Fe₃O₄ nanoparticles: a highly efficient and easily reusable catalyst for the one-pot synthesis of xanthene derivatives under solvent-free conditions

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Abstract: Magnetically separable Fe_3O_4 nanoparticles supply an environmentally friendly procedure for the synthesis of 14-aryl-14*H*-dibenzo[*a,j*]xanthene and 1,8-dioxooctahydroxanthene derivatives. These compounds were obtained in high yields and short reaction times by the reaction of dimedone and 2-naphthol with various aromatic aldehydes under solvent-free conditions. The catalyst could be easily recovered using an external magnet and reused for six cycles with almost consistent activity.

Keywords: xanthenes, nanoparticles, solvent-free condition, magnetic catalyst, Fe_3O_4.

INTRODUCTION

Nanotechnology has been one of the most active research areas in recent years. The reactivity of catalytic nanoparticles is largely determined by the energy of surface atoms, which can be easily gauged by the number of neighboring atoms and by the bonding modes and accompanying energies of small molecules to be transformed on the surfaces of nanoparticles.[1,2] Magnetic nanoparticles are a class of nanostructured materials of current interest, due to the largely advanced technology and medical applications, envisioned or realized. Among the various magnetic nanoparticles under investigation, Fe_3O_4 nanoparticles (Fe_3O_4 NPs) are arguably the most extensively studied.[3] The main characteristic of these nanoparticles is the simple and convenient separation from a reaction media by magnetic separation.[4–7] Recently, magnetite nanoparticles were used as an efficient catalyst in many organic transformations.[8–12]

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Xanthene and its derivatives are an important class of organic compounds because of their wide range of biological and pharmaceutical properties.[13–15] In addition, these compounds are widely used in dyes,[16] laser technologies[17] and as pH-sensitive fluorescent materials.[18] There are several methods for the synthesis of xanthene derivatives, such as in the reaction of aryloxymagnesium halides with triethyl orthoformate,[19] cyclodehydration,[20] trapping of benzynes by phenols,[21] intramolecular reactions of benzaldehyde and acetophenones,[22] cyclization of polycyclic aryl triflate esters,[23] cyclocondensation of 2-hydroxy aromatic aldehydes[24] and the reaction of aldehydes, 2-naphthol and dimedone.[25] The preparation of xanthenes has been achieved *via* reaction of aldehydes and 2-naphthol by cyclodehydration using diverse catalysts, such as $H_4[SiW_{12}O_{40}]$,[26] PEG––SO_3H,[27] WCl_6,[28] poly(4-vinylpyridinium) hydrogen sulfate,[29] silica sulfuric acid,[30] $Cu(CH_3CN)_4PF_6$,[31] bismuth(III) chloride,[32] sulfonic acid functionalized silica (SiO_2–Pr-SO_3H),[33] $Sc[N(SO_2C_8F_{17})_2]_3$,[34] silica-supported ferric hydrogen sulfate,[35] P_2O_5 or $InCl_3$,[36] polystyrene-supported aluminum chloride,[37] nano TiO_2,[38] AgI nanoparticles,[39] organocatalysts[40] and functionalized SBA-15.[41]

The classical method for the synthesis of 1,8-dioxooctahydroxanthenes involves the condensation of two equivalent of 1,3-diketones, such as dimedone (5,5-dimethyl-1,3-cyclohexanedione) with various aldehydes,[42] catalyzed using different catalysts including *p*-dodecylbenzenesulfonic acid,[43] $InCl_3$/ionic liquid,[44] Fe^{3+}–montmorillonite,[45] $NaHSO_4$–SiO_2 or silica chloride,[46] amberlyst 15,[47] silica sulfuric acid[48] and 1-(carboxymethyl)-3-methylimidazolium tetrafluoroborate ([cmmim][BF_4]).[49]

In accordance to the above-mentioned importance of magnetite nanoparticles and the significant importance of xanthenes derivatives, it was thought that there is scope for further innovation towards milder reaction conditions, short reaction time and improvement of yield in the synthesis of xanthene derivatives. Herein a highly efficient and clean method for the synthesis of 14-aryl-14*H*-dibenzo[*a,j*]-xanthene and 1,8-dioxooctahydroxanthene derivatives using Fe_3O_4 nanoparticles as a green and robust catalyst under thermal and solvent-free conditions is reported (Scheme 1).

EXPERIMENTAL

The employed high purity commercial reagent grade chemicals were purchased from Merck and Fluka. Fe_3O_4 nanoparticles were prepared according to the procedure reported by Zhang *et al.*[9] The IR spectra were recorded as KBr pellet on a Perkin-Elmer 781 spectrophotometer and an Impact 400 Nicolet FT-IR spectrophotometer. The ^{1}H- and ^{13}C-NMR spectra were recorded in CDCl$_3$ solvent on a Bruker DRX-400 spectrometer (400 MHz) using TMS as an internal reference. The mass spectra were recorded on a Joel D-30 instrument at an ionization potential of 70 eV. All melting points are uncorrected and were determined in a capillary tube on Boetius melting point microscope. The purity determination of the substrates and reactions monitoring were accomplished by TLC on silica gel polygram SILG/UV 254 plates. The elemental analyses (C, H, N) were realized using a Carlo Erba Model EA 1108

analyzer. Powder X-ray diffraction (XRD) patterns were obtained on a Philips diffractometer of the X'pert Company using mono chromatized Cu K_α radiation ($\lambda = 1.5406$ Å). The microscopic morphology of the products was visualized by SEM (LEO 1455VP).
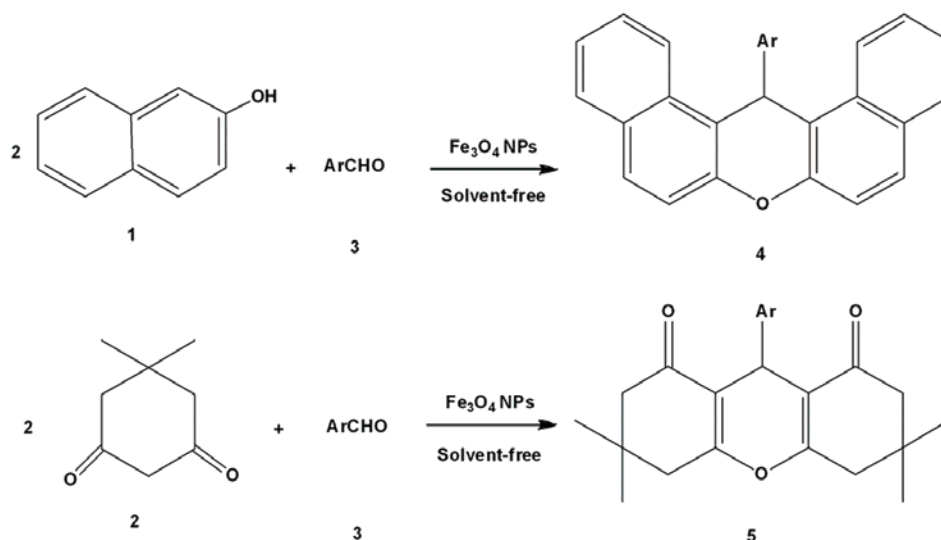
Scheme 1. One-pot synthesis of 14-aryl-14*H*-dibenzo[*a,j*]xanthenes and 1,8-dioxo-octahydroxanthenes.

Preparation of magnetic Fe₃O₄ nanoparticles

To a solution of FeCl₂·4H₂O (2.5 g) and FeCl₃·6H₂O (6 g) in 30 ml deionized water was added dropwise 1.0 mL of concentrated hydrochloric acid at room temperature. The solution was added in to 300 mL of 1.5 mol L^{-1} NaOH and then the solution was stirred vigorously at 70 °C until precipitation. Afterwards, the prepared magnetic nanoparticles were separated magnetically, washed with deionized water and then dried at 70 °C for 8 h.

General procedure for the preparation of 14-aryl-14H-dibenzo[a,j]xanthene derivatives (4a–o)

A mixture of 2-naphthol (0.14 g, 1 mmol), various aldehydes (0.5 mmol) and Fe₃O₄ NPs (0.02 g, 0.1 mmol, 10 mol %) was heated at 100 °C for 20–40 min. Progress of the reaction was continuously monitored by TLC. On completion of the reaction, the reaction mixture was cooled to room temperature and dissolved in 10 mL of dichloromethane. The catalyst is insoluble in CH₂Cl₂ and was separated magnetically.[4–7] The solvent was evaporated and the obtained solid was recrystallized from ethanol to afford the pure 14-aryl-14*H*-dibenzo[*a,j*]-xanthenes.

General procedure for the preparation of 1,8-dioxooctahydroxanthene derivatives (5a–o)

A mixture of 5,5-dimethyl-1,3-cyclohexanedione (0.14 g, 1 mmol), various aldehydes (0.5 mmol) and Fe₃O₄ NPs (0.02 g, 0.1 mmol, 10 mol %) was heated at 100 °C for 15–40 min. During the procedure, the reaction was monitored by TLC. Upon completion, the reaction mixture was cooled to room temperature and then dichloromethane was added. The catalyst is insoluble in CH₂Cl₂ and was separated magnetically. The solvent was evaporated and the

obtained solid was recrystallized from ethanol to afford the pure 1,8-dioxooctahydroxan-thenes.

Spectral data for all of the compounds are reported in the Supplementary material to this paper.

Recycling and reuse of the catalyst

After completion of the reaction, the reaction mixture was dissolved in dichloromethane and then the catalyst was separated magnetically. The magnetic Fe_3O_4 nanoparticles were washed three to four times with CH_2Cl_2 (3 ml) and then dried at 100 °C for 5 h. The separated catalyst was used in six cycles with only a slight decrease in activity, as shown in Fig. 1.

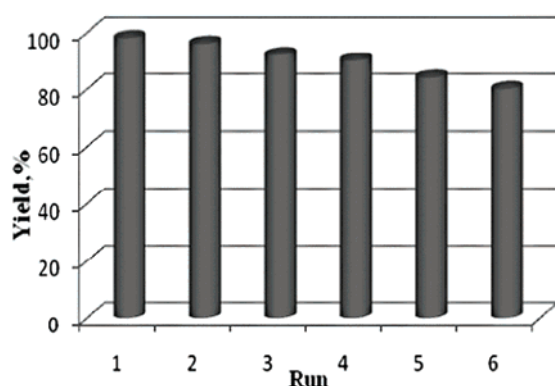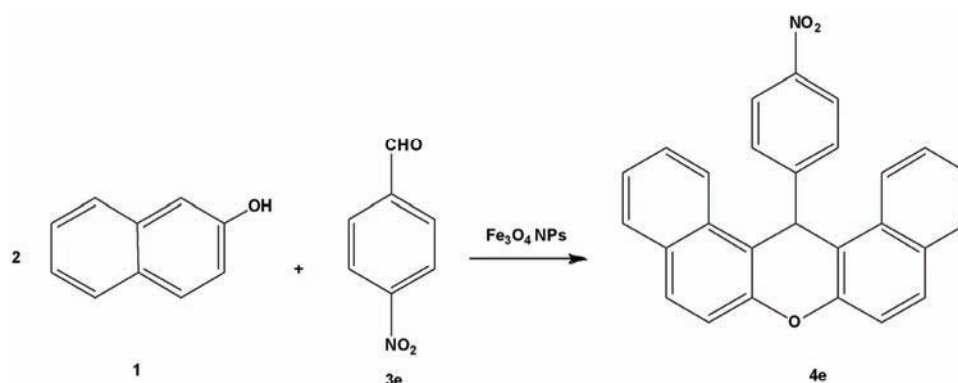Fig. 1. Recoverability of Fe_3O_4 nano-particles.

RESULTS AND DISCUSSION

In order to optimize the reaction conditions, the reaction of 4-nitrobenzalde-hyde and 2-naphthol was selected as a model system (Scheme 2).

Scheme 2. The model study for the one-pot synthesis of corresponding xanthene.

Initially, to show the merit of the present approach in comparison with other catalysts, the model reaction was performed in the presence of various catalysts, such as CuO, MgO, SiO_2 and Fe_3O_4, for the synthesis of the 14-aryl-14*H*-di-benzo[*a,j*]xanthene derivative. As shown in Table I, the magnetite nanoparticles

were the best catalysts with respect to reaction time and yield of the obtained product.

TABLE I. The model reaction catalyzed by various catalysts; yields refer to the isolated products

Entry	Catalyst	Time, min	Yield, %
1	CuO	80	60
2	MgO	87	50
3	Fe_3O_4	60	80
4	SiO_2	90	65

Three types of catalysts were examined, *i.e.*, Fe_3O_4, Fe_3O_4 nanoparticles and recovered Fe_3O_4 nanoparticles. The highest yield of the product was obtained using Fe_3O_4 NPs, which may be due to the better diffusion of nano Fe_3O_4 in the reaction mixture. Astonishingly, the recovered catalyst acted almost the same as the fresh Fe_3O_4 NPs. The results are shown in Fig. 2.
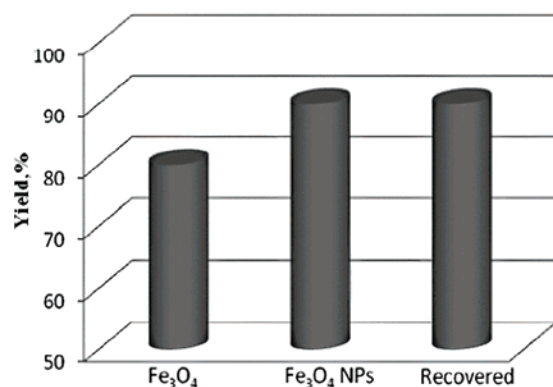

Fig. 2. The effect of different types of Fe_3O_4 in the model reaction.

In order to investigate the morphology and particle size of the Fe_3O_4 nanoparticles, SEM images of the magnetic nanoparticles were taken and the images are presented in Figs. 3a and 3b. These results show that spherical Fe_3O_4 nanoparticles were obtained with an average size between 40–50 nm.

The XRD pattern of the Fe_3O_4 nanoparticles is shown in Fig. 4. All reflection peaks can be readily indexed to pure cubic crystal phase of Fe_3O_4 with F-33m space group (JCDPS No. 75-0449). In addition, no specific peaks due to any impurities were observed. The broad peaks indicate that the particles are in nanoscale size range. The crystallite diameter (D) of the magnetic nanoparticles was calculated by Debye–Scherrer Equation ($D = K\lambda/\beta\cos\theta$), where β, the FWHM (full-width at half-maximum or half-width), is in radians and θ is the position of the maximum of the diffraction peak, K is the so-called shape factor, which usually takes a value of about 0.9, and λ is the X-ray wavelength (1.5406

Å for Cu K$_\alpha$). The average crystallite size of the prepared nano-Fe$_3$O$_4$ was found by SEM analysis to be 45 nm.
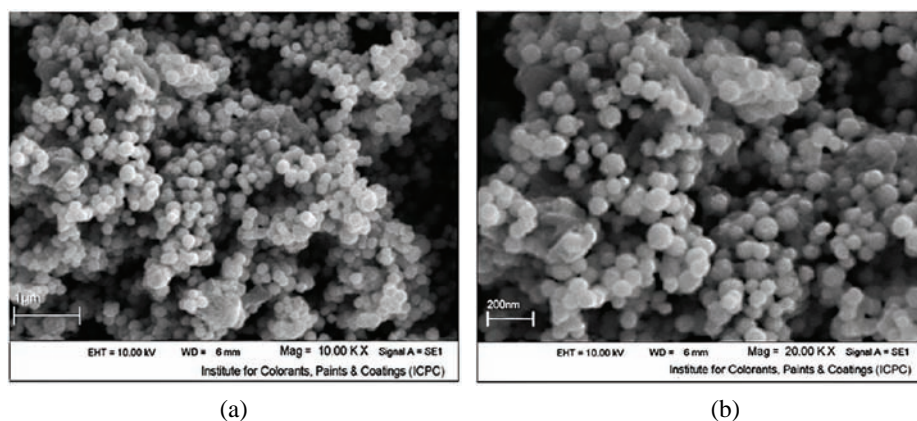

(a)   (b)

Fig. 3. SEM images of Fe$_3$O$_4$ nanoparticles; magnification: a) 10000×, b) 20000×.

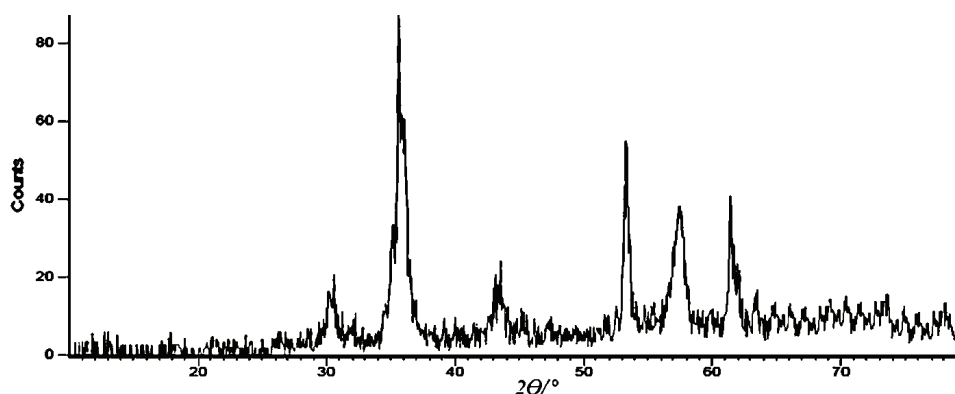Fig. 4. The XRD pattern of the Fe$_3$O$_4$ nanoparticles.

The experimental research was continued using different amounts of nano-catalyst in the reaction involving 4-nitrobenzaldehyde (1 mmol) and 2-naphthol (2 mmol) to afford the product 14-(4-nitrophenyl)-14*H*-dibenzo[*a,j*]xanthene under solvent-free conditions at 100 °C. From these experiments, the optimum amount of this catalyst was found to be 10 mol %. Increasing this amount did not result in any significant changes in the yield and time of reaction (Table II).

Then, the effect of solvent was investigated. Thus, the model reaction in the presence of magnetite nanoparticles was run using different solvents and under solvent-free conditions. As shown in Table III, it is noteworthy that the solvent-free conditions afforded the product in excellent yield and shorter reaction time than under solvent conditions.

TABLE II. The effect of the amount of Fe_3O_4 nanoparticles on the model reaction; reaction conditions: 2-naphthol, 2 mmol, 4-nitrobenzaldehyde, 1 mmol, at 100 °C

Entry	Catalyst amount, mol %	Time, min	Yield[a], %
1	–	180	15
2	4	45	55
3	7	30	70
4	10	20	92
5	15	20	92

[b]Isolated yields

In continuations of on-going research using nanoparticles in organic reactions,[50–52] the test reactions of aldehydes with dimedone were encouraging. Thus, the optimized reaction conditions in the presence of Fe_3O_4 NPs were employed to produce 1,8-dioxooctahydroxanthene derivatives. A series of experiments were performed in which a number of 1,8-dioxooctahydroxanthenes were prepared in high yields and short reaction times.

TABLE III. The model reaction in various solvents catalyzed by Fe_3O_4 nanoparticles; yields refer to the isolated products

| Entry	Solvent	Time, min	Yield, %
1	CH_2Cl_2	180	60
2	THF	120	70
3	toluene	210	45
4	ethanol	90	75
5	No solvent	20	92

In the present work, magnetic Fe_3O_4 nanoparticles were successfully prepared and then used for the synthesis of 14-aryl-14*H*-dibenzo[*a,j*]xanthene and 1,8-dioxooctahydroxanthene derivatives. Application of this catalytic system effectively gives the desired products in excellent yields (Table IV). As seen from Table IV, aromatic aldehydes with electron-withdrawing groups as expected reacted faster than those with electron-releasing groups. Sterically hindered aromatic aldehydes required longer reaction times.

A plausible mechanism for the synthesis of 1,8-dioxooctahydroxanthene derivatives using magnetite nanoparticles as catalyst is shown in Scheme 3. Based on the present experimental results together with some literature data,[54–57] it is supposed that catalytically active site of Fe_3O_4 NPs is Fe^{3+} that behaves as a Lewis acid and coordinates to the carbonyl groups of dimedone and the aldehyde. This interaction accelerates the conjugation and directs the additions of the nucleophiles to the corresponding substrates. Finally, product **5** was obtained and the Fe_3O_4 nanoparticles were released for further reactions. Moreover, the effect of the catalytic behavior of Fe_3O_4 NPs in the synthesis of 14-aryl-14*H*-dibenzo[*a,j*]xanthenes was the same as the above-mentioned mechanism for the preparation of 1,8-dioxooctahydroxanthenes.

TABLE IV. Synthesis of 14-aryl-14*H*-dibenzo[*a,j*]xanthenes (**4**) and 1,8-dioxooctahyd-roxanthenes (**5**) by Fe_3O_4 nanoparticles

Entry	Ar	Products **4a–o** Time, min/Yield[a], %	M.p. / °C	Products **5a–o** Time, min/Yield, %	M.p. / °C
a	C_6H_5	30/88	184–185[26]	30/89	203–205[43]
b	4-MeOC_6H_4	35/85	203–205[26]	35/85	242–245[43]
c	4-ClC_6H_4	20/90	289–290[26]	20/88	230–233[43]
d	4-BrC_6H_4	20/90	297–298[26]	20/88	226–229[43]
e	$4\text{-NO}_2C_6H_4$	20/92	311–312[26]	15/90	225–226[43]
f	4-MeC_6H_4	35/86	227–229[26]	35/87	212–214[43]
g	$2\text{-NO}_2C_6H_4$	25/89	215–217[28]	30/89	246–248[43]
h	4-FC_6H_4	20/91	239–240[26]	20/90	225–227[49]
i	3-ClC_6H_4	35/85	210–212[28]	35/86	184–185[49]
j	4-OHC_6H_4	40/85	135–136[26]	40/85	244–246[43]
k	$3\text{-NO}_2C_6H_4$	20/90	210–211[26]	20/89	165–167[49]
l	$4\text{-(CH}_3)_2\text{CHC}_6H_4$	35/85	152–154[36]	40/80	203–206[b]
m	4-NCC_6H_4	30/92	209–211[53]	25/90	216–217[53]
n	4-MeSC_6H_4	35/85	264–266[39]	35/86	256–257[b]
o	4-CHOC_6H_4	30/90	252–254[39]	30/89	211–213[b]

[a]Isolated yield; [b]new compounds
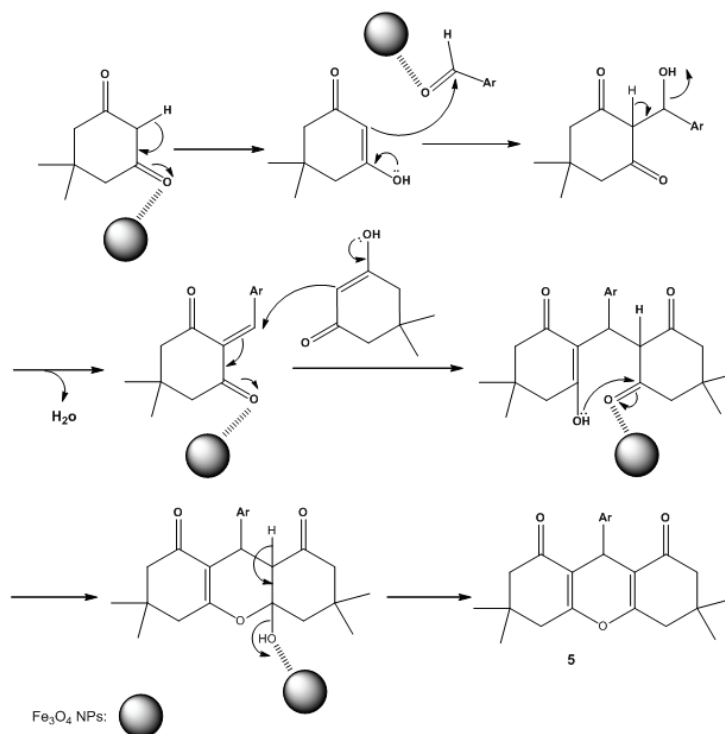
Scheme 3. Proposed reaction pathway for the synthesis of 1,8-dioxooctahydroxanthenes by Fe_3O_4 nanoparticles.

CONCLUSIONS

In conclusion, the efficient one-pot syntheses of 14-aryl-14*H*-dibenzo[*a,j*]-xanthenes and 1,8-dioxooctahydroxanthene derivatives from 2-naphthol, dimedone with various aldehydes using magnetically separable Fe_3O_4 nanoparticles, found to be an eco-environmentally friendly catalyst under solvent-free conditions, was demonstrated. The simplicity, easy workup, as well as safety and reusability of the catalyst are the advantages of this procedure over previously reported ones.

SUPPLEMENTARY MATERIAL

Spectral data for all of the compounds are available electronically from http://www.shd.org.rs/JSCS/, or from the corresponding author on request.

И З В О Д

Fe$_3$O$_4$ НАНОЧЕСТИЦЕ: ВИСОКО ЕФИКАСАН КАТАЛИЗАТОР ЗА СИНТЕЗУ ДЕРИВАТА КСАНТЕНА У ЈЕДНОМ РЕАКЦИОНОМ КОРАКУ, БЕЗ УПОТРЕБЕ РАСТВАРАЧА

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Наночестице Fe$_3$O$_4$, погодне за издвајање магнетом, омогућавају еколошки прихватљив поступак за синтезу деривата 14-арил-14*H*-дибензо[*a,j*]ксантена и 1,8-диоксо-октахидроксантена. Једињења су добијена из димедона и 2-нафтола са различитим ароматичним алдехидима, без растварача у високом приносу и кратком реакционом времену. Катализатор се може изоловати уз помоћ екстерног магнета и поново користити у шест реакционих циклуса без већег губитка активности.

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