

Design and Synthesis of Novel Cage like CuFe₂O₄ Hollow Nanostructure as an Efficient Catalyst for Synthesis of 4,4'-(aryl methylene)bis(3-methyl-1*H*-pyrazol-5-ol)s

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Abstract

Cage like $CuFe_2O_4$ hollow nanostructure has been synthesized successfully using hard template method under the hydrothermal condition. $Cu(NO_3)_2 \cdot 6H_2O$, $Fe(NO_3)_2 \cdot 9H_2O$ and glucose were dissolved in water, and the mixture was heated to 180 °C in an autoclave. The removal of carbon was achieved by calcination at 800 °C and finally, the cage like $CuFe_2O_4$ hollow structure was obtained. This cage like $CuFe_2O_4$ hollow structure was characterized by FE-SEM, EDS, TEM and XRD. The catalytic performance of this hollow nanostructure was evaluated for the synthesis of bis pyrazol-5-ols. To this end, the one pot condensation reactions of phenylhydrazine, ethyl acetoacetate and different aromatic aldehyde at 80 °C under the solvent free condition were performed. The optimum amount of applied catalyst for this transformation was obtained to be 0.04 mol %. Noteworthy, catalyst was easily recoverable and was reused for 7 times with the remaining of its initial structure as well as its catalytic activity.

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Graphical Abstract

Cage like $CuFe_2O_4$ hollow nanostructure are stably and efficiently attainable for conversion of precursors to 4,4'-(aryl meth-ylene)bis(3-methyl-1H-pyrazol-5-ol)s



Keywords Hollow nanostructure \cdot CuFe₂O₄ \cdot Heterogeneous catalyst \cdot One pot reaction \cdot Bis pyrazole derivatives

1 Introduction

Soft magnetic materials have concerned several attentions in recent years due to their unique properties which make them as one of the most promising multifunctional future materials [1]. Among these magnetic materials, the cubic spinel structures (metal ferrites) have attracted more and more attention in material science and catalysis for their mechanical, electrical, chemical and magnetic properties [2-4]. The physiochemical properties as well as the catalytic performance of these materials are strongly influenced by the shape and morphology of these structures [5]. Therefore, the different morphologies and shapes of spinel structures such as spheres, wires, cubes and hollow structures in several applied fields have been reported [6–9]. Especially, in comparison with bulk materials, hollow nanostructures with low density and large specific surface area have been extensively studied due to their unique properties and the important potential applications [10]. Among them, the hollow spherical materials are shining because of their significant performance and widespread applications in photosynthesis, sensors, solar cells, optical devices and also in catalysis [11–15].

The 4,4'-(arylmethylene)bis(1*H*-pyrazol-5-ols) compounds are known as important chemicals because they display extensive domain of approved biological activities [16, 17], such as antipyretic [18], anti-inflammatory agents [19], antifungal [20], and antitumor [21]. Several methods and catalysts have been studied for synthesis of these heterocyclic compounds. The most common method for the synthesis of 4,4'-(arylmethylene)bis(1H-pyrazol-5-ol)s are reaction of 2 equivalent 3-methyl-1-phenyl-5-pyrazolone with aldehydes or the one-pot condensation of phenylhydrazine, ethyl acetoacetate, and aldehydes. Compared to the classical stepwise synthetic routes, the one-pot condensation is a promising approach owning to some advantages such as fewer by-products, and lower costs, time, and energy [22]. Poly(ethylene glycol) bound sulfonic acid [23], acetic acid or piperidine [24], ETBA [25], 1,3,5-tris(hydrogensulfato) benzene [26], lithium hydroxide monohydrate [27], ceric ammonium nitrate [28], N-(3-silicapropyl)-N-methyl imidazolium hydrogen sulfate [29], and silica-bonded S-sulfonic acid [30] HAP@AEPH2-SO3H [31], Sulfonated Honeycomb Coral [32], Fe₃O₄ Based Vanadic Acid [33], 2-Carbamoylhydrazine-1-sulfonic acid and carbamoylsulfamic acid [34]

have been used as catalysts for this transformation. Although these procedures provide an improvement in the synthesis of 4,4'-(arylmethylene)bis(3-methyl-1H-pyrazol-5-ol)s, but using of these methods have some shortcomings and limitations, such as harsh reaction conditions, long reaction times, moderate yields, non-recoverability of the catalyst as well as the use of toxic reagents and organic solvents. So, the designation of new heterogeneous catalyst possessing the recyclability and ease of separation for the clean chemical synthesis of 4,4'-(arylmethylene)bis(3-methyl-1H-pyrazol-5-ol)s is desirable. Despite the widespread application of hollow nanostructures in variety of fields, few articles have been published about the uses of these structures as a catalyst for synthesis of organic compounds [35-37]. So, it could be profitable to study hollow spherical materials with porous shells in the catalysis of the one-pot condensation reactions. As a part of our ongoing program in designing of novel heterogeneous catalysts and development of environmentally benign methods in organic reaction [38-49], we try to develop a facile synthetic protocol to construct cage like CuFe₂O₄ hollow nanostructure as efficient catalysts with high activity. Eventually, the prepared hollow nanostructure showed an acceptable and satisfactory catalytic activity for the synthesis of 4,4'-(arylmethylene)bis(3-methyl-1H-pyrazol-5-ol)s.

2 Experimental

2.1 Materials and Apparatus

The phase analysis of catalyst was studied using XRD with a Bruker D8 ADVANCE diffractometer. NMR spectra were obtained applying Bruker AMX 300 MHz instruments. FE-SEM images were recorded using a TESCAN, Model: MIRA3 scanning electron microscope operating at an acceleration voltage of 30.0 kV. Elemental compositions were determined with an SC7620 energy-dispersive X-ray analysis (EDX) presenting a 133 eV resolution at 20 kV. Transmission electron microscopy (TEM) was performed with a Leo 912 AB (120 kV) microscope (Zeiss, Germany). All compounds were obtained from Merck in analytical grade and were used without any further purification.

2.2 General Procedure for Preparation of Cage like CuFe₂O₄ Hollow Nanostructure

20 mL of 1 M glucose solution, 2 mL of $Fe(NO_3)_3 \cdot 9H_2O$ solution (1 M), 1 mL of $Cu(NO_3)_3 \cdot 6H_2O$ solution (1 M) were dispersed in 17 mL distilled water. After being stirred for about 20 min, the mixture was transferred into a Teflon-lined stainless steel autoclave. The autoclave was maintained at 180 °C for 24 h and then naturally cooled to ambient temperature. The precipitate was filtered, washed with water, and dried at 100 °C. Finally, the hollow spheres were obtained after calcination at 800 °C for 6 h.

2.3 General Procedure for Preparation of 4,4'-(phenylmethylene)bis(3-methyl-1 phenyl-1H-pyrazol-5-ol)

A mixture of aldehyde (1 mmol), ethyl acetoacetate (2 mmol), phenylhydrazine/hydrazinhydrate (2 mmol), and 0.04 mol % of catalyst was stirred at 80 °C under solvent free condition. The reaction progress was monitored by thin layer chromatography (TLC), after the completion of the reaction, hot ethanol (10 mL) was added to the reaction mixture and the catalyst was separated with external magnet and washed with ethanol. The mixture reaction was allowed to cool and the resulting precipitates were recrystallized from EtOH/ H_2O to obtain the pure compound. All the products were confirmed by the spectroscopic method using ¹H and ¹³C NMR. (See supporting information, Spectral data).

3 Results and Discussion

The strategy for synthesis of cage like $CuFe_2O_4$ hollow nanostructure is divided into two steps: 1) the simultaneous inclusion of metal ions into the carbon spheres during their formation under the designed hydrothermal process 2) The carbon spheres containing metal-ions were calcined in air to remove the carbon component, which result in the formation of the hollow nanostructure of porous $CuFe_2O_4$ spheres (Scheme 1).

Figure 1a shows the FE-SEM image of the uniformly distributed carbon spheres prepared from glucose/water solutions without any additives under the hydrothermal condition at 180 °C. Obviously, carbon spheres have the spherical morphology and their diameters are about 1 µm. The carboxylic and hydroxyl groups on the surface of carbon spheres provide the active sites for metal ion adsorption [50]. $Cu(NO_3)_2 \cdot 6H_2O$ and $Fe(NO_3)_2 \cdot 6H_2O$ were mixed in a solution of glucose at room temperature then heated at 180 °C in the hydrothermal process to achieve the metal ions-adsorbed carbon. Thus, during the hydrothermal formation of carbon spheres, the accumulation of metal ions into the hydrophilic shell of carbon could be expected. The FE-SEM image in Fig. 1b shows a significant increase in diameter of carbon spheres that confirms the presence of metal ions on the surface. Finally, following the calcination process, carbon sphere templates are thermally removed and the formation of the spinel structure of CuFe2O4 from metal atoms in the shell.

FE-SEM micrographs in Fig. 2a display the hollow nanostructure of the calcined sample, which obviously show the



Scheme 1 Synthesis of cage like CuFe2O4 hollow nanostructure from hydrothermally treated glucose/metal salts mixtures



Fig. 1 FE-SEM of a carbon spheres produced without any additive under hydrothermal method, b carbon spheres containing metal ions that formed by hydrothermal method

spherical porous particles with a diameter of about 700 nm. Compared to carbon microspheres, the mean diameter of the obtained structure is smaller, that confirms the formation of more compact structure during the calcination. The hollow spaces inside the cavities in the structure could be clearly observable. The formation of these cavities and hollow spaces are attributed to the release of CO_2 gas during the calcination. The EDS analysis also confirmed the removal of carbon by calcination at 800 °C and the existence of Cu and Fe elements in the final nanostructure (Fig. 2b).

The transmission electron micrograph (TEM) was brought in Fig. 3. It also confirmed that the $CuFe_2O_4$ spheres have the hollow and porous structure which is in good agreement with the results of FE-SEM.

The XRD pattern (Fig. 4) exposes that the product has the cubic spinel structure of $CuFe_2O_4$ with the lattice constants:

a = 8.396 Å, b = 8.396 Å and c = 8.396 Å. The diffraction peaks at 30.32, 35.56, 43.32, 53.72, 57.48 and 63.12 are attributed to (220), (311), (400), (422), (511) and (440) lattice planes of the cubic CuFe₂O₄, respectively (JCPDS: 77-0010).

In continuation of our recent studies of the application of heterogeneous catalysts in organic reaction, the catalytic performance of cage like $CuFe_2O_4$ hollow nanostructure was investigated for the synthesis of 4,4'-(arylmethylene) bis(3-methyl-1*H*-pyrazol-5-ol). To find a suitable reaction condition for the synthesis of 4,4'-(arylmethylene) bis(3-methyl-1*H*-pyrazol-5-ol)s, one-pot condensation of ethyl acetoacetate, phenylhydrazine and benzaldehyde as the model reaction were evaluated under diverse reaction conditions, such as, different solvents, diverse temperatures and the various amounts of catalyst. The results are summarized in Table 1. In the optimization of different solvents, **Fig. 2** a FE-SEM image of the cage like $CuFe_2O_4$ hollow structure **b** EDS spectrum of cage like $CuFe_2O_4$ hollow structure



the highest yield of corresponding product was obtained in the solvent free condition (Table 1, entry 5). In H₂O, EtOH/ H₂O, EtOH and CH₃CN, only 73, 88, 70 and 43% of the corresponding product was identified, respectively (Table 1, entries 1–4). So, we examined the different temperatures for the one-pot condensation of ethyl acetoacetate, phenylhydrazine and benzaldehyde catalyzed by cage like CuFe₂O₄ hollow nanostructure in the solvent-free condition. When the model reaction was carried out at 80 °C, the desired product was observed in 98% yields at 2 min (Table 1, entry 5). However, higher reaction times and lower yield of the corresponding product were obtained, when the reaction was performed at 60, 40 °C and room temperature. (Table 1, entries 6-8) Also, the yield of product increases from 60% to 98% with increase the amount of catalyst from 0.004 to 0.04 mol%. Higher amount of catalyst (0.06 mol %) has no significant influence on the yield of the product (Table 1, entries 9–12). When the model reaction was carried out for 10 min, no further progress in reaction (over 98%) has been observed (Table 1, entry 13).



Fig. 3 TEM image of cage like $CuFe_2O_4$ hollow structure



Fig. 4 The XRD pattern of cage like CuFe₂O₄ hollow structure

Based on the optimized reaction conditions, with 0.04 mol % of nanocatalyst under the solvent free condition at 80 °C, the efficiency of this protocol for other substrates

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were surveyed, and the results are summerized in Table 2. As demonstrated in Table 2, different types of aromatic aldehydes bearing electron-donating groups such as OH, OCH₃ and CH₃, were converted into the corresponding products in the excellent yields under the optimal reaction conditions (Table 2, entries 2–6). Also, we explored the synthesis of bis-pyrazole-5-ols starting from the phenylhydrazine and ethyl acetoacetate, by the one pot condensation reactions with aromatic aldehydes bearing electron-withdrawing groups such as Cl, NO₂ and obtained the corresponding products in high yields (Table 2, entries 7-11). Pyridine carboxaldehyde also reacted without any problems to provide the intended bis-pirazole-5-ols in high yields. (Table 2, entry 12) The use of furan-2-carbaldehyde afforded the bridged bis-pirazole-5-ols in 90% yield (Table 2, entry 13). In purpose to broaden the scope of our methodology, the feasibility of performing the reaction with hydrazine instead of phenylhydrazine was also studied. In this way, hydrazine was reacted with ethyl acetoacetate and aromatic aldehydes bearing both electron-withdrawing and electron-donating groups in the presence of cage like CuFe₂O₄ hollow nanostructure at 80 °C. This reaction resulted in the rapid formation of the corresponding products, in high yields (Table 2, entries 14-20). Therefore, these investigation revealed that cage like $CuFe_2O_4$ hollow structure catalyst system is also effective for the one pot condensation of phenylhydrazine or hydrazine, ethyl acetoacetate and aromatic aldehydes with high yields and short reaction times under the solvent free condition.

Recyclability of the cage like $CuFe_2O_4$ hollow nanostructure catalyst was also studied under the described reaction conditions. For this purpose, after completion of the reaction, catalyst was carefully separated using external magnetic field, washed and dried to be applied in the next synthesis run. No significant loss of activity of the catalyst is observed after seven reuse cycles (Fig. 5). In addition, TEM

Entry	Solvent	Catalyst (Mol %)	Temperature (°C)	Time (min)	Yield (%)
1	H ₂ O	0.04	80	40	73
2	H ₂ O/EtOH(1:1)	0.04	80	20	88
3	EtOH	0.04	Reflux	35	70
4	CH ₃ CN	0.04	Reflux	120	43
5	Solvent-free	0.04	80	2	98
6	Solvent-free	0.04	60	10	96
7	Solvent-free	0.04	40	45	94
8	Solvent-free	0.04	r.t.	120	45
9	Solvent-free	-	80	24 h	10
10	Solvent-free	0.004	80	40	60
11	Solvent-free	0.02	80	15	87
12	Solvent-free	0.06	80	2	98
13	Solvent-free	0.04	80	10	98

 Table 1
 Optimization of reaction conditions for one pot condensation reaction of phenylhydrazine, ethyl acetoacetate benzaldehyde
 Table 2 Synthesis of bis-pirazole-5-ols from various aldehyde, phenylhydrazine/hydrazinhydrate and ethyl acetoacetate in the presence of cage like $CuFe_2O_4$ hollow nanostructure as catalyst



Entry	R ¹	R^2	Time (min)	Yield (%)
1	Ph	C ₆ H ₅	2	98
2	Ph	$4-OHC_6H_4$	3	98
3	Ph	$3-OHC_6H_4$	6	90
4	Ph	$2-OHC_6H_4$	10	89
5	Ph	$4-OMeC_6H_4$	5	98
6	Ph	$4-MeC_6H_4$	6	93
7	Ph	$2-ClC_6H_4$	15	89
8	Ph	$4-ClC_6H_4$	8	96
9	Ph	2,4-(Cl) ₂ C ₆ H ₃	5	90
10	Ph	$3-NO_2C_6H_4$	10	92
11	Ph	$2-NO_2C_6H_4$	12	89
12	Ph	2-Pyridyl	15	94
13	Ph	2-Furyl	25	90
14	Н	C ₆ H ₅	8	98
15	Н	$4-ClC_6H_4$	20	98
16	Н	$4-\text{MeC}_6\text{H}_4$	7	95
17	Н	$3-OHC_6H_4$	8	96
18	Н	$2-OHC_6H_4$	10	90
19	Н	$3,5-(OMe)_2C_6H_3$	5	93
20	Н	$4-OMeC_6H_4$	8	98



Fig. 5 One pot condensation reaction of phenylhydrazine, ethyl ace-toacetate benzaldehyde for synthesis of bis pyrazol-5-ols in the presence of reused cage like $CuFe_2O_4$ hollow nanostructure

of the reused catalyst showed that there was no significant change in its morphology after the seven times of recycles (Fig. 6).



Fig. 6 TEM image of cage like $CuFe_2O_4$ hollow nanostructure after 7 times reuse

Entry	Reaction conditions	Yield (%)	Refer- ences
1	Sodium dodecyl sulfate, H ₂ O, Reflux, 60 min	86.8	[51]
2	Ultrasonic irradiation, H ₂ O/EtOH (1:1), r.t., 15 min	98	[52]
3	2-Hydroxy ethylammonium propionate, Solvent-free, 90 °C, 30 min	91	[53]
4	Silica sulfuric acid, EtOH/H ₂ O, 60	93	[54]
5	Silica-bonded N-propylpiperazine sulfamic acid, Solvent-free, 80 °C, 45 min	93	[55]
6	Cage like $CuFe_2O_4$ hollow nanostructure, Solvent-free, 80 °C, 2 min	98	This work

Table 3 Comparison of the efficiencies of a number of different reported catalysts with cage like $CuFe_2O_4$ hollow nanostructure in the synthesis of 4,4'-(phenyl methylene)bis(3-methyl-1*H*-pyrazol-5-ol)

The catalytic activity of cage like $CuFe_2O_4$ hollow nanostructure was compared with various reported catalysts in the literature [51–55] toward the synthesis of bis pyrazol-5-ols. It was observed that cage like $CuFe_2O_4$ hollow nanostructure promotes the yield of products and reaction time effectively in comparison with other catalysts. So, cage like $CuFe_2O_4$ hollow nanostructure can be considered as efficient catalyst in the synthesis of bis pyrazol-5-ols compounds (Table 3).

4 Conclusion

In summary, a general process applicable to the synthesis of cage like $CuFe_2O_4$ hollow nanostructure via hydrothermal method using $Cu(NO_3)_2 \cdot 6H_2O$, $Fe(NO_3)_3 \cdot 9H_2O$ and glucose solution as carbon source was reported. The study results indicate that the cage like $CuFe_2O_4$ hollow nanostructure shows a high catalytic activity in one pot condensation reaction of phenylhydrazine, ethyl acetoacetate and aldehyde for synthesis of bis pyrazol-5-ols. The high activity of this catalyst could be described by the presence of open cavities in this structure. It allows the reaction to be carried out into the interior spaces of catalyst structure due to the ability of the reactants for diffusion. In addition, the catalyst could be easily recovered and reused for seven times.

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