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Original Research Article

Bifunctional Polyethylene Glycol/ethylenediamine Nanomagnetic Phase-Transfer Catalyst: Preparation, Characterization, and Application in Knoevenagel Condensation

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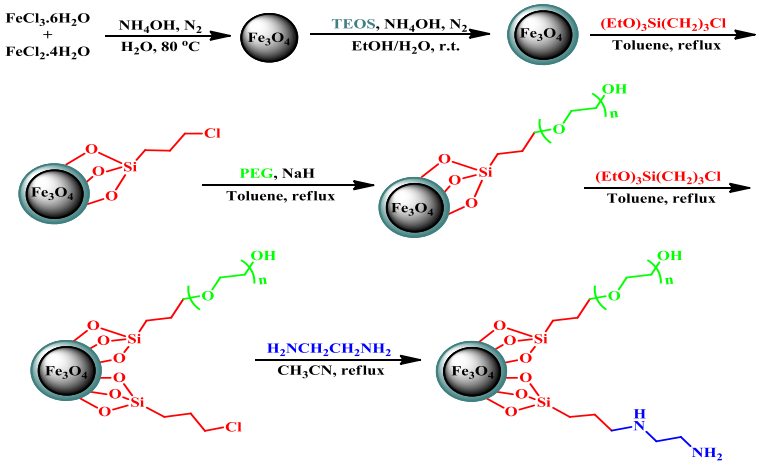
ABSTRACT

In the present study, an efficient and recyclable heterogeneous phase-transfer catalyst was prepared through the functionalization of magnetic nanoparticles with ethylenediamine and polyethylene glycol. After being characterized by various physico-chemical techniques, the bifunctional magnetic nanocomposite was used as a heterogeneous phase-transfer catalyst in the Knoevenagel condensation of aryl aldehydes with active methylene compounds under mild and green conditions. This procedure has several advantages such as high yield of products, short time of reaction, easy workup, mild reaction conditions, low amount of catalyst, and catalyst recoverability.

GRAPHICAL ABSTRACT



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Introduction

The Knoevenagel condensation, which is one of the greatest valuable and extensively working methods for carbon-carbon bond formation, has often been used for the production of biologically important heterocyclic compounds and fine chemicals [1, 2]. In the Knoevenagel condensation reaction, an aldehyde reacted with an active methylene compound, including dimedone, malononitrile, ethyl acetoacetate, etc. [3-5]. This reaction is generally catalyzed by homogeneous or heterogeneous catalysts containing weak bases such as Nano CeO_2 [6], ethylenediamine [7], and alkaline earth metals [8], Nano CeO_2/Al_2O_3 [9], $LaCl_3.7H_2O$ [10], $NbCl_5$ [11], $KF–Al_2O_3$ [12], and L-proline-Cu/TCT@NH_2@Fe_3O_4 [13] have been used as catalysts in this reaction.

Homogeneous catalysts show high efficiency in chemical reactions [14-17], but they commonly have low chemical and thermal stability. In addition, their recovery is mostly difficult and expensive, and causes additional waste [18]. These issues can be solved using environmentally friendly heterogeneous catalysts instead of homogeneous ones [19].

The simplest way to achieve this goal is to support homogeneous catalysts on insoluble materials like silica, graphene, polymers, and magnetic nanoparticles (MNPs) [20-23]. Today, MNPs are widely used due to their special features including large surface area, biocompatibility, low toxicity, and retrievability [24-27]. Magnetic separation makes it much easier to recover the catalyst from the reaction mixture by magnet than by centrifugation and filtration [28].

In the present work, continuing our research on the preparation of nanomagnetic-supported organocatalysts [29-34], we synthesized a recoverable heterogeneous phase-transfer catalyst, and after characterization, its catalytic performance was investigated in Knoevenagel condensation. The results showed that this catalyst efficiently catalyzes the reaction between active methylene compounds and aryl aldehydes.

Materials and Methods

Chemicals

The materials were procured from Sigma-Aldrich and Merck companies. The magnetic property was determined at room temperature on VSM (Meghnatis Daghigh Kavir Co Iran). TEM measurements were carried out on a Zeiss EM10C microscope at 100 kV. SEM and EDS analyses were carried out using a Zeiss-Sigma VP instrument for the morphology and elemental analysis of the synthesized nanocatalyst. XRD analysis was carried out using PANalytical X'Pert Pro X-ray diffractometer. FT-IR spectra were recorded using a Shimadzu IRPrestige-21 spectrometer and samples were analyzed as a KBr disk.

Preparation of the catalyst

PEG-300 (3 mmol) and sodium hydride (3 mmol) were poured in 20 mL toluene at 0 °C under an inert atmosphere. After stirring the mixture for one hour at 60 °C, 1.0 g of Fe_3O_4@SiO_2@$(CH_2)_3$-Cl [35] suspended in 50 mL toluene was added and stirred under reflux conditions for 12 h. The resulting MNPs were dried at 60 °C after washing by ethanol and acetone. In the next step, 1.0 g of Fe_3O_4@SiO_2-PEG was dispersed by ultrasonic irradiation in 100 mL toluene for 20 min. After adding 2 mL of (3-chloropropyl) triethoxysilane and refluxing the mixture for 12 h, the obtained Fe_3O_4@SiO_2-PEG/Cl was washed with EtOH and dried at 60ºC. Finally, to the ultrasonicated suspension of Fe_3O_4@SiO_2-PEG/Cl (1.0 g) in 100 mL acetonitrile, 5 mL ethylenediamine was added and the mixture was stirred under reflux conditions for 12 h. After cooling, the resultant MNPs were separated by a magnet and dried at 60ºC after washing by ethanol [36].

Knoevenagel condensation catalyzed by Fe_3O_4@SiO_2-PEG/en

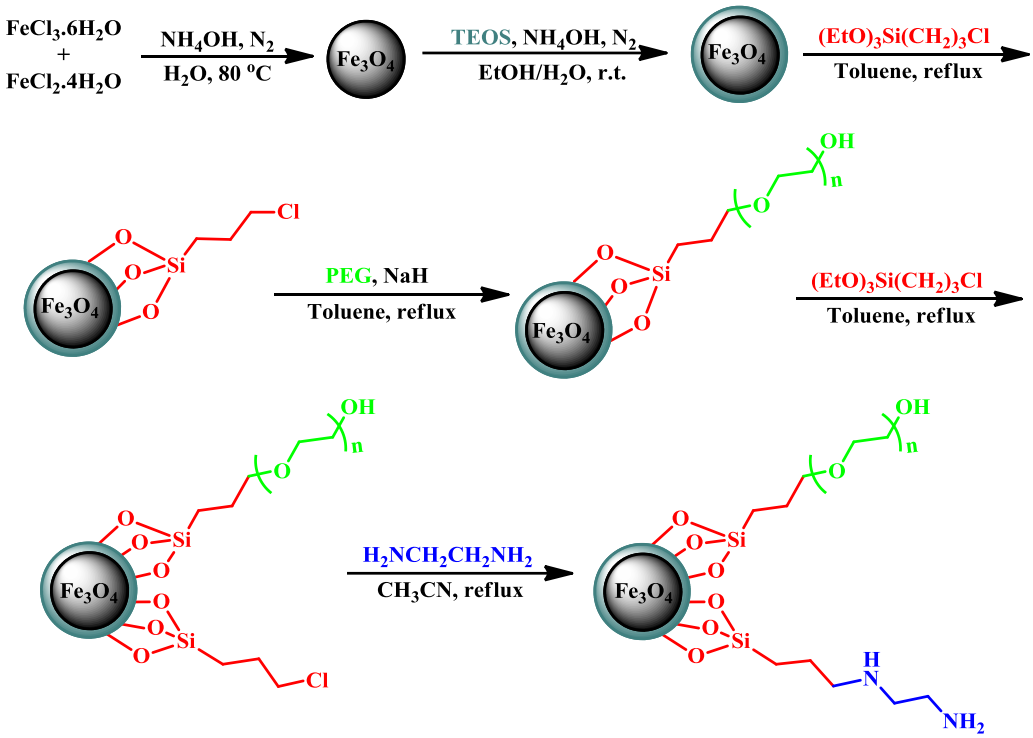
The Fe_3O_4@SiO_2-PEG/en catalyst (0.01 g) was added to a mixture of an aldehyde (1 mmol) and an active methylene compound (1 mmol) in 3 mL water. The mixture was stirred at room temperature until TLC indicated the end of

reaction. After completion of the reaction, the catalyst was easily removed with an external magnet. The residue suspension was filtered and recrystallized from EtOH to give a pure compound.

Results and Discussion

Scheme 1 represents the concise route for preparing the nanomagnetic-supported bifunctional polyethylene glycol/ethylenediamine phase-transfer catalyst, Fe_3O_4@SiO_2-PEG/en.

As previously reported,[34] various physico-chemical techniques such as FT-IR, SEM, EDS, TEM, XRD, and VSM were utilized to characterize the Fe_3O_4@SiO_2-PEG/en nanocatalyst (Figures 1, 2, 3, 4 and 5).



Scheme 1: Synthesis of magnetic nanoparticles coated with polyethylene glycol/ethylene diamine

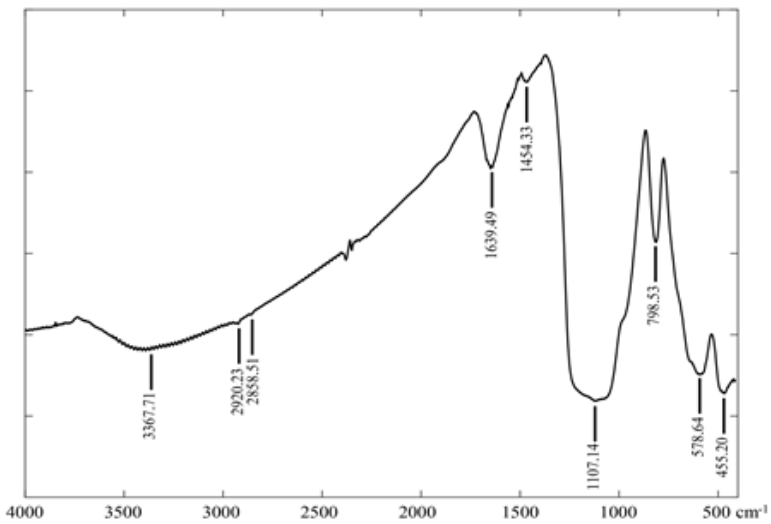


Figure 1: FT-IR spectrum of the catalyst

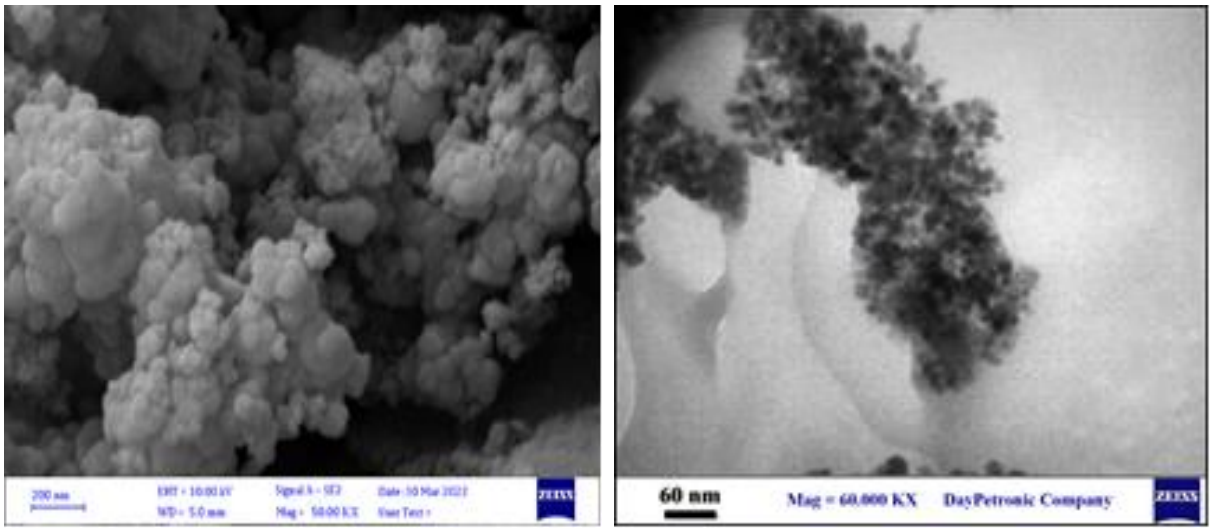


Figure 2: SEM (left) and TEM (right) images of the catalyst

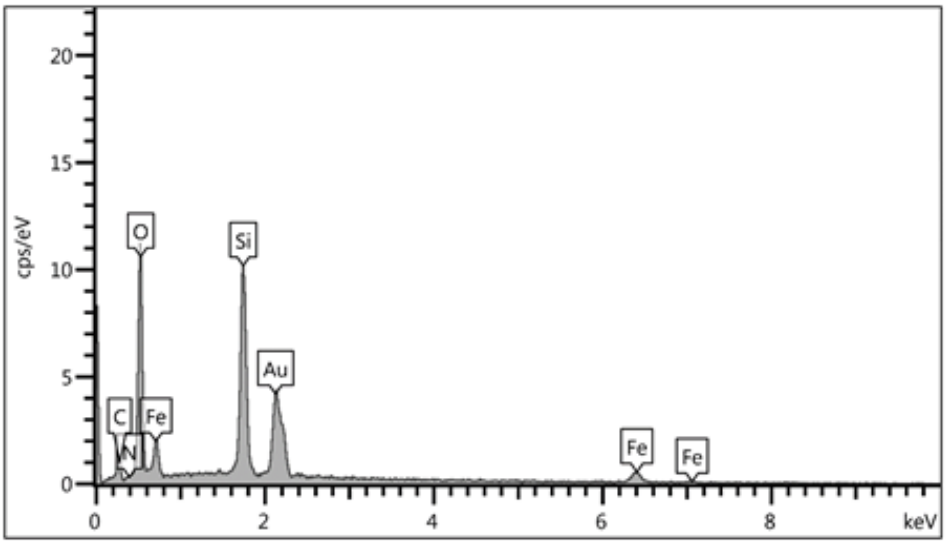


Figure 3: EDS spectrum of the catalyst

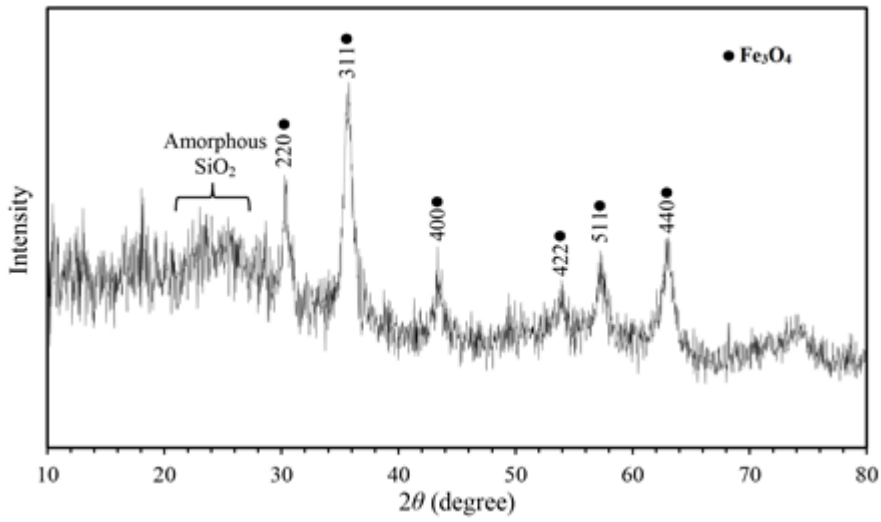


Figure 4: XRD pattern of the catalyst

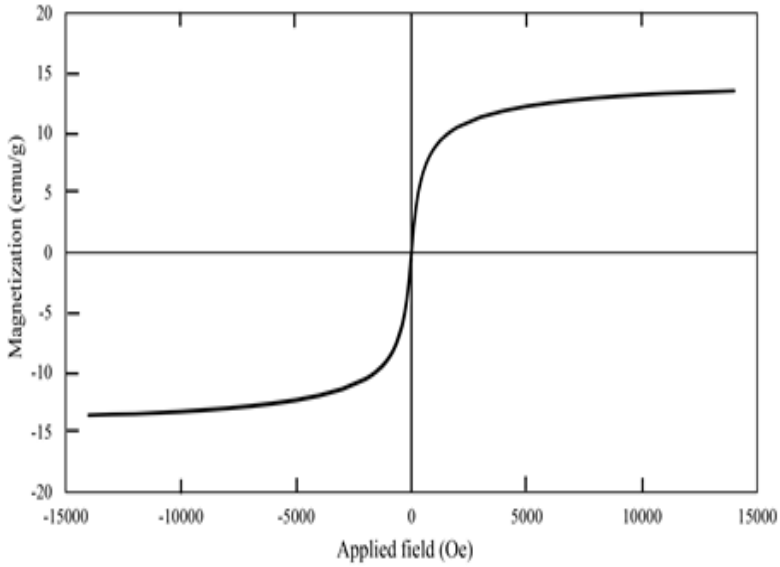


Figure 5: Magnetization vs. applied magnetic field for the catalyst

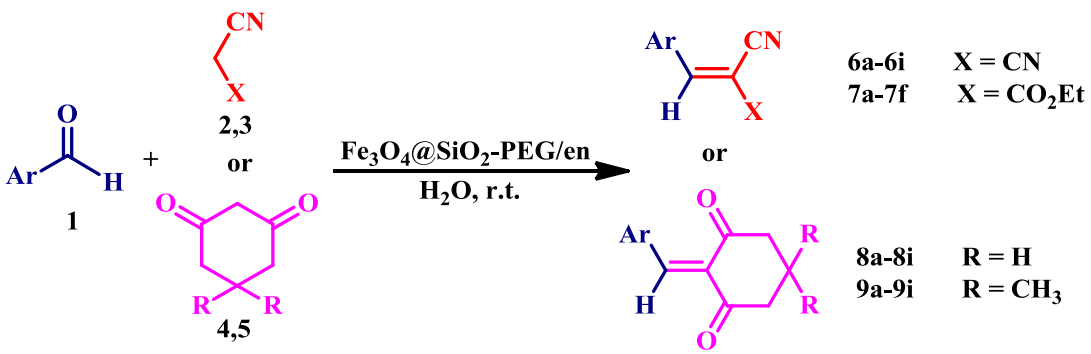
The catalytic performance of the catalyst was surveyed in the reaction of active methylene compounds with aryl aldehydes. The most acceptable result was obtained when 0.01 g of the nanomagnetic PTC was used (refer to Table 1, entry 1 for more information about the catalyst amount optimization).

After optimization, the reaction of various aryl aldehydes (1) with active methylene compounds, including malononitrile (2), ethyl cyanoacetate (3), 1,3-cyclohexanedione (4), and dimedone (5) achieved product **6a-i** (2-benzylidene malononitrile, 2-(2-Chlorobenzylidene) malononitrile, 2-(3-Chlorobenzylidene) malononitrile, 2-(4-Chlorobenzylidene) malononitrile, 2-(4-hydroxybenzylidene) malononitrile, 2-(3-nitrobenzylidene) malononitrile, 2-(4-metylebenzylidene) malononitrile, 2-(4-methoxyebenzylidene) malononitrile, 2-(2-(furan-2-yl) benzylidene) malononitrile)) product **7a-f** ((E)-Ethyl 2-cyano-3-phenylacrylate, (E)-Ethyl 3-(3-chlorophenyl)-2-cyanoacrylate, (E)-Ethyl 3-(4-chlorophenyl)-2-cyanoacrylate, (E)-Ethyl 3-(4-hydroxyphenyl)-2-cyanoacrylate, (E)-Ethyl 3-(3-nitrophenyl)-2-cyanoacrylate, (E)-Ethyl 2-cyano-3-(2-(furan-2-yl) phenyl) acrylate) product 8a-i (2-benzylidenecyclohexane-1, 3-Dione, 2-(2-chlorobenzylidene) cyclohexane-1, 3-Dione, 2-(3-chlorobenzylidene) cyclohexane-1, 3-Dione, 2-(4-chlorobenzylidene) cyclohexane-1, 3-Dione, 2-(4-hydroxybenzylidene) cyclohexane-1, 3-Dione, 2-(3-nitrobenzylidene) cyclohexane-1, 3-Dione, 2-(4-methylbenzylidene) cyclohexane-1, 3-Dione, 2-(4-methoxybenzylidene) cyclohexane-1, 3-Dione, 2-((furan-2-yl) methylene) cyclohexane-1, 3-Dione) and product **9a-i** (2-benzylidene-5, 5-dimethylenecyclohexane-1, 3-Dione, 2-(2-chlorobenzylidene)-5, 5-dimethylenecyclohexane-1, 3-Dione, 2-(3-chlorobenzylidene)-5, 5-dimethylenecyclohexane-1, 3-Dione, 2-(4-chlorobenzylidene)-5, 5-dimethylenecyclohexane-1, 3-Dione, 2-(4-hydroxybenzylidene)-5, 5-dimethylenecyclohexane-1, 3-Dione, 2-(3-nitrobenzylidene)-5, 5-dimethylenecyclohexane-1, 3-Dione, 2-(4-methylebenzylidene)-5, 5-dimethylenecyclohexane-1, 3-Dione, 2-(4-methoxybenzylidene)-5, 5-dimethylenecyclohexane-1, 3-Dione, 2-((furan-2-yl) methylene)-5, 5-dimethylenecyclohexane-1, 3-Dione) was done under optimal conditions (Scheme 2 and Table 1). Aromatic aldehydes with electron-withdrawing and electron–donating groups have led to the related alkenes in high yield (Table 1).

Table 1: Knoevenagel condensation of aromatic aldehydes catalyzed by Fe_3O_4@SiO_2-PEG/en in water

Entry	Aldehyde	Product	Time (h)	Yield (%)[a]
1	PhCHO	6a	0.25	92, trace[b], 40[c], 92[d]
2	2-ClPhCHO	6b	0.33	93
3	3-ClPhCHO	6c	0.5	91
4	4-ClPhCHO	6d	0.33	88
5	4-HOPhCHO	6e	0.5	87
6	3-NO_2PhCHO	6f	0.5	90
7	4-MePhCHO	8g	0.25	91
8	4-MeOPhCHO	6h	0.25	93
9	2-Furaldehyde	6i	0.75	87
10	PhCHO	7a	0.5	92
11	2-ClPhCHO	7b	0.5	91
12	4-ClPhCHO	7c	0.5	90
13	4-HOPhCHO	7d	0.75	86
14	3-NO_2PhCHO	7e	1	88
15	2-Furaldehyde	7f	1	87
16	PhCHO	8a	2	91
17	2-ClPhCHO	8b	3	90
18	3-ClPhCHO	8c	3	91
19	4-ClPhCHO	8d	2.5	89
20	4-HOPhCHO	8e	3	86
21	3-NO_2PhCHO	8f	3	91
22	4-MePhCHO	8g	3	93
23	4-MeOPhCHO	8h	2	92
24	2-Furaldehyde	8i	2.5	89
25	PhCHO	9a	2.5	94
26	2-ClPhCHO	9b	3.5	91
27	3-ClPhCHO	9c	3	93
28	4-ClPhCHO	9d	2.5	92
29	4-HOPhCHO	9e	3.5	87
30	3-NO_2PhCHO	9f	3	89
31	4-MePhCHO	9g	2.5	93
32	4-MeOPhCHO	9h	2	90
33	2-Furaldehyde	9i	3	88

[a] Isolated yield, [b] without catalyst, [c] 0.005 g of the catalyst, and [d] 0.02 g of the catalyst.



Scheme 2: The reaction of various aryl aldehydes with active methylene compounds

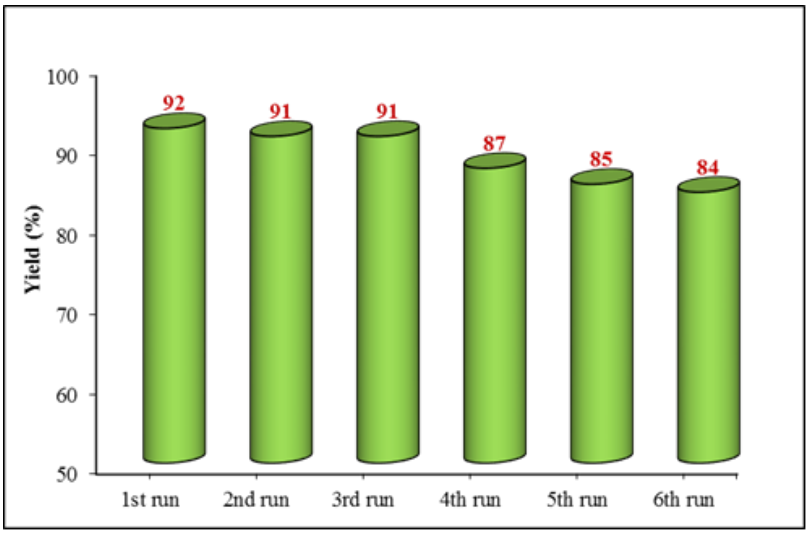


Figure 6: Reusability of Fe_3O_4@SiO_2-PEG/en

The reusability of Fe_3O_4@SiO_2-PEG/en in the Knoevenagel condensation between benzaldehyde and malononitrile in aqueous media at room temperature is depicted in Figure 6. After separation by a magnet, the catalyst was used for the next run. The catalyst can be reused six times with no considerable loss of catalytic activity.

Conclusion

In this work, to produce an efficient bifunctional phase-transfer catalyst, ethylenediamine, and polyethylene glycol were anchored on the MNPs surface. After characterization, the magnetic nanocomposite was utilized in Knoevenagel condensation at room temperature under aqueous media. The high yield of products, short reaction time, mild reaction conditions, and reusability of the catalyst have made the present method a green and effective alternate to the existing methods in the literature.

Disclosure Statement

No potential conflict of interest was reported by the authors.

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Authors' Contributions

All authors contributed to data analysis, drafting, and revising of the paper and agreed to be responsible for all the aspects of this work.

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