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Cellulose/Fe_3O_4/Co_3O_4 Nanocomposite as a Highly Efficient and Reusable Catalyst for the Synthesis of 1-((Benzo[d]thiazol-2-ylamino)(aryl)-methyl)naphthalen-2-ol Derivatives

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Magnetic Cellulose/Fe_3O_4/Co_3O_4 nanocomposite as a new catalyst was synthesized and characterized by FT-IR, XRD, TGA, SEM, TEM, ICP-AES and VSM techniques. The catalytic activity of this heterogeneous catalyst was studied through one-pot synthesis of 1-((benzo[d]thiazol-2-ylamino)(aryl)-methyl)naphthalen-2-ol derivatives from three component reactions of aromatic aldehydes, 2-aminobenzothiazole and β-naphthol. This benign and economical nanocatalyst was easily recovered using an external magnet and reused in at least five successive runs with no significant loss of activity, and provided clean production in a short reaction time.

Keywords: Magnetic properties, Aldehyde, 2-Aminobenzothiazole, β-Naphthol, Cellulose/Fe_3O_4/Co_3O_4

INTRODUCTION

Nanomaterials are of great interest in organic synthesis due to their extremely small size and large surface to volume ratio, which lead to both chemical and physical property differences compared to bulk of the same chemical composition, such as mechanical and biological properties, higher catalytic activity, thermal and electrical conductivity, optical absorption and melting point [1-3]. Surface functionalized iron oxide magnetic nanoparticles (MNPs) have been widely used in biotechnology and catalysis [4-9]. Magnetic nanocatalysts can easily be separated and recycled from the products by their response to an external magnet. Good biocompatibility and biodegradability as well as basic magnetic characteristics could be designated for functional organic materials grafted to magnetic nanoparticles [10-13].

Cellulose has very long been an indispensable raw material and it is no great leap to credit the evolution of mankind, in some part, to its effective manipulation. The relationship between human industriousness and cellulose is ancient, perhaps most significantly beginning in the Stone Age with the burning of woods and grasses and progressing into current times to help meet the needs of industrialized man. Systematic comparative study of nanocellulose particles as a biodegradable and renewable reinforcing agent can help develop criteria for selecting an appropriate candidate to be incorporated in nanocomposites. Of particular interest has been nanocellulosic materials including cellulose nanocrystal (CNC) and micro/nanofabricated cellulose (MFC/NFC) possessing a hierarchical structure that permits an ordered structure with unique properties that has served as building blocks for the design of green and novel materials composites for applications in flexible electronics, medicine and composites [14-17].

Composite materials include two main components of continuous phase (a matrix which is mostly a polymer resin) and a discrete phase dispersed in the matrix. The aim of nanocomposite engineering is to create novel materials whose properties are improved somewhat in comparison to those of the individual components, with the beauty of the material often derived from the combination of dissimilar

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entities. The greatest challenge to the successful preparation of nanocomposites is the requirement of intimate mixing of components. There exist various approaches to promote miscibility, such as using common solvents, high energy sonication treatments, surfactants, chemical modification, *etc*. In the case of cellulose-based nanocomposites, the cellulosic component may play the role of either nanoparticle filler or polymer host, with the method used to produce a homogeneous material dependent upon the chemical nature of the different material elements [18-19]. For example, superparamagnetic nanocomposite films produced by synthesizing iron oxide (Fe_2O_3) nanoparticles within the pores of regenerated cellulose films [20], and $CaCO_3$-cellulose nanocomposites produced by synthesizing the $CaCO_3$ nanoparticles in the presence of hardwood [21].

Multi-component reactions (MCRs) belong to the most challenging areas of modern chemistry for several reasons, such as reducing the number of steps, time saving, material saving, higher yield compared to other multistep reactions and fewer by-products [22-24]. Synthesis of medicine and complex molecules should be easy, and efficient with minimal workup in this methodology. In addition, these kinds of molecules can be synthesized from simple starting materials and can be simply purified with high selectivity [25-28].

In continuation of our previous efforts to develop new organic-inorganic hybrid materials as heterogeneous catalysts [29-33], herein, we wish to introduce a new reusable and efficient heterogeneous magnetic Cellulose/Fe_3O_4/Co_3O_4 nanocomposite catalyst and its catalytic application for the synthesis of 1-((benzo[d] thiazol-2-ylamino)(aryl)-methyl)naphthalen-2-ol derivatives from the three-component reactions of aromatic aldehydes, 2-aminobenzothiazole and β-naphthol at 60 °C in ethanol solvent, as shown in Scheme 1.

EXPERIMENTAL

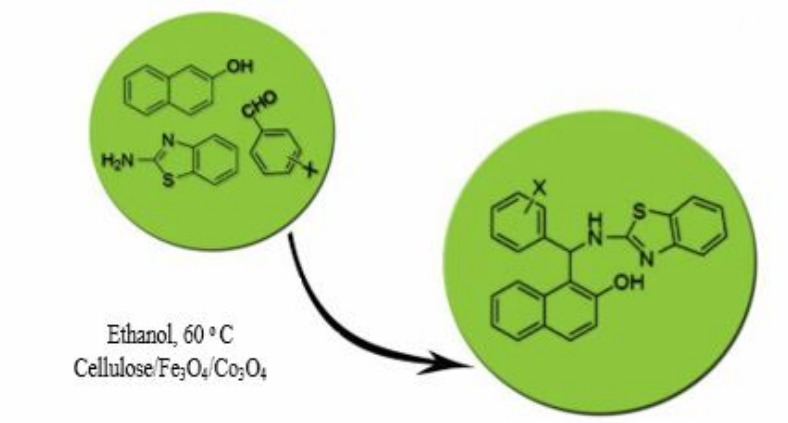
Chemicals and Materials

The materials were purchased from Merck and used without any purification. All reactions were monitored by TLC. Melting points were measured on an Electrothermal 9100 apparatus. X-ray powder diffraction (XRD) patterns were recorded with a Philips PW 1830 X-ray diffractometer using CuKα source (λ = 1.5418 Å) in a range of Bragg's angle (10-80°) at room temperature. Scanning electron microscopy (SEM) analysis was taken using a VEGA//TESCAN KYKY-EM 3200 microscope (acceleration voltage 26 kV). Transmission electron microscopy (TEM) experiments were conducted on a Philips EM 208 electron microscope. Thermogravimetric analysis (TGA) was obtained with a Stanton Red craft STA-780 (London, UK). NMR spectra were recorded by a Bruker DRX-400 AVANCE instrument (300.1 MHz for ^{1}H, 75.5 MHz for ^{13}C). The spectra were obtained in DMSO-d_6 as solvent. FT-IR spectra were obtained with a Bruker vector 22 spectrophotometer. Magnetic measurements were performed using vibrating sample magnetometer (VSM, MDK, and Model 7400) analysis. Energy-dispersive X-ray (EDX) spectroscopy patterns were obtained using a Seron model AIS2300C. The cobalt loading was detected by inductively coupled plasma-atomic emission spectrometer (ICP-AES, Varian Vista MPX).

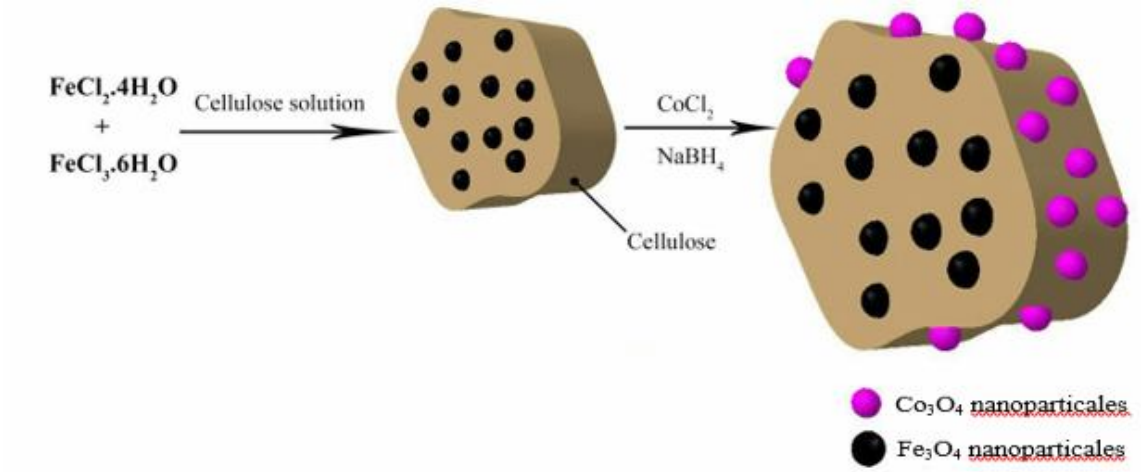
GENERAL PROCEDURE

Preparation of Catalyst

A 250 ml flask containing a mixture of 80 ml deionized water, 12 g urea and 7 g NaOH was immersed in a heat bath at -8 °C, and 4 g cellulose was dissolved in this solution. $FeCl_3.6H_2O$ (1 g, 4 mmol) and $FeCl_2.4H_2O$ (0.5 g, 2.5 mmol) were dissolved in 50 ml distilled water with vigorous stirring at room temperature, and then this solution was added drop wise into the cellulose solution for 2.5 h. The precipitated magnetic Cellulose/Fe_3O_4 was separated from the aqueous solution by magnet, and it was washed with deionized water four times and dried in an oven for 10 h. Then, 1 g of the obtained ferromagnetic Cellulose/Fe_3O_4 was dispersed in 50 ml deionized water and sonicated for 1 h. To this solution, 0.8 g $CoCl_2$ and 3 ml $NaBH_4$ were added and the pH of mixture maintained at 12 by using NaOH (0.5 M) and stirring for 12 h. Finally, the precipitated Cellulose/Fe_3O_4/Co_3O_4 nanocomposite was separated by a magnet and washed several times with deionized water (Scheme 2).



Scheme 1. Synthesis of 1-((benzo[d]thiazol-2-ylamino)(aryl)-methyl)naphthalen-2-ol derivatives using Cellulose/ Fe_3O_4/Co_3O_4 nanocomposite as a nanomagnetic catalyst.



Scheme 2. Preparation of magnetic Cellulose/Fe_3O_4/Co_3O_4

General Procedure for the Synthesis of 1-((Benzo[d]thiazol-2-ylamino)(aryl)-methyl) naphthalen-2-ol Derivatives at Ambient Conditions

A mixture of aldehyde (1 mmol), 2-aminobenzothiazole (1 mmol, 0.15 g) and β-naphthol (1 mmol, 0.144 g) and the catalyst (0.84 mmol%) in ethanol (3 ml) was stirred in 60 °C for the specific time. After completion of the reaction as indicated by TLC, the catalyst was separated with an external magnetic for the reaction mixture. The solvent of the solution containing the product was evaporated to give the solid product. The solid was recrystallized from ethanol to give the pure solid. All desired products were characterized by comparison of their physical data with those of known compounds.

RESULTS AND DISCUSSION

Characterization of the Synthesized Cellulose/Fe_3O_4/Co_3O_4 Nanocomposite

Fourier Transform Infrared (FT-IR) Analysis

In the FT-IR data of cellulose, the characteristic absorbing peaks are the O-H stretching vibration around

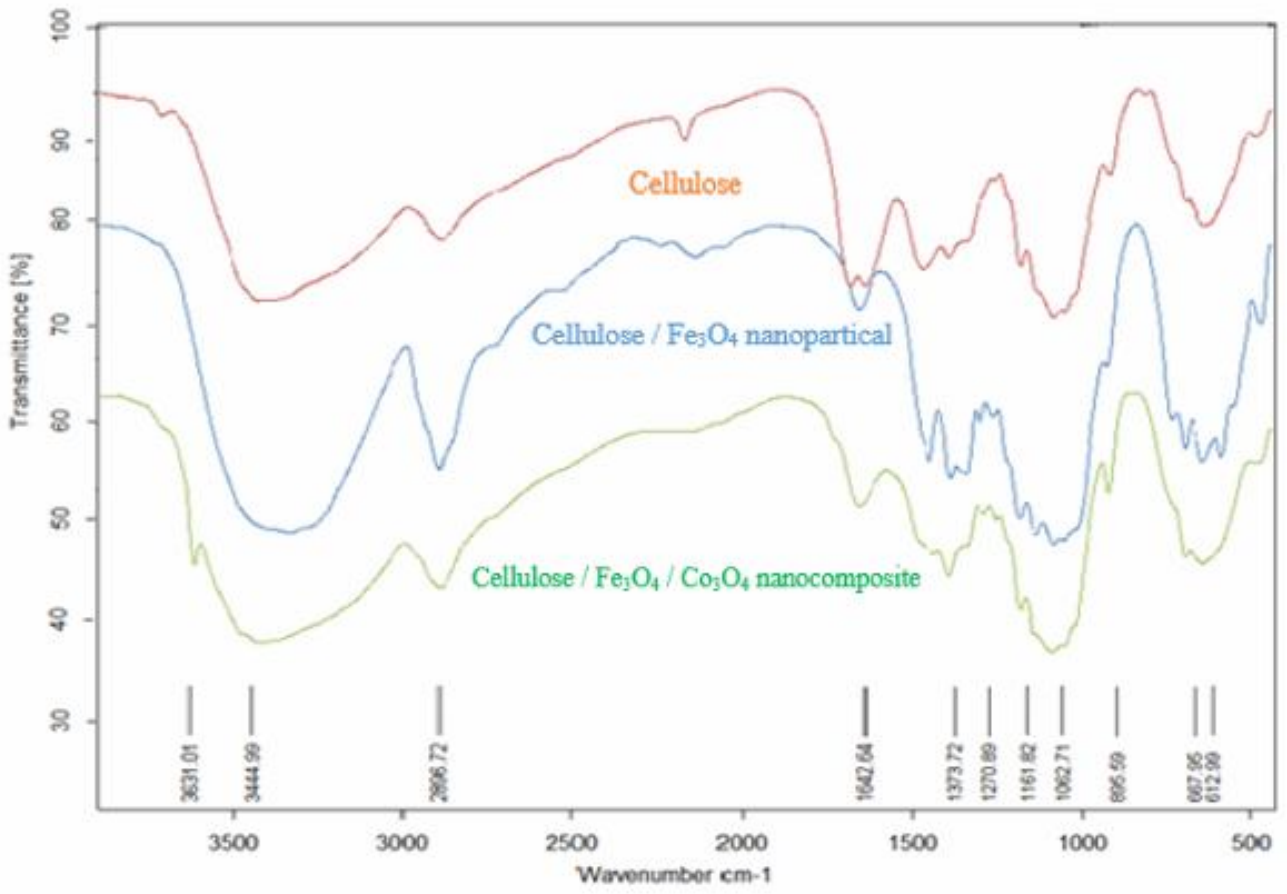


Fig. 1. FT-IR spectra for Cellulose, Cellulose/ Fe_3O_4 nanoparticle and Cellulose/ Fe_3O_4/Co_3O_4 nanocomposite.

3444 cm^{-1}, C-H stretching vibration at 2896 cm^{-1}, and the H-C-H and O-C-H in plane bending at 1373 cm^{-1}. The characteristic absorbing peaks of Fe_3O_4 are appeared at 667 and 612 cm^{-1}, which can be ascribed to the vibrations of Fe-O group. Therefore, the data obtained from FT-IR spectroscopy can confirm the existence of the nanomagnetic particle in the structure of Cellulose/Fe_3O_4/Co_3O_4 nanocomposite (Fig. 1).

X-ray Diffraction (XRD) Analysis and Energy-dispersive X-ray Spectroscopy (EDX)

X-ray diffraction using a Cu K_α irradiation was used to characterize the preservation of the crystal structure of the samples after the functionalization step. The results shown in Fig. 1 was fitted for the six peaks with the following Miller indices: (2 2 0), (3 1 1), (4 0 0), (4 2 2), (5 1 1), and (4 4 0) that the existing phases were identified as Fe_3O_4 nanoparticles and the other peak at 2Θ = 20 with the following Miller index : (1 1 0) that is related to cellulose [34] (Fig. 2). The average particle diameter was determined by the Scherrer equation [35,36]. The calculation led to particle sizes of about 13 nm for the nanocatalyst which was in a good agreement with TEM observations.

The characteristic peaks of Co species such as Co(0), CoO and Co_3O_4 in the XRD pattern are different [37]. Since the characteristic peaks of the above species were not observed in the XRD pattern, the presence of these species in the catalyst cannot be ruled out. It was observed that sometimes the diffraction peaks of Co species are not observed in the XRD spectrum possibly due to small size of Co species in which they homogeneously disperse on the surface of the Fe_3O_4 nanoparticles. In addition, the Co

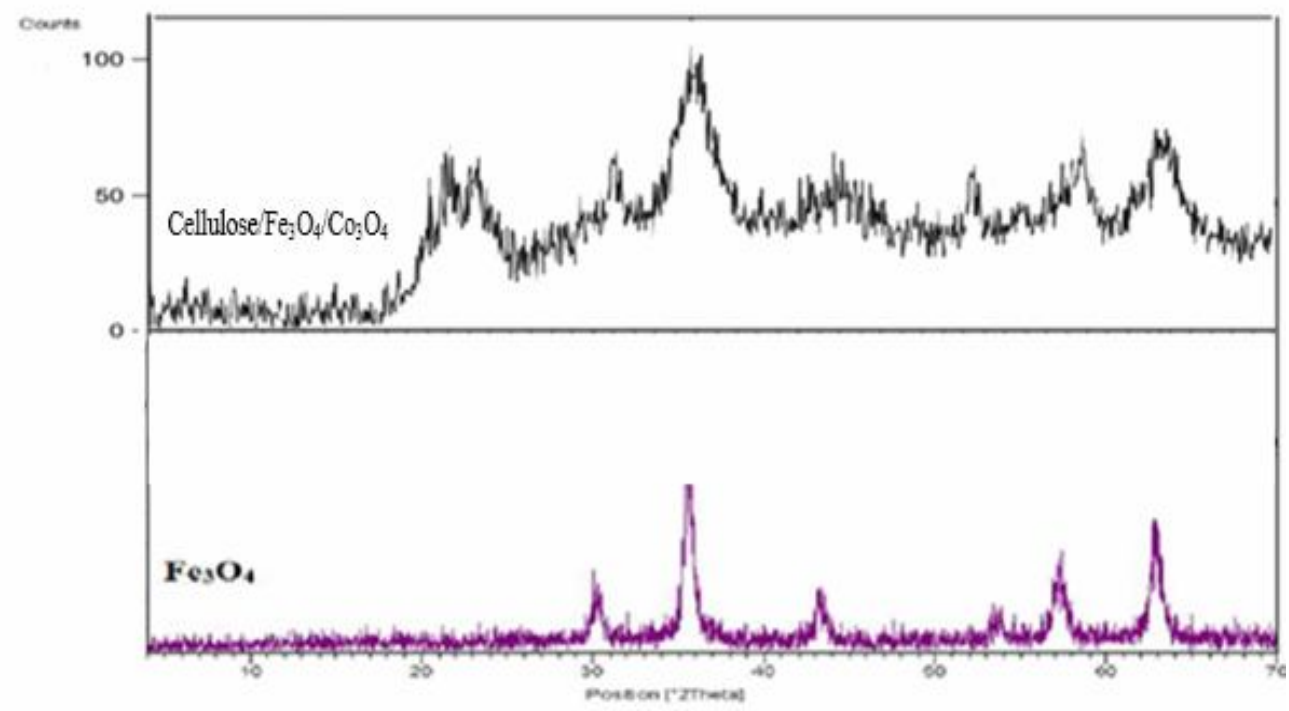


Fig. 2. XRD pattern of Cellulose/Fe_3O_4/Co_3O_4 nanocomposite.

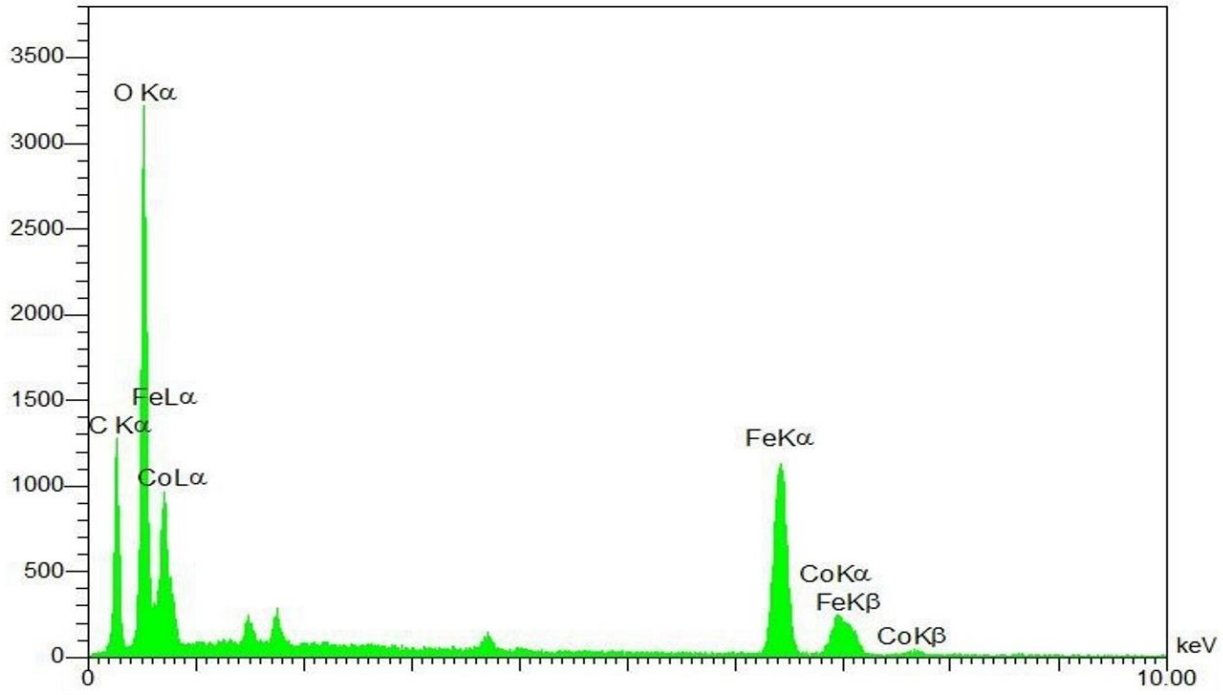


Fig. 3. EDX pattern of Cellulose/Fe_3O_4/Co_3O_4 nanocomposite.

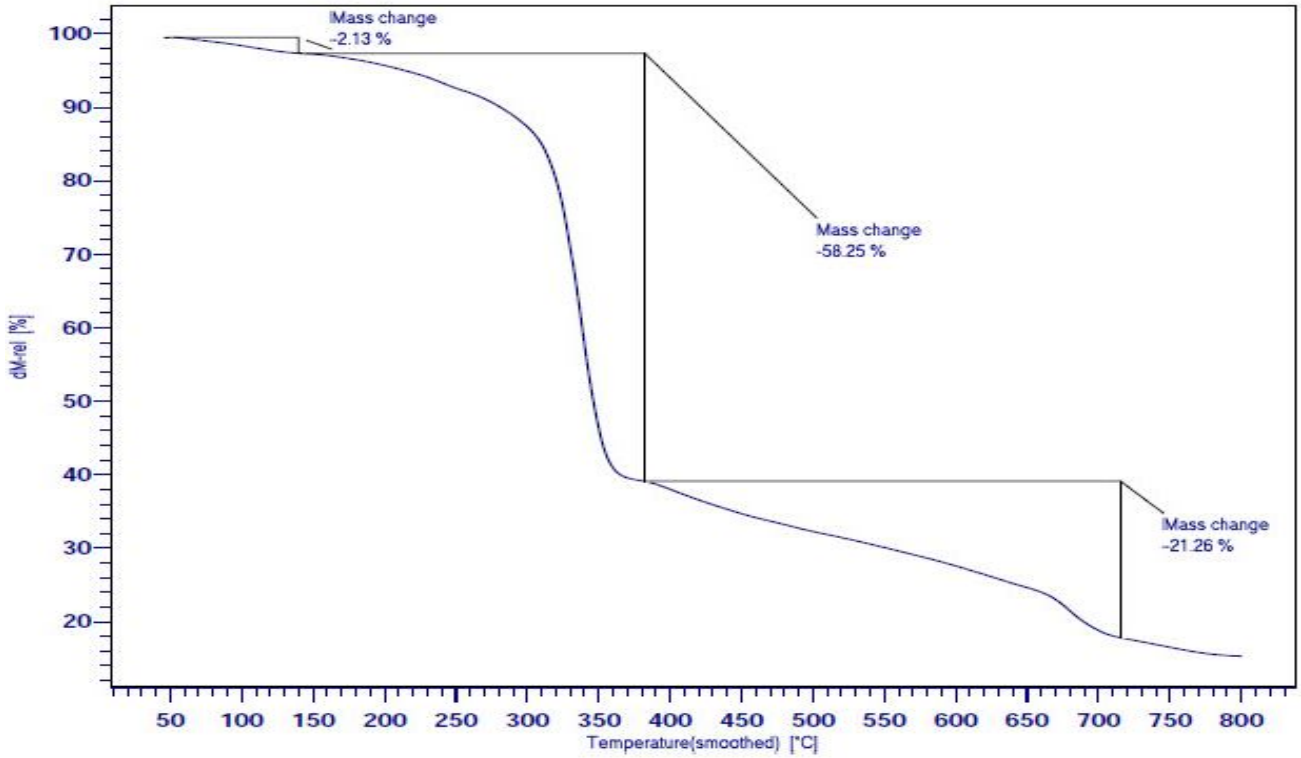


Fig. 4. TGA thermograms of cellulose/ Fe_3O_4/Co_3O_4 nanocomposite.

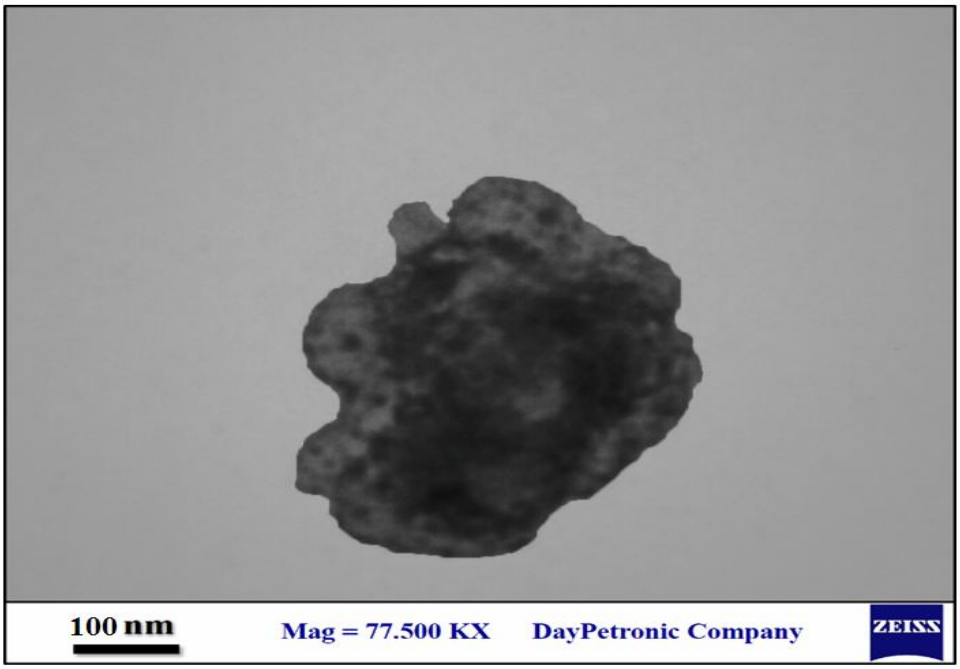
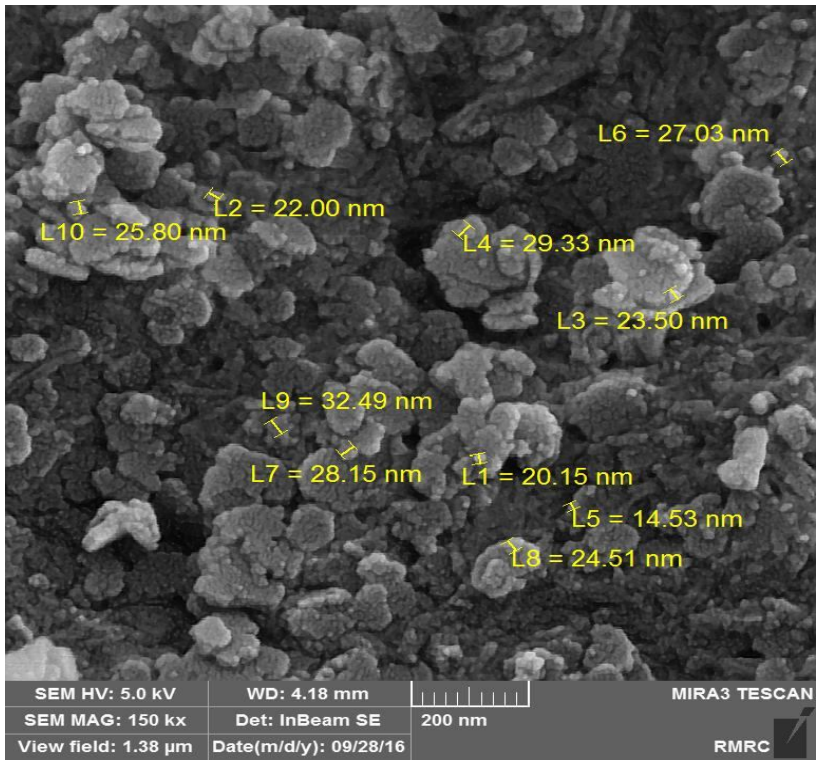


Fig. 5. TEM micrographs of magnetic Cellulose/Fe_3O_4/Co_3O_4.



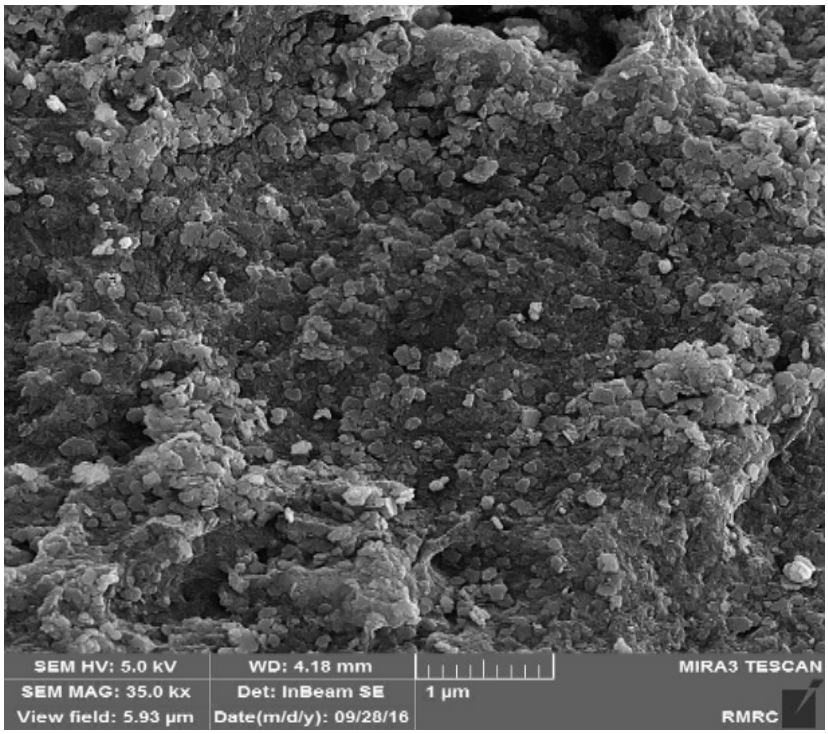


Fig. 6. The SEM images of Cellulose/Fe_3O_4/Co_3O_4 nanocomposite.

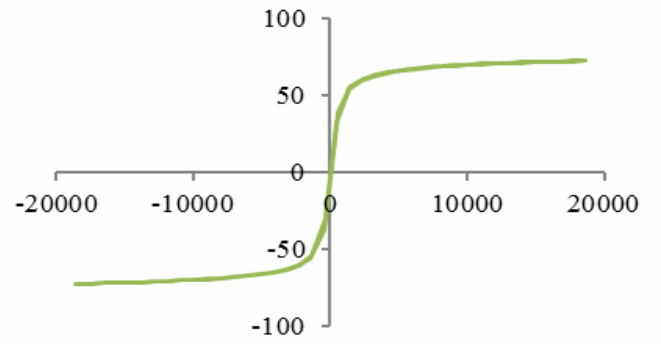


Fig. 7. Room temperature magnetization curves of Cellulose/Fe_3O_4/Co_3O_4 nanocomposite.

Table 1. Optimization of the Amount of Cellulose/Fe_3O_4/Co_3O_4 Nanocomposite as Catalyst in the Synthesis of 1-((Benzo[d]thiazol-2-ylamino)(phenyl)methyl)naphthalen-2-ol under Different Temperatures

Entry	Catalyst (g)	Temperature (^oC)	Time (min)	Yield (%)[a]
1	3	25	60	-
2	4	25	60	10
3	5	25	60	22
4	6	25	60	40
5	3	60	35	78
6	4	60	27	85
7	**5**	**60**	**20**	**92**
8	6	60	20	92

[a]Yields refer to the isolated products.

species cannot be seen, because the amount of these species are low [38]. In the literature it was discussed that even though Co^{2+} was reduced to a $Co^{(0)}$ nanoparticle by $NaBH_4$ during the catalyst preparation, it was oxidized to a high valence state species. For example, metal nanoparticles such as Ru are active and sensitive to oxygen and are oxidized into a high oxidation state when exposed to oxygen during storage [39]. As a consequence, the surface of all Co-containing particles in the catalyst consists the Co_3O_4 species. In addition, it was reported that CoO on the surface is not stable and thus it is oxidized to Co_3O_4 [40]. Therefore, it can be concluded that Co_3O_4 is responsible for catalysing the reaction.

Energy dispersive X-ray spectroscopy (EDX) data for the obtained nanomaterials also indicate the presence of iron, oxygen, cobalt and carbon in the structure of the catalyst (Fig. 3). The presence of oxygen and cobalt in the catalyst confirmed that Co species are in the Co_3O_4 form.

Thermogravimetric Analysis (TGA)

As shown in Fig. 4, information about loading of cellulose with Fe_3O_4/Co_3O_4 was obtained by TGA. The first weight losses, about 58.25%, was observed at around 120-350 °C that is related to the decomposition of cellulose in the nanocomposite (volatile components disappearing at about 100 ºC were neglected). The second weight losses occurred at 370-710 °C that is likely due to the degradation of oxide content in MNPs [41]. On the basis of these results, the well grafting of Fe_3O_4/Co_3O_4 on the cellulose is confirmed.

Transmission Electron Microscopy (TEM)

The morphology of the catalyst was surveyed by TEM, as shown in Fig. 5. Based on the TEM micrographs, the average size of nanocomposite was about 15 nm that was in a very good settlement with the crystallite size computed from XRD (13 nm).

Scanning Electron Microscope (SEM)

The morphological features of the synthesized Cellulose/Fe_3O_4/Co_3O_4 nanocomposite were studied by SEM. Figure 6 shows that the nanocomposite is spherical.

Vibrating Sample Magnetometer (VSM)

Information about magnetic properties of Cellulose/ Fe_3O_4/Co_3O_4 nanocomposite was obtained using VSM (Fig. 7). The saturation magnetization (Ms) of the catalyst is 62.39 emu g^{-1} which is not significantly lower than that of bare FMNPs. It was expected that Ms of the catalyst should be lower than Ms of FMNPs significantly due to the coating of cellulose on the magnetic nanoparticles. The reason for having this relatively high Ms is related to Co which is paramagnetic [42]. The obtained nanocomposite with a great magnetic property can be separated from the reaction medium easily by a magnet.

Catalytic Application of the Nanocatalyst

At first, to find the optimized conditions, the reaction of benzaldehyde, 2-aminobenzothiazole and β-naphthol in the presence of the Cellulose/Fe_3O_4/Co_3O_4 nanocomposite as catalyst was nominated as a model reaction in ethanol at 60 °C. Initially, we tried to find optimum amount of the catalyst in the reaction model. Thus, 3, 4, 5 and 6 mol% of Cellulose/Fe_3O_4/Co_3O_4 nanocomposite were used (Table 1). It was observed that 0.015 g of the nanocatalyst was the most effective in catalysing preparation of 1-((benzo[d] thiazol-2-ylamino)phenylmethyl)naphthalen-2-ol at 60 °C in ethanol solvent.

To consider the generality of the procedure, the reactions of various arylaldehydes under the optimized conditions were investigated and the corresponding products were obtained in high to excellent yields (Table 2). The presence of electron donating groups and electron-withdrawing groups on the arylaldehydes afforded the corresponding products in a shorter reaction time with higher yields (Table 2).

The suggested mechanism for the formation of the products is presented in Scheme 3 [43]. The aldehyde is first activated with Co_3O_4 to give (1) and then 2-aminobenzothiozole reacts with (1) to produce (2). This intermediate can be attacked by β-naphthol to afford intermediate (3), and finally the product is obtained by aromatization of β-naphthol ring of the intermediate (3).

The recycling of the Cellulose/Fe_3O_4/Co_3O_4 nanocomposite was studied using the model reaction (Experimental section). It was observed that the recovered catalyst can be used again for four runs without any loss of its activity (Figs. 8 and 9). Metal leaching was considered by ICP-AES analysis of the catalyst before and after the reaction cycles. The Co concentration was earned to be 16.48 mg l^{-1} before reaction and 16.44 mg l^{-1} after the reaction, which confirmed negligible Co leaching.

In order to show the efficiency of the present work in comparison with several reported results in the literature, some of the results for the preparation of 1-((benzo[d] thiazol-2-ylamino)phenylmethyl)naphthalen-2-ol in are summarized in Table 3. Based on the results, compared to the other methods, the Cellulose/Fe_3O_4/Co_3O_4 nanocomposite at 60 °C is one of the most efficient catalysts with respect to the reaction time and exhibits wide applicability in terms of yields.

Table 2. Three-component Synthesis of 1-((Benzo[d]thiazol-2-ylamino)(aryl)-methyl)naphthalen-2-ol Derivatives from the Reaction of Arylaldehydes, 2-Aminobenzothiazole and β-Naphthol in the Presence of Cellulose/Fe_3O_4/Co_3O_4 Nanocomposite as a Catalyst under Ethanol Solvent at 60 °C

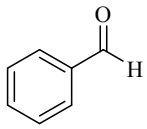
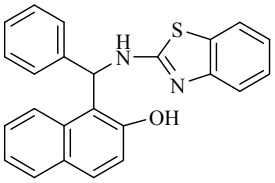
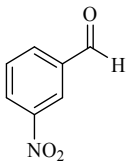
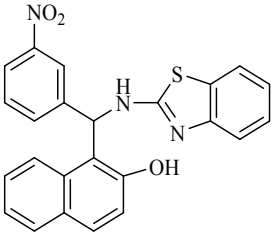
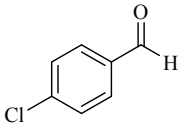
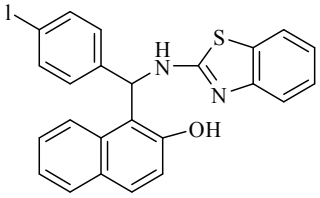
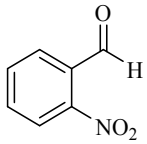
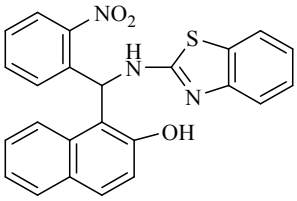
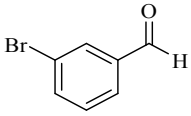
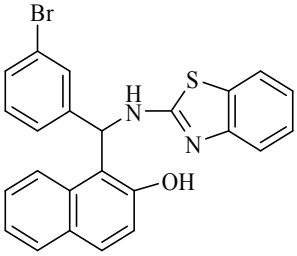
Entry	Aldehyde	Product	Time (min)	Yield (%)	Melting point (°C)	
					Found	Reported [Ref.]
1			20	92	204-205	202-203 [43]
2			20	93	200-201	198-199 [44]
3			21	94	211	209-210 [43]
4			21	90	217-218	215-216 [43]
5			22	91	202-204	205-206 [43]

Table 2. Continued

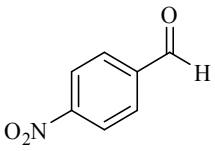
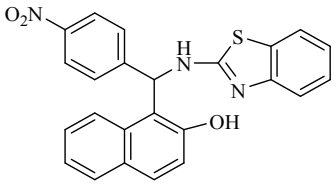
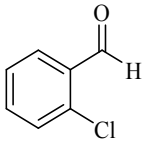
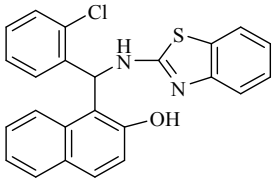
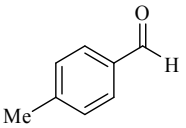
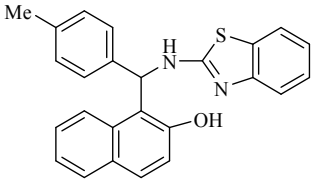
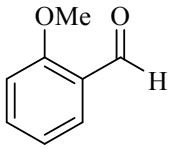
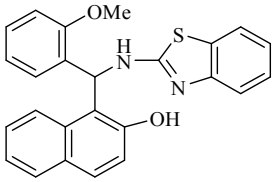
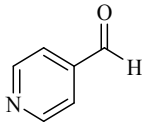
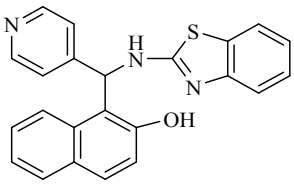
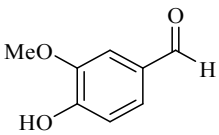
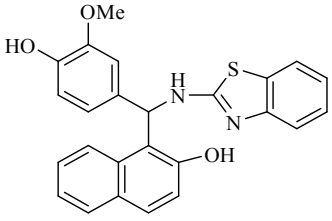
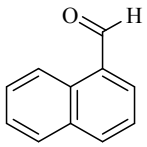
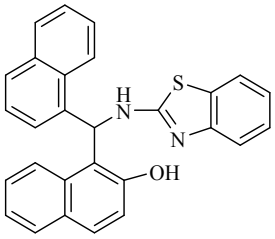
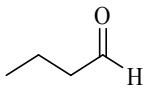
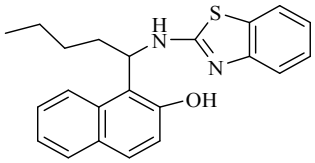
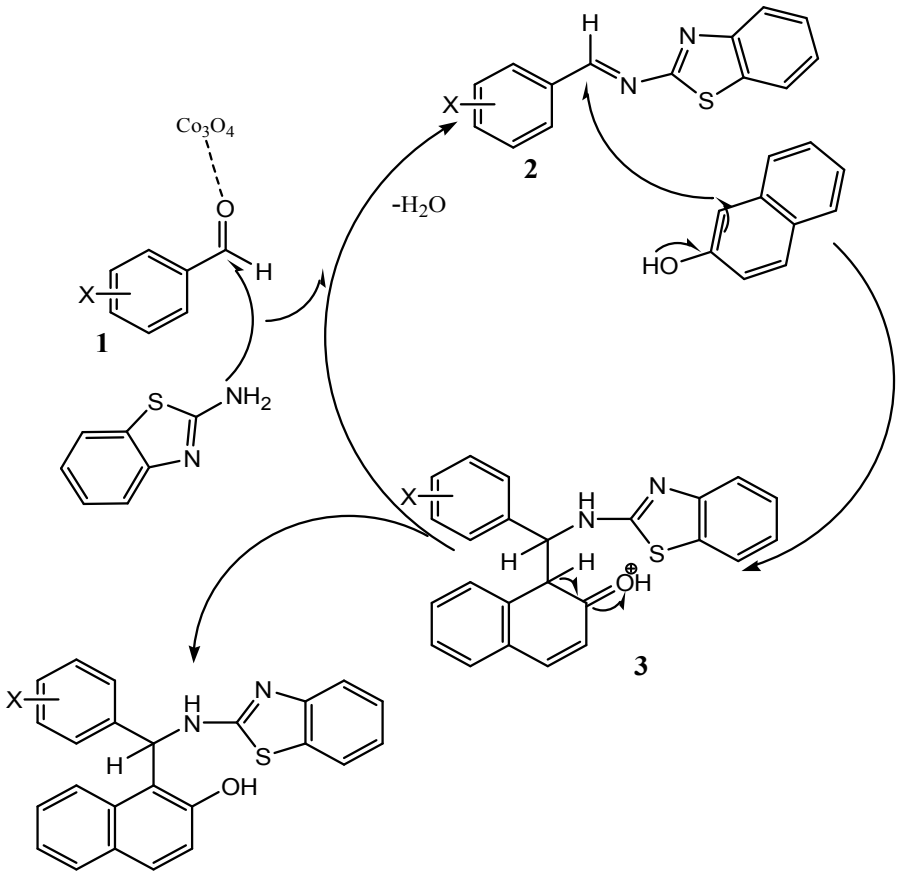
6			21	93	192-193	190-191 [44]
7			20	92	192	189-190 [43]
8			20	92	180-181	182-183 [44]
9			22	90	169-171	168-170 [45]
10			20	90	211	210-212 [45]
11			23	89	189-191	192-194 [45]

Table 2. Continued

12			22	90	200-202	203-205 [45]
13			24h	-	-	-

[a]Yields refer to the isolated products.



Scheme 3. The suggested mechanism for the formation of the products 1-((benzo[d]thiazol-2-ylamino) (aryl)-methyl) naphthalen-2-ol derivatives.

Fig. 8. Image shows that Cellulose/Fe_3O_4/Co_3O_4 nanocomposite can be separated by using magnetic field. A reaction mixture in the absence (right) and presence of a magnetic field (left).

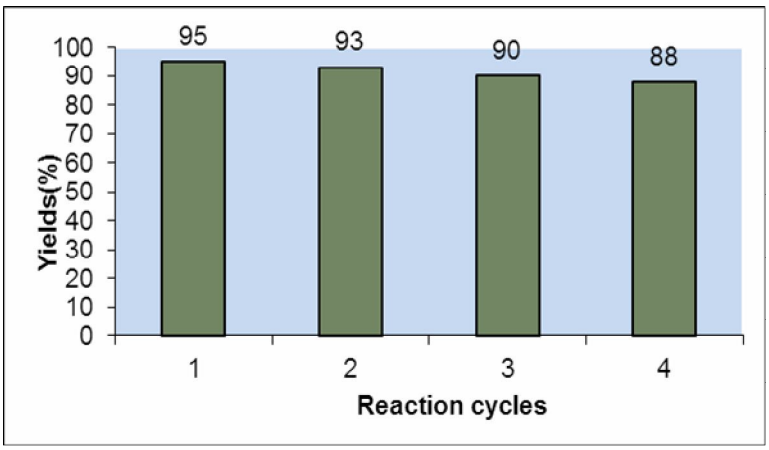


Fig. 9. Recycling of the Cellulose/Fe_3O_4/Co_3O_4 nanocomposite as catalyst.

CONCLUSIONS

We synthesized the Cellulose/Fe_3O_4/Co_3O_4 nanocomposite as a catalyst, and characterized it by XRD, EXD, FT-IR, TEM, SEM, ICP-AES and VSM techniques. Size evaluation *via* various techniques show that the size of Cellulose/Fe_3O_4/Co_3O_4 nanocomposite is around 15-30 nm. The most interesting features of this work include stability as well as the efficient catalytic activity for the synthesis of 1-((benzo[d]thiazol-2-ylamino)(aryl)-methyl) naphthalen-2-ol derivatives *via* the reaction of arylaldehydes, 2-aminobenzothiazole and β-naphthol at 60 °C in ethanol solvent. The attractive features of this synthesis are simple procedure, cleaner reaction, and use of a good reversible nanocatalyst. High yield of products, simplicity of the method, and simple purification of the products make it a useful protocol for the green synthesis.

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Table 3. Comparison the Catalytic Performance of the Cellulose/Fe_3O_4/Co_3O_4 Nanocomposite with other Catalysts in the Synthesis of 1-((Benzo[d]thiazol-2-ylamino)(aryl)methyl)naphthalen-2-ol Derivatives

Entry	Catalyst	Conditions	Time	Yield (%)[a] [Ref.]
1	[Hnhp]HSO_4 (10 mol%)	Solvent-free, 80 °C	5-10 min	92-96 [43]
2	LiCl (0.5 g, 71 mmol)	Water (5 ml), 90 °C	5-7 h	88-96 [44]
3	(Mg-Al-CO_3) (0.08 g)	Solvent-free, 70 °C	2.5-30 h	78-90 [46]
4	SLS/cetrimide/Triton X-100 (10 mol%)	Water (10 ml), r.t.	2-3 h	79-93 [47]
5	Oxalic acid	Solvent-free, 80 °C	10 min	96 [48]
6	Cellulose (0.015 g)	Ethanol, 80 °C	90 min	7No reactions (Present work)
7	Fe_3O_4 (0.015 g)	Ethanol, 60 °C	90 min	60 (Present work)
8	Cellulose/Fe_3O_4 (0.015 g)	Ethanol, 60 °C	90 min	58 (Present work)
9	Fe_3O_4/Co_3O_4 (0.015 g)	Ethanol, 60 °C	90 min	70 (Present work)
10	Cellulose/Fe_3O_4/Co_3O_4 nanocomposite (0.84 mmol%)	Ethanol, 60 °C	20-22 min	90-92 (Present work)

[a]Yields refer to isolated products.

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