency and it is related to the apparent viscosity. The flow behaviour index  $n$  determines the degree of non-Newtonian behaviour and varies in the range between 0 and 1. The

non-Newtonian character of an investigated system is more pronounced for smaller values of constant  $n$ .

The values of  $K$  and  $n$  for the same concentration of oil phase, which are located in the same row, are presented in Table I. The values are presented at the confidence interval 95 % and the values of the individual measurements are given as mean value  $\pm$  error of determination.

TABLE I. Flow curves parameters  $K$  and  $n$  of the emulsions stabilized with OSA starch and OSA starch–SDS mixtures; the values are presented at the confidence level 95 % and the values of the individual measurement are given as the mean value  $\pm$  the error of determination; the mean values do not differ significantly ( $p > 0.05$ ) if they are followed by the same letter in the superscript

Oil content, %	( $c_{\text{OSA starch}} + c_{\text{SDS}}$ ), %	$K / \text{Pa s}$	$n$
5	10 + 0	0.0088990 $\pm$ 0.00016 <sup>(cd)</sup>	0.9463667 $\pm$ 0.00588 <sup>(d)</sup>
	9 + 1	0.0088623 $\pm$ 0.00041 <sup>(cd)</sup>	0.9772000 $\pm$ 0.00578 <sup>(c)</sup>
	7 + 3	0.0070097 $\pm$ 0.00032 <sup>(b)</sup>	0.9533667 $\pm$ 0.00745 <sup>(b)</sup>
	5 + 5	0.0042753 $\pm$ 0.00005 <sup>(a)</sup>	0.9634333 $\pm$ 0.00225 <sup>(a)</sup>
20	10 + 0	0.0216700 $\pm$ 0.00342 <sup>(cd)</sup>	0.9314000 $\pm$ 0.00375 <sup>(cd)</sup>
	9 + 1	0.0209600 $\pm$ 0.00038 <sup>(cd)</sup>	0.9300667 $\pm$ 0.00428 <sup>(cd)</sup>
	7 + 3	0.0190533 $\pm$ 0.00076 <sup>(b)</sup>	0.8834333 $\pm$ 0.00373 <sup>(b)</sup>
	5 + 5	0.0103467 $\pm$ 0.00048 <sup>(a)</sup>	0.9095333 $\pm$ 0.00621 <sup>(a)</sup>
50	10 + 0	0.2717333 $\pm$ 0.01986 <sup>(d)</sup>	0.7781333 $\pm$ 0.01249 <sup>(ad)</sup>
	9 + 1	0.2010000 $\pm$ 0.01288 <sup>(c)</sup>	0.7586000 $\pm$ 0.01102 <sup>(c)</sup>
	7 + 3	0.1697000 $\pm$ 0.00366 <sup>(b)</sup>	0.7423000 $\pm$ 0.01032 <sup>(b)</sup>
	5 + 5	0.1094000 $\pm$ 0.02085 <sup>(a)</sup>	0.7815333 $\pm$ 0.02315 <sup>(ad)</sup>

The conclusion of the results presented in Table I is that increasing the amount of SDS in the emulsifier mixtures of OSA starch and SDS, used to stabilize emulsions, led to a decrease in consistency index  $K$ , meaning a reduction in the apparent viscosity of the emulsions. The reason for such behaviour lies in the fact that OSA starch is macromolecular emulsifier, which has a significant influence on the viscosity of continuous phase due to its macromolecular nature. A characteristic of polymers is their capability of modifying the viscosity of water. OSA starch replacement with a low-molecular mass emulsifier, such as SDS, which has a negligible effect on the viscosity, still resulted in a decrease in the viscosity of the continuous phase. The decrease in aqueous phase viscosity reflected on the consistency index of the emulsions. Changes in the SDS concentration did not show a specific dependence on the flow behaviour index  $n$ .

Increasing the oil concentration in the emulsions led to an increase in consistency index  $K$  due to the increase in the packing density of oil droplets, as well as a decrease in flow behaviour index  $n$ , indicating a pronounced shear thinning behaviour of the systems at higher oil contents. This phenomenon is related to the fact that the droplets are closer to one another at higher oil concentrations and they tend to flocculate. Small hydrodynamic forces at low shear rates are not able



to disrupt the flocs, but at higher shear rates, the flocs became deformed and eventually disrupted, causing a reduction in the emulsion viscosity.<sup>21,24,25</sup>

#### *Emulsion stability*

Emulsions with only OSA starch were more stable in terms of their creaming appearance compared to emulsions stabilized by the OSA starch–SDS mixtures.

Particularly, on comparing the occurrence of creaming of emulsions stabilized by OSA starch–SDS mixtures with those that were stabilized only by OSA starch, it was noticed that the addition of SDS enhanced the separation of the droplets. The influence of different amount of SDS in the OSA starch–SDS mixtures on the creaming rate of the examined emulsions is shown in Fig. 2.

In the presence of SDS, rapid creaming was noticed. In the emulsions that contained 5 % and 20 % of dispersed phase, for all OSA starch and SDS ratios, the creaming index reached maximal values after an hour. In addition, increasing the amount of SDS in the mixture led to an increase of the maximum values of creaming index for the emulsions with 20 and 50 % of the oil phase, while this effect was less pronounced in the emulsions with an oil content of 5 % (Fig. 2).

The emulsions stabilized only by OSA starch showed a slower creaming behaviour and the maximum value of the creaming index was achieved after two days. Increasing the SDS concentration in the OSA starch–SDS mixtures led to a decrease in the creaming time. Emulsions that contained 50 % oil showed a delay time of creaming. For the emulsions with an OSA starch–SDS ratio of 9:1, as well as for the emulsions stabilized only by OSA starch, the delay period was 72 h. The delay time decreased with increasing amount of SDS, and it was 24 h for an emulsifier ratio of 1:1.

A significant difference in the turbidity of the transparent serum layer was noticed between the emulsions that contained SDS and those that were stabilized only by OSA starch. Namely, the serum layer of the emulsion that did not contain SDS, after the appearance of creaming, was turbid, and the border of the two layers was not sharp. The serum layer became clearer during storage while the border between the two layers became more noticeable. This is typical behaviour for highly polydisperse systems, and is in accordance with the Stocks Law. The larger droplets move faster, while the smaller ones remain longer in the serum layer thereby making it turbid.

Thus, emulsions stabilized with OSA starch and SDS mixtures after the creaming had a clear serum layer without remaining oil droplets. According to Dickinson<sup>26</sup> and McClements,<sup>21</sup> surfactants micelles and non-adsorbed biopolymers can lead to destabilization of an emulsion system by the depletion flocculation mechanism, which occurred in these examined systems. The oil droplets moved faster during the creaming process because of flocculation and the clear serum layer evidenced that all droplets were in the top layer.



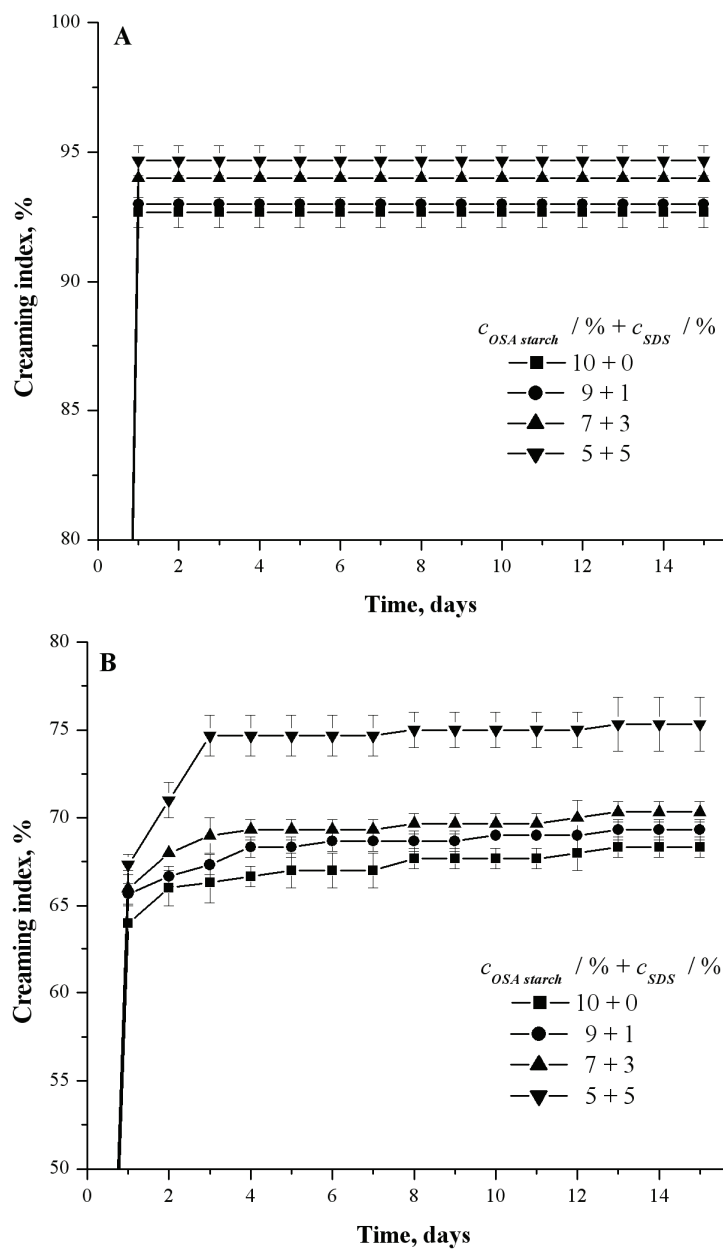


Fig. 2. The influence of OSA starch-SDS ratio on the creaming rate of the emulsions with different oil contents A) 5 and B) 20 %.

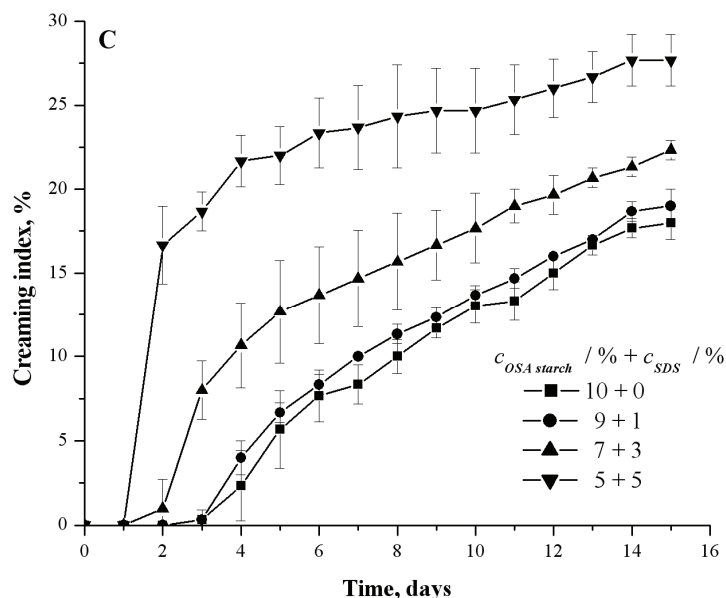


Fig. 2. (Continued) The influence of OSA starch–SDS ratio on the creaming rate of the emulsions with oil content 50 %.

#### CONCLUSIONS

From the presented results, it can be concluded that increasing the amount of SDS in OSA starch–SDS mixtures decreased the apparent emulsion viscosity and the mean diameter of the dispersed droplets. However, despite the slow adsorption, OSA starch molecules provide a better stability to the emulsion than a combination of OSA starch and SDS. This is because surfactant micelles and non-adsorbed biopolymers destabilize the emulsions by the depletion flocculation of the droplets when OSA starch and SDS are employed together.

*Acknowledgements.* This research was realized within the National Project No. 31014: “Development of new functional confectionery products based on oil crops”, financed by the Ministry of Education and Science of the Republic of Serbia.

#### ИЗВОД

УТИЦАЈ КОНЦЕНТРАЦИЈЕ НАТРИЈУМ ДОДЕЦИЛ СУЛФАТА НА ДИСПЕРЗИОНЕ И РЕОЛОШКЕ КАРАКТЕРИСТИКЕ ЕМУЛЗИЈА ТИПА УЉА У ВОДИ СТАБИЛИЗОВАНИХ СМЕШАМА ОСА СКРОБА И НАТРИЈУМ ДОДЕЦИЛ СУЛФАТА

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Стабилизација емулзија типа уља у води може се остварити хемијски модификованим скробом, октенил сукцинат анхидридом скроба – ОСА скробом, као емулгатором. У циљу испитивања дисперзионих и реолошких особина емулзија које садрже две врсте емулгатора део ОСА скроба може да буде замењен адекватном концентрацијом натријум-додецил-сулфата, који је сурфактант истог наелектрисања као и ОСА скроб. Садржај уља испитиваних емулзија износио је 5, 20 и 50 %. Поред одабране концентрације ОСА скроба од 10 %, натријум-додецил-сулфат је уведен у концентрацијама 1, 3 и 5 % заменом адекватног дела ОСА скроба. Методом одређивања величине и расподеле величине капи дефинисан је средњи пречник диспергованих капи – Саутеров пречник,  $d_{32}$ . Испитивањем кривих протицања утврђена су реолошка својства емулзија. Такође је праћена стабилност емулзија која је изражена преко криминг индекса. Добијени резултати указали су на смањење Саутеровог средњег пречника капи диспергованог уља и стандардне девијације и смањење вредности привидних вискозитета емулзија са повећањем удела натријум додецил сулфата у смеси емулгатора. Емулзије које су садржале само ОСА скроб, на основу праћења појаве криминга, показале су већу стабилност од емулзија стабилованих комбинацијом ОСА скроба и натријум-додецил-сулфата.

(Примљено 30. јуна 2010, ревидирано 7. фебруара 2011)

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