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Research Article

Polyethylene glycol stabilized on Fe_3O_4 nanoparticles as a magnetic nanocatalyst in the Hantzsch synthesis

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ABSTRACT

In the present project, Fe_3O_4 nanoparticles were prepared from the co-precipitation reaction of Fe^{II} , Fe^{III} in ammonia medium. The obtained nanoparticles were used as a substrate to stabilize polyethylene glycol. To stabilize PEG on Fe_3O_4 surface, Fe_3O_4 hydroxy groups and PEG hydroxy group reacted in the presence of HCL. The resulting nanocomposite was structured by FT-IR spectroscopy and its morphology was examined by SEM. Nanocomposite was used as an effective catalyst for four-component compaction between ammonium acetate, ethyl acetate, dimidone and aromatic aldehyde under solvent-free conditions. Polyhydroquinoline products were prepared in low times and with high efficiencies.

Keywords: Fe_3O_4 nanoparticles, polyethylene glycol, Polyhydroquinoline, solvent-free, Acidic catalyst.

1. Introduction

The widespread use of catalysts in the present century in industry has led researchers to design high-catalytic and recyclable catalysts. This has been achieved by using nanotechnology in the production of catalytic particles in nanometer dimensions [1-4]. The unique properties of nanoparticles can be attributed to the very high active surface and the many active sites in such particles. In MCR technique), during simple and one-step experimental operations, more than two low molecular weight structural blocks are combined simultaneously and two or more bonds are formed[5-8]. And so useful and valuable molecules and complex structures are synthesized very quickly, efficiently, effectively and in a short time without any intermediate separation. While this achievement also allows us to avoid adding any additional reagents, we minimize the amount of waste and waste of products and products[9-13]. Single-step multicomponent reactions are used to generate important structural frameworks due to their acceleration, unparalleled structural diversity, efficiency, rapid availability of small, multifunctional organic molecules with multiple functional groups[14-19]. So we can express some characteristics of multi-component reaction, fast, efficient, effective with low time and low costs. Due to the many benefits of dihydropyridine derivatives after Hantzsch, many methods have been developed to produce these derivatives, all of which have been based on the Hantzsch reaction [20-22]. The classical method of making these derivatives involves the reflux of materials in the presence of an acidic catalyst in an organic solvent. Although these methods are efficient, they are not without defects. is considered [23-25]. Therefore, it seems necessary to provide a suitable solution for the preparation of these compounds. Several more efficient and alternative methods have been introduced due to the biological importance of PHQ derivatives for the synthesis of various drug sources [26-27]. Several catalysts have been reported for the synthesis of these compounds such as: cyanuric chloride, ionic liquids [28,29], $Yb(OTf)_3$ [30], silica gel/$NaHSO_4$ [31], PSA [32], L-proline [33], $Sc(OTf)_3$ [34], p-TSA [35], ZnO [36], and TMSCl [37].

On the other Polyethylene glycols are dense polymers of ethylene oxide and water with the general formula $(CH_2CH_2O)_n$ and the chemical formula $(C_{2n+2}H_{4n+6}O_{n+2})$. Which is the most important commercial type of polyethers [38-39] Although polyethylene glycols with different molecular weights have different uses, their physical properties are different at different molecular weights while their chemical properties are almost the same [40-41]. Polyethylene glycol (PEG) is insoluble in water, methanol, gasoline, dichloromethane and insoluble in hexane and diethylene ether [42]. It is non-toxic, odorless, neutral, lubricating and non-inflammatory and is used in many pharmaceutical products as a solvent, dispersant, ointment and suppository base, carrier and absorbent in a variety of tablets. . In the present project, an attempt was made to stabilize polyethylene glycol as an environmentally friendly catalyst on the substrate of Fe_3O_4 nanoparticles [43-45]. Due to the reaction conditions, its surface has hydroxy agents that can react with the end hydroxy agents of polyethylene glycol and attach to it under certain conditions [46-48].

2. Experimental

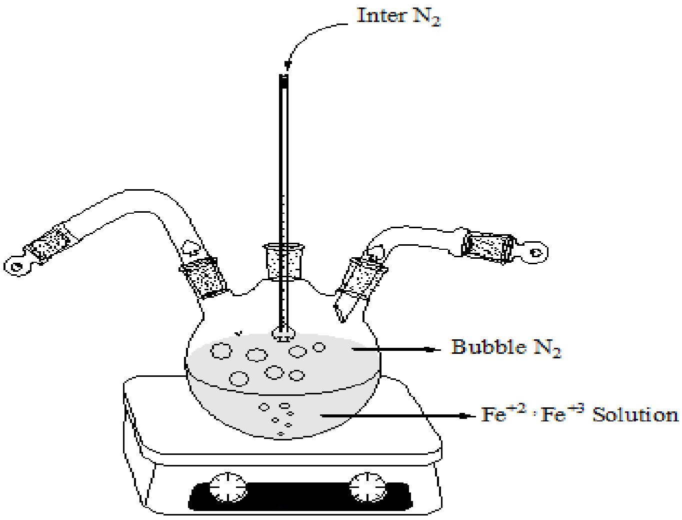
2.1. General

Chemicals were purchased from Chemical Companies with at least for synthesis grade and were used without further purification. All yields refer to isolated products. Products were characterized by comparison of their physical data such as IR, ^{1}H NMR and ^{13}C NMR spectra with authentic samples. By using TMS as internal standard, NMR spectra were recorded in $CDCl_3$ on a Bruker Advance DPX 250 MHz spectrometer. Determination of the products' purity in the course of the reaction were monitored by TLC on silica gel polygram SILG/UV 254 plates. IR spectra were recorded on a BOMEM MB-Series 1998 FT-IR spectrophotometer as KBr disks. The melting points were recorded in open capillary tubes and were uncorrected. The SEM images were recorded by TESCAN MIRA III FESEM.

2.2. Preparation of polyethylene glycol stabilized on Fe_3O_4 magnetic nanoparticles as nanocomposites at Hantzsch density

2.2.1. Method of preparation of magnetic Fe_3O_4 nanoparticles

The amount of 32 mmol of $FeCl_2.4H_2O$ salt (6.4 g) and 56 mmol of $6H_2O$ salt. $FeCl_3$ was added to a 1000 ml beaker and the resulting mixture was dissolved in 640 ml of deionized water. The yellow-orange solution was immediately transferred to a three-mouth balloon, one of which was a return distillation refrigerant and the other was a nitrogen gas inlet and a nitrogen gas outlet. In order to remove oxygen gas from the solution, nitrogen gas is injected into the solution for about 2-3 minutes. For this purpose, a clean pipette or paste pipette is attached to the end of the nitrogen capsule hose. After adjusting the exhaust gas velocity, the pipette is rotated into solution to remove oxygen from the solution (Scheme 1). The resulting solution is mixed at 80 °C with a magnetic stirrer at a uniform speed under nitrogen gas for 1 hour. To the resulting solution is added 80 ml of 25% ammonia solution. As soon as ammonia enters, a black solution color and fine Fe_3O_4 deposits appear in it. The mixture is mixed under the same conditions for 1 hour. The contents of the balloon are then transferred to a human. After the specified time, the system is allowed to cool to room temperature. The resulting precipitates are washed with distilled water (5 × 50 ml). For this purpose, a strong magnet is carefully glued to the bottom of the container and given time until the magnetic nanoparticles settle completely and the surface solution becomes clear. The supernatant is then overflowed while the magnetic nanoparticles are held in place by a magnet on the bottom of the container. In case of lack of strong magnet, nanoparticles should be made using filter paper. After 5 washing steps, the obtained black sediments are placed at room temperature under a dry vacuum and the sediments are placed in an oven at 60-70 °C for 12 hours.



Scheme 1. Schematic of nitrogen injection

2.2.2. Binding of polyethylene glycol to the surface of Fe_3O_4 nanoparticles

2 g of Fe_3O_4 magnetic nanoparticles are placed in 15-20 ml of benzene solvent and subjected to ultrasonic waves for 10 minutes. To the resulting mixture was added 2 ml of polyethylene glycol with a molecular weight of 400 and a drop of concentrated hydrochloric acid and the mixture was stirred under reflux conditions for 24 hours. After 24 hours, the mixture is smooth and after washing with ethanol and then with distilled water for 2 hours, it is dried in an oven at 50°C.

2.3. General procedure for synthesis of polyhydroquinoline derivatives catalyzed by Fe_3O_4-PEG nanoparticles under solvent free conditions

In a test tube to 1 mmol of aromatic aldehydes, 1 mmol of ethyl acetate (0.130 g), 1.5 mmol of ammonium acetate (0.115 g), 1 mmol of dimidone (0.140 g), 0.01 g of *Fe_3O_4-PEG* catalyst, added and the resulting mixture was placed in solvent-free conditions in an oil bath at 80°C.

Progression of the reaction was followed by TLC (normal hexane / ethyl acetate 5: 1). After the reaction, add a few milliliters of hot ethanol to the reaction mixture and, if there is a strong magnet, hold it at the bottom of the test tube with the help of a catalyst magnet and transfer the supernatant to a balloon. The catalyst was then used with ethanol and washing water as a recycled catalyst. The product was then crystallized with ethanol and water with a yield of 80 to 99% . The general results are given in Table (2-3).

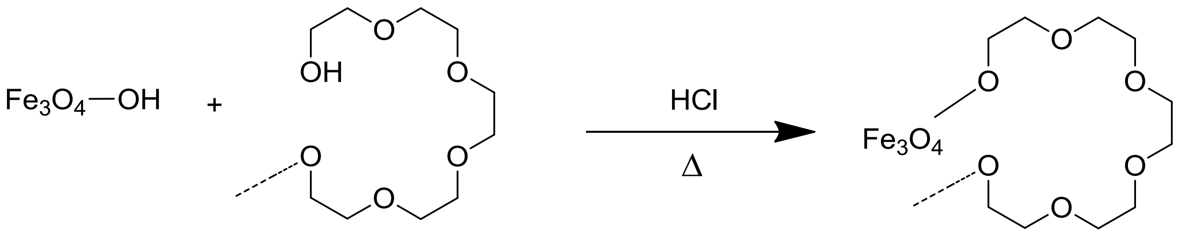
Spectral data for selected compounds

2,7,7-Trimethyl-5-oxo-4-(4-colorophenyl)-1,4,5,6,7,8-hexahydroquinoline-3-carboxylic acid ethyl ester (**6e**) (Table 3 entry 5) : Yield: 95%; m.p 242–244°C (Lit. 244–246°C) [38] ;IR (KBr) ν_{max}: 3271, 3202, 3071, 1704, 1646, 1275, 1211.; ^{1}HNMR (250 MHz, $CDCl_3$) δ 0.90 (s, 3H), 1.04 (s, 3H), 1.17 (t, *J 7 Hz*, 3H), 2.08–2.24 (m, 4H), 2.31 (m, 3H), 4.04 (q, *J6.9 Hz*, 2H), 5.00 (s, 1H), 6.50 (s, 1H), 7.12 (d, *J 8 Hz*, 2H),7.31 (d, *J 8 Hz*, 2H). ^{13}C NMR (250 MHz, $CDCl_3$): 15.12, 20.1, 28.2, 30.34, 33.5, 37.1, 41.7, 51.5, 60.4, 106.4, 112.2, 120.6, 130.5, 131.7, 144.4, 147.2, 149.2, 168.1, 196.4.

3. Results and discussion

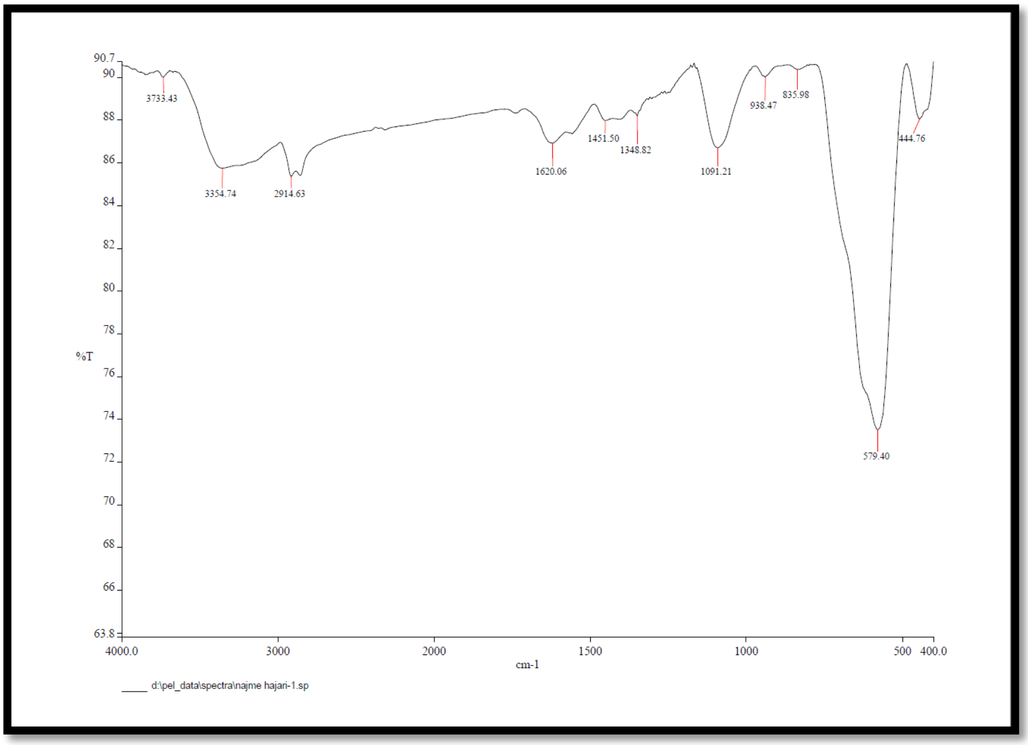
Fe_3O_4 is initially prepared from the co-precipitation reaction of $Fe^{\text{Ц}}$, $Fe^{\text{Ш}}$ in alkaline conditions. . In the present project, an attempt was made to stabilize polyethylene glycol as an environmentally friendly catalyst on the substrate of Fe3o4 nanoparticles. Due to the reaction conditions, its surface has hydroxy agents that can react with the end hydroxy agents of polyethylene glycol and attach to it under certain conditions. An overview of PEG/Fe_3O_4 preparation is given briefly in Scheme 2.

$$Fe^{\text{Ц}} + Fe^{\text{Ш}} \xrightarrow{\text{NH3}} Fe_3O_{4\ \text{nano particle}}$$



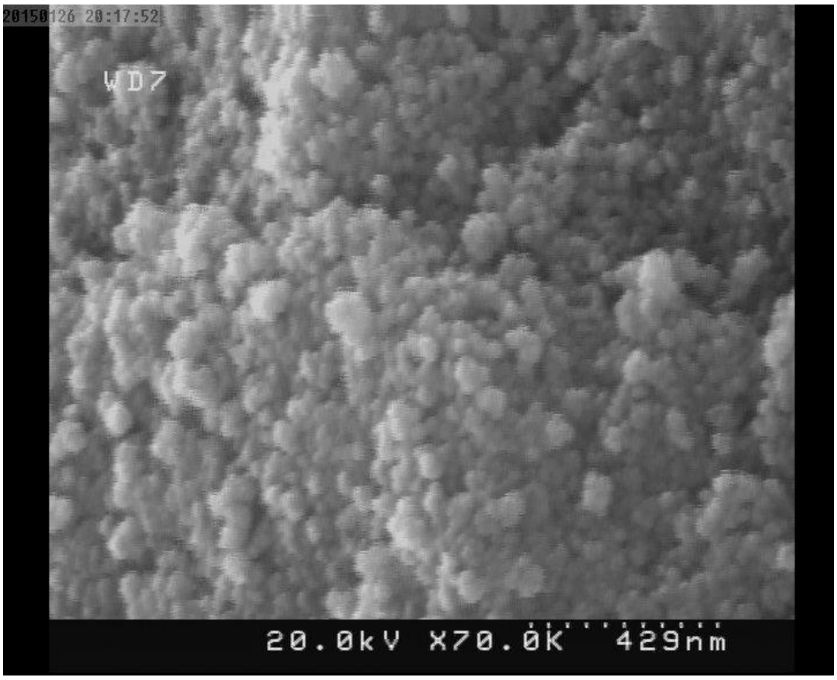
Scheme 2. Fabrication of PEG/Fe3O4 nanocomposite

In order to confirm the formation of the nanocomposite and the attachment of polyethylene glycol to the substrate of Fe_3O_4 nanoparticles, the FT-IR spectrum was taken from this compound. (FT-IR spectrum Scheme 3). Strong peak in the 579 nm region related to the tensile vibrations of Fe-o agents and line-sticking peaks 3000 cm^{-1} and below 291 cm^{-1} are related to the tensile vibrations of C-H of the organic species. In addition, C-O vibrations in the range of 1091 cm^{-1} are observed in the spectrum.



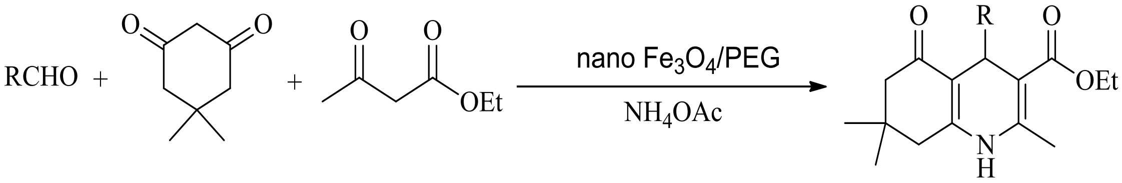
Scheme 3. FT-IR spectrum PEG/Fe_3O_4

In order to study the morphology of synthetic nanocomposites, SEM images in the **(Scheme4)** clearly confirm the formation of a nanostructure with an average size of 42 nanometers.



Scheme4. SEM PEG/Fe_3O_4 nano

In the next step, the catalytic effect of PEG/Fe_3O_4 nano on the synthesis of these compounds is investigated. Initially the densities between the four components of aromatic aldehyde, ethyl acetate as diketone and ammonium acetate and dimidone were investigated under solvent-free conditions (3-1).



Reaction (1): Preparation of polyhydroquinoline in the presence of PEG/Fe_3O_4 catalyst

Initially, in order to optimize the reaction conditions of benzaldehyde as an aromatic aldehyde, one millimole of it was added to one millimole of ethyl acetoacetate, 1 millimole of dimidone and one and a half moles of ammonium acetate and the resulting mixture in the presence of different temperatures Was investigated. The result of this reaction in Table (1) clearly shows that in the presence of 0.01 g of the catalyst, the reaction occurs in the shortest time and with higher efficiency. As a result, 0.01 g of catalyst was selected as the optimal value at 80°C.

According to the results, a temperature of 80°C and an amount of 0.01g of catalyst were selected as the optimal reaction conditions. After obtaining the optimal conditions, a variety of aromatic aldehydes containing electron donor and electron donor substitutions were reacted in a compression reaction with ethyl acetate and ammonium acetate and dimidone in the presence of PEG/Fe_3O_4 nanocomposite catalyst in solvent-free conditions. The general results obtained after recrystallization are given in Table (2).

Examination of the results of the table clearly shows the catalytic effect of synthetic nanocomposites in the preparation of various derivatives of polyhydroquinoline. Initially, in order to optimize the reaction conditions of benzaldehyde as an aromatic aldehyde, one millimole of it was added to one millimole of ethyl acetoacetate, 1 millimole of dimidone and one and a half moles of ammonium acetate and the resulting mixture in the presence of different temperatures Was investigated.

Table 1: Optimization of conditions for the condensation reaction of benzaldehyde (1 mmol), ethyl acetoacetate (2 mmol) and ammonium acetate under solvent-free conditions

Entry	NH_4OAc (mmol)	Catalyst (gr)	Temp (°C)	Time (min)	Yield (%)
1	2	0.01	80	5	98
2	1.5	0.00	80	----	----

3	1.5	0.01	80	5	99
4	1.5	0.01	60	20	42
6	1.5	0.01	25	100	35

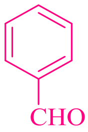
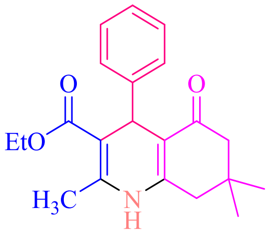
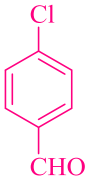
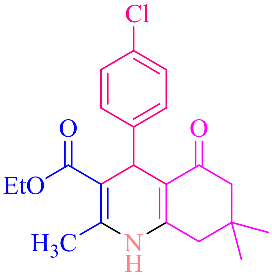
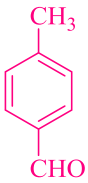
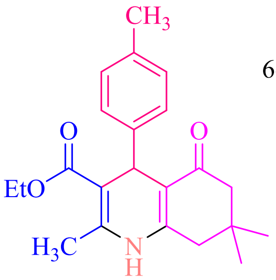
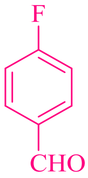
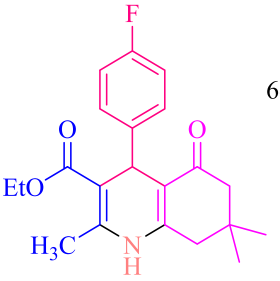
The result of this reaction in Table (3-1) clearly shows that in the presence of 0.01 g of the catalyst, the reaction occurs in the shortest time and with higher efficiency. As a result, 0.10 g of catalyst was selected as the optimal value at 80 °C.

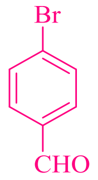
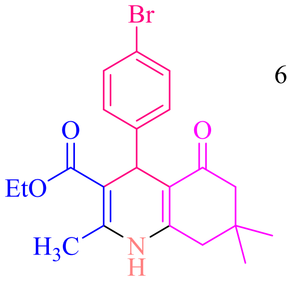
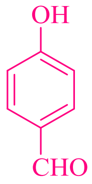
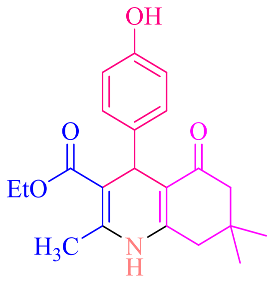
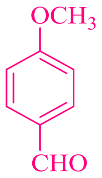
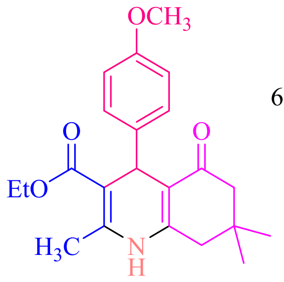
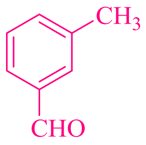
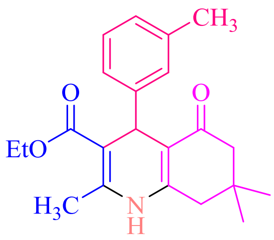
According to the results, a temperature of 80 °C and an amount of 0.01 g of catalyst were selected as the optimal reaction conditions. After obtaining the optimal conditions, a variety of aromatic aldehydes containing electron donor and electron donor substitutions were reacted in a compression reaction with ethyl acetate and ammonium acetate and dimidone in the presence of PEG/Fe_3O_4 nanocomposite catalyst in solvent-free conditions. The general results obtained after recrystallization are given in Table 2.

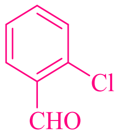
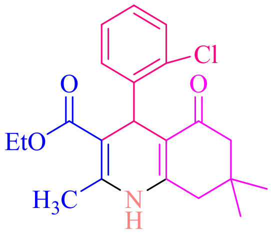
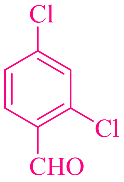
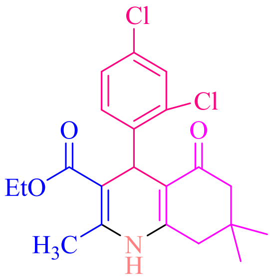
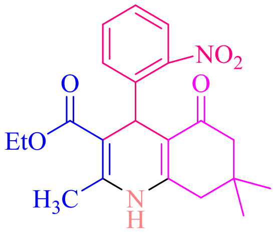
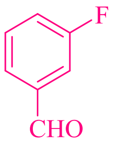
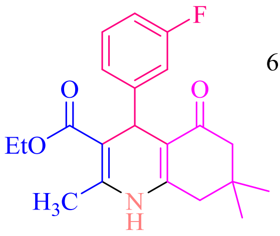
It can be postulated that the reaction is proceeded via a hydrogen abstraction and a knoevenagel condensation reaction. After nucleophilic attack of another ethylacetoacetate molecule, the subsequent imine formation occurs with the liberated ammonia from ammonium acetate. After dehydration, the target 1,4-dihydropyridine will be prepared (scheme 3).

Table 2 Synthesis of polyhydroquinoline derivatives catalyzed by PEG/Fe_3O_4 under solvent free thermal conditions.

Entry	R	Product	Time (min)	Yeild (%)	Melting point (°C) Found	Melting point (°C) Lit

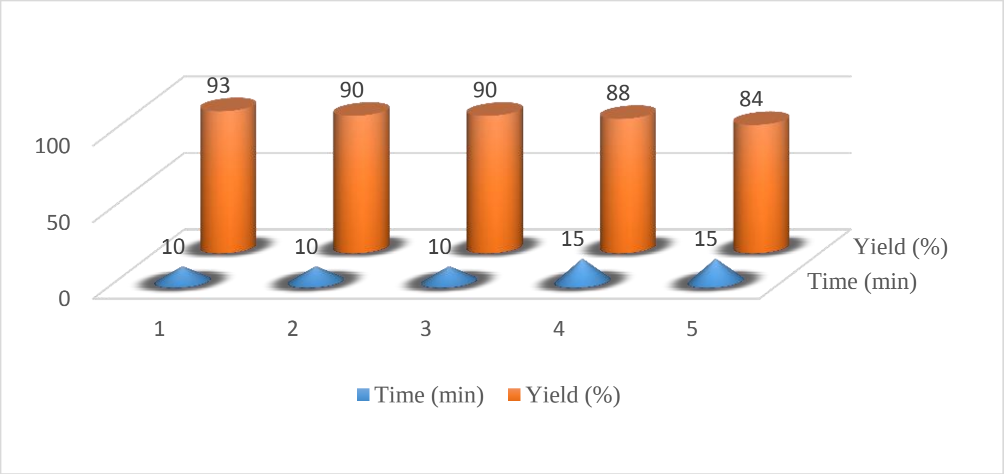
1		 6a	5	99	201-203	202-204[46]
2		 6b	10	98	242-244	244-246[46]
3		 6c	5	96	262-264	260-262[47]
4		 6d	5	96	186-188	185-187[47]

5		 6e	5	95	249-251	252–254[48]
6		 6f	10	88	234-236	232-234[48]
7		 6g	10	87	251-253	254-255[49]
8		 6h	5	90	257-259	256-258[49]

9		 6i	10	92	207-209	208-210[50]
10		 6j	10	84	242-244	240-242[50]
11		 6k	5	94	204-206	204-206[51]
12		 6l	10	90	241-243	242-245[51]

Examination of the results of the table clearly shows the catalytic effect of synthetic nanocomposites in the preparation of various derivatives of polyhydroquinoline. It can be proposed that the reaction proceeds as 1,4-dihydropyridines with the difference that in the first step, the knoevenagel condensation occur with dimedone and aromatic aldehyde, afterwards, a Michael addition occurs and after cyclization, the target polyhydroquinolines are prepared.

In order to evaluate the recyclability and reuse of the synthetic catalyst, after a series of compression reactions between benzaldehyde, ethyl acetate, dimidone and ammonium acetate in the presence of the nanocomposite, the catalyst was separated and after rinsing the water and ethanol were reactivated. There was no significant change in reaction time and frequency. The results are shown in Scheme 5.



Scheme5**.** Reusability of the catalyst in the reaction of benzaldehyde, ethyl acetoacetate and ammonium at 90 °C under solvent-free conditions.

At last, to show the superiority of the present approach in comparison with other catalysts, we compared our results with the reported results in the literature for the synthesis of PHQ. Results showed that while in most of these cases comparative yields of the desired product were obtained following the PEG/Fe_3O_4-catalyzed procedure, some of the previous procedures required long reaction time (Entries 3,4, 5, 6), or high catalyst loading (Entry 1, 2, 5, 7). These results clearly demonstrate that the presented methodology can be utilized as a useful acidic catalyst in organic methodology.

Table 3. Comparison of catalytic activity of PEG/Fe_3O_4 Nanostructures with some other catalysts

Entry	Catalyst	Conditions		Time (min)	Yield (%)	Catalyst loading	Ref
		Solvent	T/°C				
1	L-proline	EtOH	Reflux	360-420	81-92	10 mol%	[17]
2	ionic liquid	CH_3OH	Reflux	8	97	10 mol%	[10,11]
3	$Yb(OTf)_3$	EtOH	r.t	300	90	5 mol%	[12]
4	silica gel/$NaHSO_4$	Solvent-free	r.t	360	85	5 mol%	[13]
5	L-proline	EtOH	Reflux	360-420	81-92	10 mol%	[15]

6	$Sc(OTf)_3$	EtOH	r.t	120-360	86-95	5 mol%	[16]
7	ZnO	EtOH	80	60	92	10 mol%	[18]
8	PEG/Fe_3O_4	Solvent-free	80	5-10	87-99	5 mol%	This work

4. Conclusion

Due to the accelerating effect of the catalyst on the Hantzsch compression reaction, it can be imagined that the polyethylene glycol species encapsulate the reaction raw materials including aromatic aldehyde, dimidone, ethyl acetoacetate and increase the probability of material collision by creating a space. Increased collision probability has accelerated the reaction speed. The resulting products can be easily identified by the FT-IR spectrometer due to their special structures. The presence of the NH group, which forms a sharp peak in the range 3300 to 3600 cm^{-1}, and the carbonyl ester groups of vectors that peak in the range 1680-1735 cm^{-1} can be used to identify products. In addition, physical properties such as melting point were used to confirm the structure.

Acknowledgements

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