RESEARCH PAPER

Synthesis of N,N´-Alkylidenebisamides in the Presence of Fe_3O_4@ Nano-Cellulose/B(III) as a Natural Based Super Paramagnetic Nanocatalyst

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ARTICLE INFO

ABSTRACT

Fe_3O_4@nano-cellulose/B(III) was synthesized as natural based super paramagnetic nano-catalyst and characterized by FT-IR, VSM, XRD, XRF, BET, FESEM, TEM, TGA and EDS(EDX) techniques. Natural base of this nano magnetic catalysis is nano-cellulose which prepared from hydrolysis of glucoside linkage of cotton cellulose. Bis-amides can be easily transformed into other functionalities and also, used for the synthesis of pharmacological materials such as peptidomimetic compounds. In this work, Fe3O4@nano-cellulose/B(III) was successfully applied to the synthesis of N,N'-alkylidenebisamides derivatives via one-pot three-component condensation reaction of various aldehydes and amides. N,N'-alkylidenebisamides have been prepared under solvent-free conditions at 70 °C. The super paramagnetic catalyst was removed from reaction mixture by an external magnet without any filtration. The structure of obtained products were investigated by FTIR (ATR), ^{1}H-NMR and ^{13}C NMR. All of the reactions proceeded in high yields and in short reaction times. This method offers several advantages including recyclability of catalyst, easy work-up, excellent yields and short reaction time.

INTRODUCTION

Bis-amides are an important class of organic compounds because of these groups can be easily transformed into other functionalities (such as gem-diaminoalkyl and aminoalkyl group) and also, used for the synthesis of pharmacological materials such as peptidomimetic compounds [1]. The first synthesis of *gem*-bis-amides was achieved via condensation reaction between an aldehyde and an amide by Noyes *et al* in 1933 [2]. Various catalysts, such as montmorillonite K10 [3], silica supported calcium chloride (SiO_2-$CaCl_2$) [4], hydroxyapatite [5], $ZnCl_2$/SiO_2 [6], acetyl chloride [7], zinc chloride [8], phosphotungstic acid,[9] nano copper ferrite ($CuFe_2O_4$) [10], silica supported polyphosphoric acid (SiO_2-PPA) [11] and silica-bonded S-sulfonic acid nanoparticles (SBSSANPs) [12] have been applied for the preparation of *N,N'*-alkylidenebisamides.

In this study, Fe_3O_4@nano-cellulose/B(III) was synthesized as natural based super paramagnetic nano-catalyst. The catalyst was characterized by FT-IR, VSM, XRD, XRF, BET, FESEM, TEM, DSC(DSX) and TGA techniques. Fe_3O_4@nano-cellulose/B(III) was successfully applied to the synthesis of *N,N'*-alkylidenebisamides derivatives *via* one-pot three-

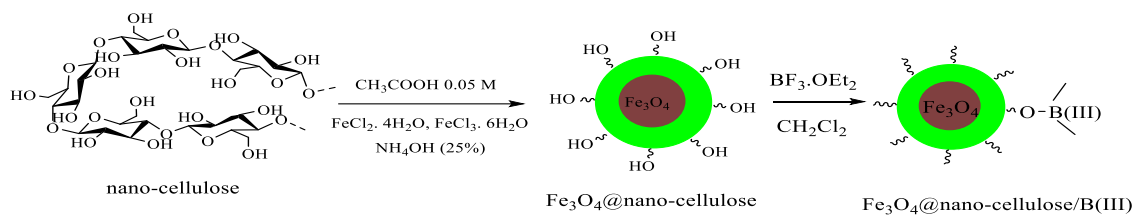* *Corresponding Author Email: fmirjalili@yazd.ac.ir*

Fig. 1. Graphical representation of Fe_3O_4@nano-cellulose/B(III) preparation

component condensation reaction of various aldehydes and amides.

MATERIAL AND METHODS

All compounds were purchased from Aldrich, Merck, and Fluka chemical companies. Nano-cellulose and Fe_3O_4@nano-cellulose were synthesized *via* our previously reported methods [13-16]. FT-IR spectra were run on a Bruker, Equinox 55 spectrometer. A Bruker (DRX-400 Avance) NMR was used to record the ^{1}H NMR and ^{13}C NMR spectra. The X-ray diffraction (XRD) pattern was obtained by a Philips Xpert MPD diffract meter equipped with a Cu Ka anode (k = 1.54 A°) in the 2θ range from 10 to 80°. XRF analysis was done with Bruker, S4 Explorer instrument. VSM measurements were performed by using a vibrating sample magnetometer (Meghnatis Daghigh Kavir Co. Kashan, Iran). Melting points were determined by a Buchi melting point B-540 B.V.CHI apparatus. Field emission scanning electron microscopy (FESEM) was obtained on a Mira 3-XMU. Transmission electron microscopy (TEM) was obtained using a Philips CM120 with a LaB6 cathode and accelerating voltage of 120 kV. Quantitative elemental information (EDS) of Fe_3O_4@nano-cellulose/B(III) was measured by an EDS instrument and Phenom pro X. Thermal gravimetric analysis (TGA) was conducted using "STA 504" instrument. Brunauer–Emmett–Teller (BET) surface area of catalyst was done with Micromeritics, Tristar II 3020 analyzer.

Preparation of Fe_3O_4@nano-cellulose/B(III)

In a beaker, 10 ml of dichloromethane was added to a Fe_3O_4@nano-cellulose (0.5 g) and stirred at room temperature. Then, 0.5 ml BF_3.OEt_2 was added dropwise to the mixture and stirred for 1 hour at room temperature. After this time, the mixture was filtered, washed with dichloromethane, dried at room temperature and Fe_3O_4@nano-cellulose/B(III) catalyst was

obtained.

General procedure for synthesis of N,N'-alkylidenebisamide derivatives

A mixture of aldehyde (1.0 mmol) and amide (2.0 mmol) was stirred at 70 °C in the presence of Fe_3O_4@nano-cellulose/B(III) (0.06 g). After completion of the reaction (monitored by TLC, *n*-hexane: EtOAc, 80:20), the reaction mixture was dissolved in ethanol and the catalyst was removed by an external magnet and reaction mixture was filtered. Then, by adding water to the filtrate, the pure products were obtained.

RESULTS AND DISCUSSION

In this work, Fe_3O_4@nano-cellulose/B(III) was prepared in several steps. At first, nano-cellulose was obtained from the acid hydrolysis of cotton. By this step, the free OH groups in nano-cellulose have been increased and could be used to synthesis of nano-cellulose supported catalysts. Fe_3O_4@nano-cellulose, were obtained simply through co-precipitation of ferric and ferrous ions with ammonium hydroxide in an aqueous solution containing nano-cellulose. At the end, preparation of Fe_3O_4@nano-cellulose/B(III) was done *via* mixing of Fe_3O_4@nano-cellulose and BF_3.OEt_2 at room temperature. The OH groups in the nano-cellulose act as nucleophile and B–O–C bonds were formed by the interaction between the BF_3 and the OH groups (Fig. 1). The morphology and structure of the prepared Fe_3O_4@nano-cellulose/B(III) composite was characterized through FT-IR, VSM, XRD, XRF, BET, FESEM, TEM, EDS and TGA techniques.

The FT-IR spectra of Fe_3O_4, nano-cellulose, Fe_3O_4@nano-cellulose and Fe_3O_4@nano-cellulose/B(III) are shown in Fig.2.

The FT-IR spectrum of Fe_3O_4@nano-cellulose/B(III) shows the stretching vibrations of the OH group at 3334 cm^{-1} and the stretching vibrations of the C–O group appears at around 1032 and 1160
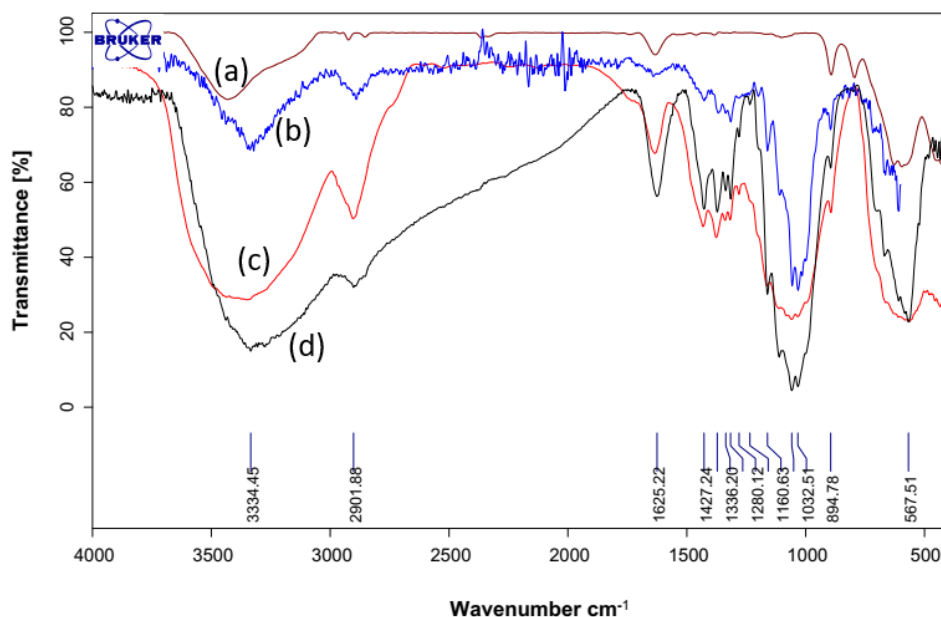
Fig. 2   FT-IR spectra of a) Fe_3O_4, b) nano-cellulose, c) Fe_3O_4@nano-cellulose and d) Fe_3O_4@nano-cellulose/B(III)

cm^{-1}. In addition to the above mentioned bands, a broad band at 567 cm^{-1} corresponds to Fe/O stretching vibrations in Fe_3O_4 Lattice.

Fig. 3 represents the FESEM and TEM images of Fe_3O_4@nano-cellulose/B(III) which was applied to investigate the particle size and surface morphology. This image indicates the Fe_3O_4@ nano-cellulose/B(III) particles are on average below 20 nm.

The X-ray diffraction (XRD) patterns of Fe_3O_4, Fe_3O_4@nano-cellulose and Fe_3O_4@nano-cellulose/ B(III) are shown in Fig. 4. According to XRD pattern of Fe_3O_4@nano-cellulose/B(III) (Fig. 4b), signal in 2θ equal to 23 confirmed the existence of cellulose in its structure. The signals in 2θ= 30, 36, 43, 54, 57 and 63 demonstrate the existence of Fe_3O_4.

The magnetic properties of Fe_3O_4 and Fe_3O_4@ nano-cellulose/B(III) were investigated at room temperature (300 K) by a vibrating sample magnetometer (VSM) studies (Fig. 5) no hysteresis loop and no remanence was detected and also the coercivity value is zero for two samples,
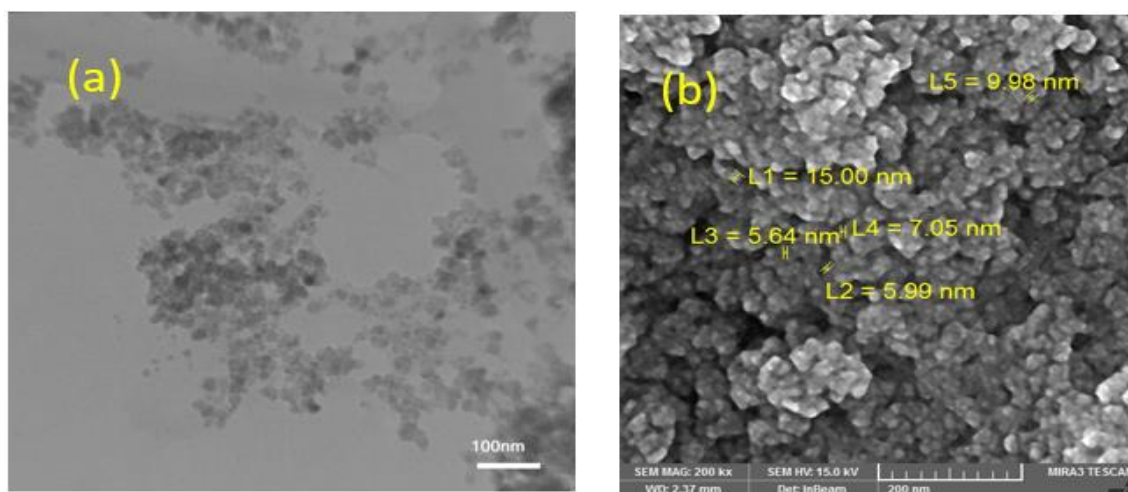

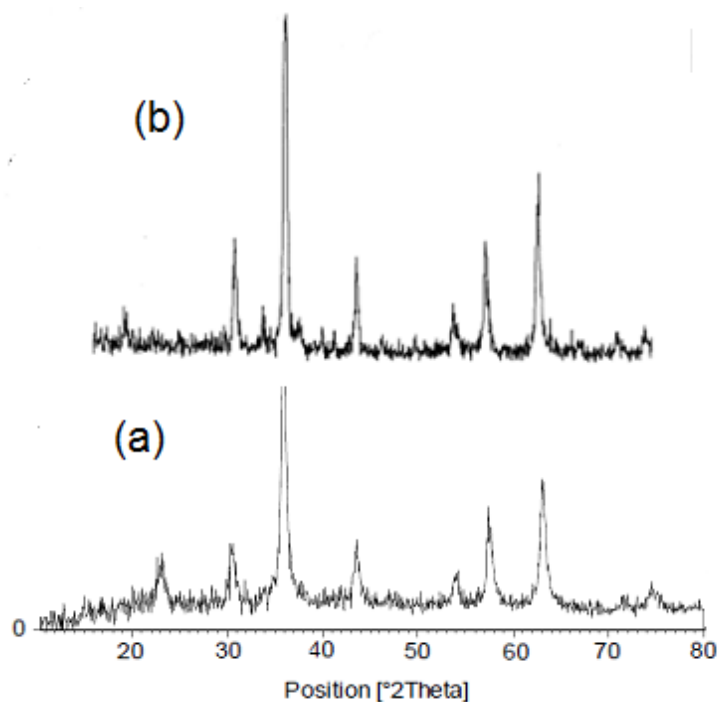Fig. 3. (a) TEM and (b) FESEM images of Fe_3O_4@nano-cellulose/B(III)

Fig. 4. The XRD patterns of: a) Fe_3O_4 , b) Fe_3O_4@nano-cellulose/B(III)

suggesting typical super-paramagnetic property at room temperature. The amounts of saturation magnetization for Fe_3O_4 and Fe_3O_4@nano-cellulose/B(III) are 50 and 23 emu g^{-1}, respectively. Despite this significant decrease, the catalyst can still be easily separated from the reaction mixture by using an external magnet.

To investigate the elemental component of Fe_3O_4@nano-cellulose/B(III), XRF analysis of catalyst was done by comparison of its kilo counts per second (KCPS) of elements in the catalyst with pure samples (NaF, H_3BO_3), (Table 1). The amounts obtained for B and F are 3.496 g (0.32 mol) and 1.112 g (0.059 mol), respectively. Thus, the B:F
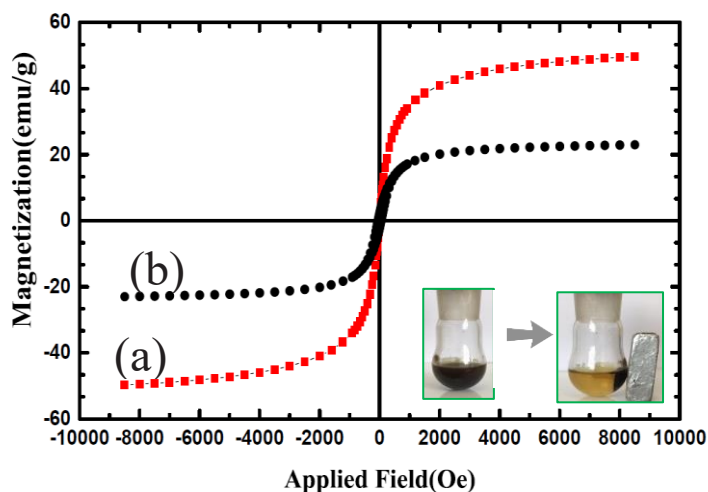
Fig. 5. Magnetization loops of a) Fe_3O_4 and b) Fe_3O_4@nano-cellulose/B(III)

Table 1. XRF analysis of catalyst and pure samples

Elemental component	Fe$_3$O$_4$@NCs/BF$_3$			H$_3$BO$_3$		NaF	
	B%	F%	KCPS	B%	KCPS	F%	KCPS
B	3.496	-	0.5	17.48	2.5	-	-
F	-	1.112	1	-	-	45.25	40.7

Fig. 6. a) BET of Fe$_3$O$_4$@nano-cellulose/B(III) and b) Nitrogen adsorption isotherms on Fe$_3$O$_4$@nano-cellulose/B(III)

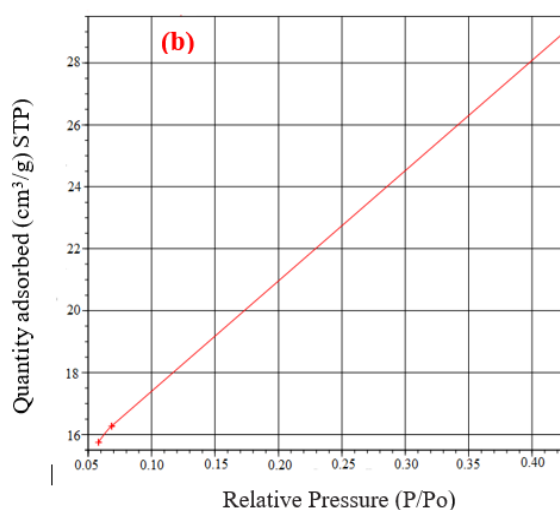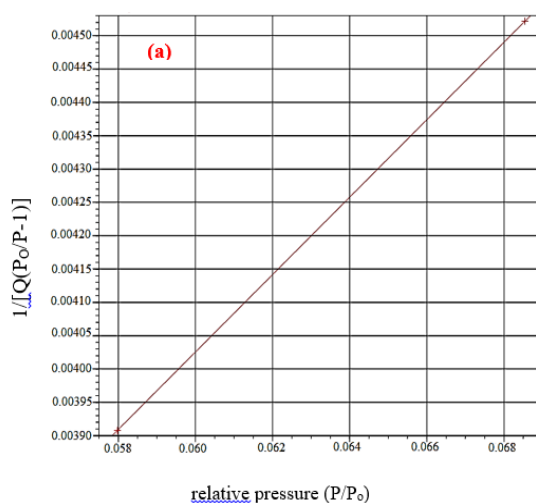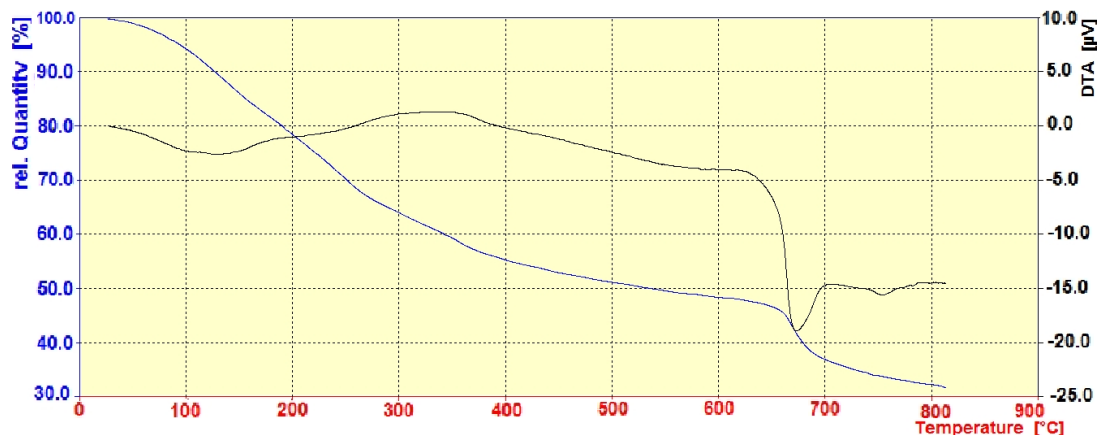Fig. 7. Thermal gravimetric analysis pattern of Fe_3O_4@nano-cellulose/B(III)

ratio, in the catalyst is approximately 5:1.

The specific surface area of catalyst was measured by Brunauer–Emmett–Teller (BET) theory (Fig. 6a). The single point surface area at P/Po = 0.0685 is 65.9890 m^2 g^{-1} and BET surface area is 74.2480 m^2 g^{-1}. The N_2 adsorption isotherm of the catalyst is depicted in Fig. 6b.

TGA-DTA analysis was performed to estimate thermal stability of the Fe_3O_4@nano-cellulose/B(III) in the temperature range of 26–814 °C

(Fig. 7). The TGA curve illustrates three stages of weight loss. The first weight loss in 93 °C (5% weight loss) is related to the removal of moisture from the catalyst. The next weight loss (52%) appears in the range of 100–670 °C and relates to the decomposition of cellulosic units in the nano-composite. Finally, the main weight loss (6%) is observed in the range of 670–700 °C.

The EDS spectrum of Fe_3O_4@nano-cellulose/ B(III) (Fig. 8), shows the presence of the elements

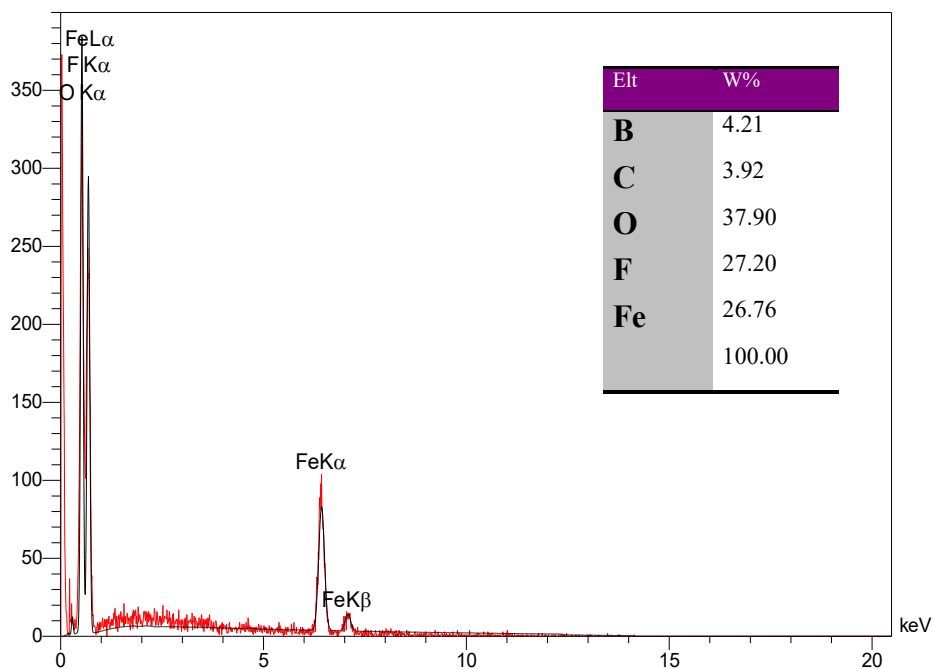| Elt	W%
B	4.21
C	3.92
O	37.90
F	27.20
Fe	26.76
	100.00

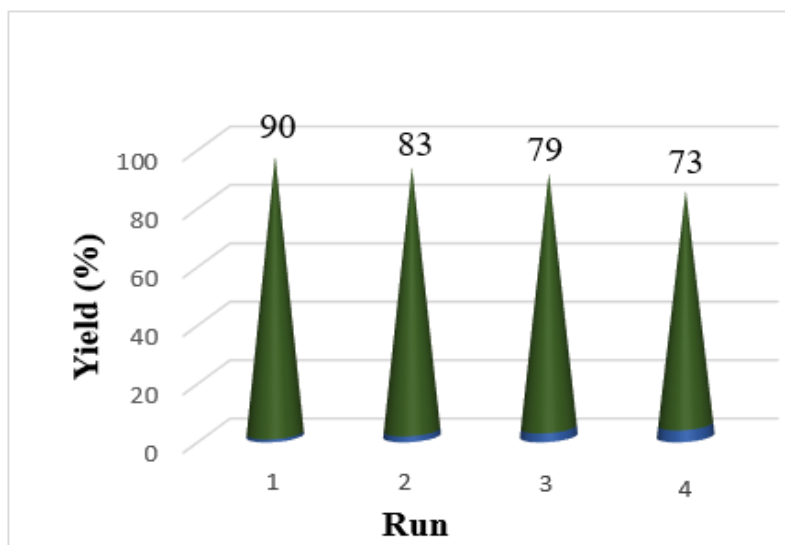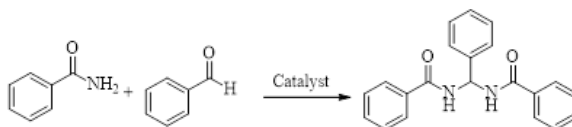Fig. 8. EDS spectrum of Fe_3O_4@nano-cellulose/B(III)

Fig. 9. Catalyst recycling experiments

Table 2. The reaction of benzamide (2.0 mmol), and benzaldehyde (1.0 mmol), in the presence of Fe_3O_4@nano-cellulose/B(III) under various conditions.


Entry	Catalyst (g)	Condition	Solvent	Time (min)	Yield (%)[b]
1	-	70 °C	-	10	46
2	Fe_3O_4@nano-cellulose (0.06)	70 °C	-	10	14
3	Fe_3O_4@nano-cellulose/B(III) (0.015)	70 °C	-	10	58
4	Fe_3O_4@nano-cellulose/B(III) (0.03)	70 °C	-	10	61
5	Fe_3O_4@nano-cellulose/B(III) (0.05)	70 °C	-	10	87
6	Fe_3O_4@nano-cellulose/B(III) (0.06)	70 °C	-	10	97
7	Fe_3O_4@nano-cellulose/B(III) (0.07)	70 °C	-	10	87
8	Fe_3O_4@nano-cellulose/B(III) (0.05)	Reflux	*n*-Hexane	24 h	19
9	Fe_3O_4@nano-cellulose/B(III) (0.05)	Reflux	Ethyl acetate	6 h	23
10	Fe_3O_4@nano-cellulose/B(III) (0.05)	Reflux	EtOH	6 h	42
11	Fe_3O_4@nano-cellulose/B(III) (0.05)	Reflux	H_2O	6 h	30
12	Fe_3O_4@nano-cellulose/B(III) (0.05)	r.t.[c]	-	18 h	41
13	Fe_3O_4@nano-cellulose/B(III) (0.05)	50 °C	-	45	52
14	Fe_3O_4@nano-cellulose/B(III) (0.05)	60 °C	-	45	55
15	Fe_3O_4@nano-cellulose/B(III) (0.06)	Microwave	-	10	51
16	Fe_3O_4@nano-cellulose/B(III) (0.06)	Mixer mill	-	10	0
17	Fe_3O_4@nano-cellulose/B(III) (0.06)	Ultrasound (r.t.[b])		10	36
18	Fe_3O_4@nano-cellulose/B(III) (0.06)	Ultrasound (70 °C)		10	95
19	Fe_3O_4@nano-cellulose/B(III) (0.06)	Ultrasound	H_2O	10	50
20	Fe_3O_4@nano-cellulose/B(III) (0.06)	Ultrasound	*n*-Hexane	10	34
21	Fe_3O_4@nano-cellulose/B(III) (0.06)	Ultrasound	PEG 400	10	40
22	Fe_3O_4@nano-cellulose/B(III) (0.06)	Ultrasound	Acetone	10	58
23	Fe_3O_4@nano-cellulose/B(III) (0.06)	Ultrasound	EtOH	10	48
24	Fe_3O_4@nano-cellulose/B(III) (0.06)	Ultrasound	Ethyl acetate	10	34
25	Fe_3O_4@nano-cellulose/B(III) (0.06)	Ultrasound	H_2O:EtOH (1:1)	10	37

[a]Benzamide (1 mmol) and benzaldehyde (1 mmol) were applied.
[b]Isolated yield
[c]Room temperature

Table 3. Synthesis of N,N'-alkylidenebisamides derivatives in the presence of Fe_3O_4@nano-cellulose/B(III)[a].
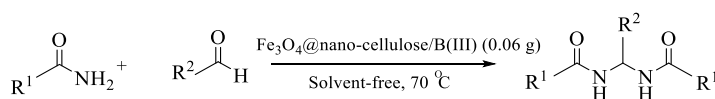
Entry	R^1	R^2	Time (min)	Yield (%)[b]	Melting point Found	Melting point reported[Ref.]
1	C_6H_5	C_6H_5	10	97	235-237	237-238[17]
2	C_6H_5	$4-NO_2-C_6H_4$	10	98	261-263	263-264[17]
3	C_6H_5	$3-NO_2-C_6H_4$	20	90	226-228	227-230[12]
4	C_6H_5	$2-OH-C_6H_4$	25	75	187-189	188-190[5]
5	C_6H_5	$CH_3-CH_2-CH_2-$	15	90	173-175	172-173[18]
6	C_6H_5	$Ph-CH_2-CH_2-$	35	85	251-252	253-254[18]
7	C_6H_5	$Ph-CH=CH-$	20	93	199-201	200-202[19]
8	OCH_3	$4-NO_2-C_6H_4$	5	92	194-196	196-198[18]
9	OCH_3	$2,4-di-Cl-C_6H_3$	10	80	247-249	248-250[18]
10	OCH_3	$3-NO_2-C_6H_4$	10	85	185-187	184-185[18]
11	CH_3	$2,4-di-Cl-C_6H_3$	30	70	265-267	262-263[7]
12	CH_3	$4-NO_2-C_6H_4$	30	90	272-274	271-273[7]
13	$CH_2=CH-$	$3-NO_2-C_6H_4$	40	89	221-223	222-223[18]
14	C_6H_5	$(CH_3)_2-CH-$	20	87	125-127	126-127[18]

[a]Reaction conditions: Amide (2.0 mmol), aldehyde (1.0 mmol), and Fe_3O_4@nano-cellulose/B(III) (0.06 g).
[b]Isolated yield

Table 4. Comparison of Fe_3O_4@nano-cellulose/B(III) catalyst with reported catalysts for the synthesis of N,N'-(phenylmethylene) dibenzamide

Entry	Catalyst	Condition	Solvent	Time (min)	Yield (%)[Ref.]
1	Boric acid(0.3 mmol)	Reflux	Toluene	16 h	92[17]
2	$ZnCl_2/SiO_2$ (0.2 g)	100	-	35	83[6]
3	$ZnCl_2$ (2 mol %)	Reflux	Toluene	7 h	91[8]
4	$NiFe_2O_4$@SiO_2-PPA (0.1 g)	Reflux	MeOH	80	86[19]
6	Phosphotungstic acid (0.3 mmol)	Reflux	Toluene	16 h	94[9]
7	Hydroxyapatit	80 °C	Acetonitrile	3 h	95[5]
8	Trityl chloride	60 °C	Ethanol	35	90[20]
9	Choline chloride/L-(+)-Tartaric acid	100 °C	-	15	97[21]
10	Fe_3O_4@nano-cellulose/B(III) [0.06 g]	70 °C	-	10	97[this work]

O, Fe, F, and B with the corresponding weight percentages (37.90, 26.76, 27.20 and 4.21 %).

Catalytic activity of Fe_3O_4@nano-cellulose/B(III)

After characterization of catalyst, The catalytic activity of Fe_3O_4@nano-cellulose/B(III) was investigated for the synthesis of *N,N'*-alkylidenebisamides derivatives. For optimization of *N,N'*-(phenylmethylene) dibenzamide the *via* reaction of benzamide (2.0 mmol) and benzaldehyde (1.0 mmol) as the model reaction. To optimize the reaction conditions, the effect of several parameters such as catalyst amount, solvent, temperature reaction time and laboratory conditions was studied (Table 2). According to

Table 1, the best condition for the reaction is a solvent-free condition using 0.06 g of Fe_3O_4@nano-cellulose/B(III) as an efficient catalyst at 70 °C (Table 2, entry 6).

Based on optimal reaction conditions, *N,N'*-alkylidenebisamides derivatives were synthesized using the three-component condensation of various aldehydes (1.0 mmol) and various amides (2.0 mmol) (Table 3). As the results in Table 3 show, all these reactions occurred smoothly affording high yields. The structures of these products were characterized by melting point, FT-IR, ^{1}H- and ^{13}C-NMR spectroscopy. To indicate the capability of the present method and efficiency of our catalyst in comparison with the reported methods
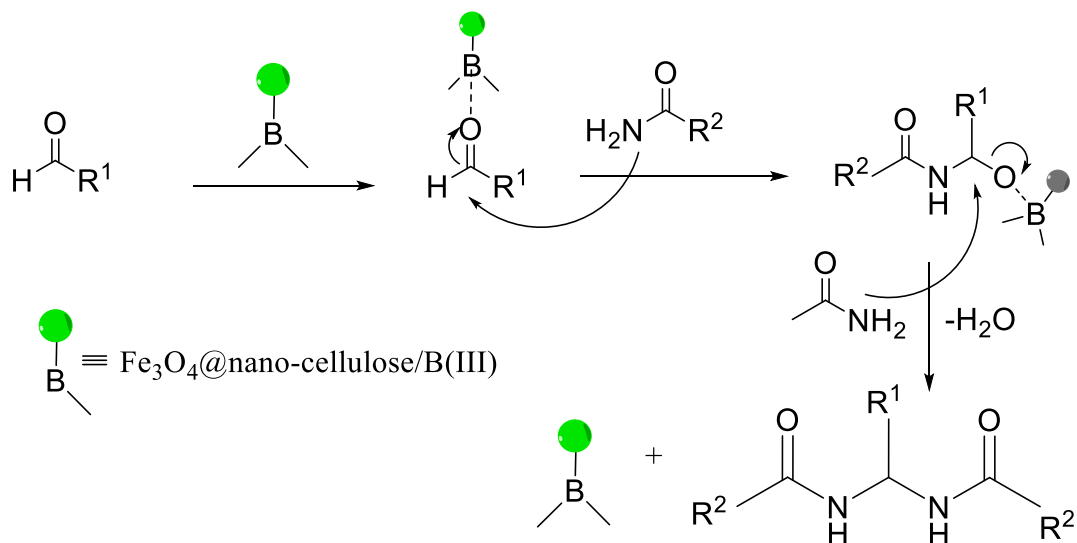
Fig. 10. Proposed mechanism for the synthesis of N,N'-alkylidenebisamides derivatives

to prepare *N,N'*-(phenylmethylene) dibenzamide, we compared our results with other methods in the literature for the model reaction (Table 4). we have observed good and high yields of products in very mild and green conditions using $Fe_3O_4@$ nano-cellulose/B(III). The reusability of the catalyst was also investigated on the model reaction. The magnetic nature of the catalyst allowed its facile recovery by simple separation by an external magnet, then washed with ethanol, dried at room temperature to be used in the subsequent run of the reaction with fresh reactants under similar conditions. It was observed that the recovered biocatalyst could be used at least four times with marginal decrease in their catalytic activity (Fig. 9). The proposed mechanism for the synthesis of *N,N'*-alkylidenebisamides derivatives in the presence of $Fe_3O_4@$nano-cellulose/B(III) is shown in Fig. 10.

CONCLUSION

In summary, we have demonstrated the preparation and characterization of $Fe_3O_4@$nano-cellulose/B(III) as highly efficient, magnetically recyclable, cheap and bio-based catalysts. The catalytic activity of the prepared catalyst was investigated in the synthesis of *N,N'*-alkylidenebisamides derivatives reaction *via* the condensation reaction of various aldehydes and various amides under mild reaction conditions.

This method offers several advantages including easy work-up, excellent yields, short reaction time, reusability of the catalyst and environmental friendliness.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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