# Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>-CPTMS-Guanidine-SO<sub>3</sub>H-catalyzed One-Pot Multicomponent Synthesis of Polysubstituted Pyrrole Derivatives under Solvent-Free Conditions

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**Abstract**—A highly efficient procedure for the one-pot synthesis of polysubstituted pyrrole derivatives by the reaction between of aniline derivatives,  $\beta$ -diketones or  $\beta$ -ketoesters, and  $\beta$ -nitrostyrene derivatives in the presence of Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>–CPTMS-guanidine–SO<sub>3</sub>H as a reusable magnetic nanocatalyst is reported. The magnetic nanocatalyst was prepared and fully characterized by FTIR spectroscopy, scanning electron microscopy, energy dispersive X-ray spectroscopy, X-ray diffraction analysis, thermogravimetric analysis, and vibrating sample magnetometry.

Keywords: polysubstituted pyrrole derivatives, magnetic nanocatalyst, aniline derivatives,  $\beta$ -diketones,  $\beta$ -ketoesters,  $\beta$ -nitrostyrenes

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#### INTRODUCTION

Pyrrole derivatives [1] is an important group of heterocyclic compounds [2]. Compounds that contain the pyrrole ring have diverse biological activities such as antitumor, antibacterial, anti-inflammatory, antioxidant, and antifungal [3–9]. Moreover, natural pigments, such as bile pigments and chlorophyll, and enzymes similar cytochromes have the pyrrole ring in their structures [10]. In view of the importance of biologically active pyrrole derivatives, the development of efficient and environmentally friendly procedures for their synthesis is of special urgency.

The classical methods for the synthesis of pyrrole derivatives include the Hantzsch, Knorr, and Paal–Knorr reactions [11]. However, many reported syntheses of pyrrole derivatives feature such drawbacks as long reaction times, expensive reagents, heavy metal contaminations, and harsh reaction conditions.

Multicomponent reactions offer advantages over classical stepwise procedures, including the lack of need to isolate intermediates, atom economy, better yields, and reduction of time, cost, and waste [12, 13]. Heterogeneous catalysts [14], due their easy separation, are being more and more extensively used instead of homogeneous catalysts [15]. The advantages of magnetic nanoparticles (MNPs) [16], for example  $Fe_3O_4$ , as heterogeneous catalysts include the possibility of being simply separated by means of external magnet [17], low toxicity [18], highly active surface [19], and comfortable recovery and high stability [20]. This explains the increasing use of MNPs as heterogeneous catalysts in organic reactions.

To improve the mentioned limitations of the existing protocols, we prepared a new magnetic nanocatalyst  $Fe_3O_4@SiO_2$ -CPTMS-guanidine-SO\_3H and tested it in the synthesis of pyrrole derivatives under solvent-free conditions.

## **RESULTS AND DISCUSSION**

Synthesis and characterization of  $Fe_3O_4@SiO_2$ -CPTMS-guanidine-SO<sub>3</sub>H MNPs. The method of synthesis of  $Fe_3O_4@SiO_2$ -CPTMS-guanidine-SO<sub>3</sub>H MNPs presented in Scheme 1. The catalyst was synthesized in five steps. Initially,  $Fe_3O_4$  MNPs were synthesized from iron(II) and iron(III) salts. In the second stage,  $Fe_3O_4$  MNPs were coated with silica using tetraethoxysilane (TEOS). Then,  $Fe_3O_4@SiO_2$ 



Scheme 1. General scheme of synthesis of Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>-CPTMS-guanidine-SO<sub>3</sub>H MNPs.

MNPs were consecutively modified with (3-chloropropyl)trimethoxysilane (CPTMS) and guanidine. Finally, Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>-CPTMS-guanidine MNPs were treated with chlorosulfonic acid. The resulting Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>-CPTMS-guanidine-SO<sub>3</sub>H nanoparticles were characterized by FTIR spectroscopy, scanning electron microscopy (SEM), energy dispersive X-ray (EDX) spectroscopy, X-ray diffraction (XRD) analysis, thermogravimetric analysis (TGA), and vibrating sample magnetometry (VSM).



Fig. 1. FTIR spectra of (a)  $Fe_3O_4$ , (b)  $Fe_3O_4$ @SiO<sub>2</sub>, (c)  $Fe_3O_4$ @SiO<sub>2</sub>-CPTMS, (d)  $Fe_3O_4$ @SiO<sub>2</sub>-CPTMS-guanidine, and (e)  $Fe_3O_4$ @SiO<sub>2</sub>-CPTMS-guanidine-SO<sub>3</sub>H.

The FTIR spectra of Fe<sub>3</sub>O<sub>4</sub> (a), Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub> (b), Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>-CPTMS (c), Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>-CPTMS-guanidine (d) and Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>-CPTMS-guanidine-SO<sub>3</sub>H (e) are shown in Fig. 1. The FTIR spectrum of Fe<sub>3</sub>O<sub>4</sub> MNPs displays a stretching vibration band at 3391 cm<sup>-1</sup> related to symmetrical and asymmetrical modes of the O-H bonds and a stretching vibration band at 589 cm<sup>-1</sup> of the Fe-O bonds, which confirms the presence of Fe<sub>3</sub>O<sub>4</sub> (Fig 1a). The observation of a stretching vibration band at 1084 cm<sup>-1</sup> assignable to Si-O bonds provides evidence for the presence of SiO<sub>2</sub> on the surface of Fe<sub>3</sub>O<sub>4</sub> MNPs (Fig. 1b). The presence of C-H stretching vibration bands at 2898–3048 cm<sup>-1</sup>



200 nm Fig. 2. SEM image of Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>-CPTMS-guanidine-SO<sub>3</sub>H.

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nanocatalyst surface (Fig. 1c). The broad stretching vibration band near 3390 cm<sup>-1</sup> related to NH and NH<sub>2</sub> groups, as well as the stretching vibration band at 1657 cm<sup>-1</sup> related to the C=N bond confirms the presence of guanidine on the surface of Fe<sub>3</sub>O<sub>4</sub> particles (Fig. 1d). The presence of SO<sub>3</sub>H in the nanocatalyst is confirmed by the stretching vibration band at 2950–3500 cm<sup>-1</sup> (Fig. 1e).

The particle shape, surface morphology, and size distribution in the  $Fe_3O_4@SiO_2$ -CPTMS-guanidine-SO\_3H MNPs we characterized using SEM (Fig. 2). The SEM images show that the  $Fe_3O_4@SiO_2$ -CPTMSguanidine-SO\_3H nanoparticles are nearly spherical in shape, and their average size falls in the nano range.

The elemental composition of the  $Fe_3O_4@SiO_2-CPTMS$ -guanidine-SO<sub>3</sub>H nanoparticles was confirmed by EDX spectroscopy. The presence in the EDX



Fig. 4. XRD patterns of (a) Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub> and (b) Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>-CPTMS-guanidine-SO<sub>3</sub>H.

spectrum (Fig. 3) of peaks corresponding to Fe, Si, O, N, and S confirms that the successful synthesis of  $Fe_3O_4@SiO_2$ -CPTMS-guanidine-SO<sub>3</sub>H nanoparticles.

The XRD patterns of the Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub> and Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>–CPTMS–guanidine–SO<sub>3</sub>H nanoparticles are shown in Fig. 4. The two patterns display six peaks at 20 30.40°, 35.60°, 43.30°, 53.40°, 57.20°, and 63.80° with close intensity distributions, which are in a good agreement with the standard XRD pattern of Fe<sub>3</sub>O<sub>4</sub>.

The TGA curve of  $Fe_3O_4@SiO_2$ -CPTMS-guanidine-SO<sub>3</sub>H (Fig. 5) shows a small weight loss below 200°C, which is associated with the desorption of physically adsorbed solvents and surface hydroxyl groups. The  $Fe_3O_4@SiO_2$ -CPTMS-guanidine-SO<sub>3</sub>H weight loss between 200 and 600°C is related to the degradation of organic moieties.



Fig. 6. Magnetization curves of (a)  $Fe_3O_4$ , (b)  $Fe_3O_4$ @SiO<sub>2</sub>, and (c)  $Fe_3O_4$ @SiO<sub>2</sub>-CPTMS-guanidine-SO<sub>3</sub>H.



Fig. 5. IGA curve of  $Fe_3O_4(@SIO_2-CPTMS-guantatine-SO_3H.$ 





<sup>a</sup> p-Chloroaniline (0.5 mmol), ethyl acetoacetate (0.5 mmol),  $\beta$ -nitrostyrene (0.5 mmol), solvent (3 mL).

The magnetic properties of the nanocatalyst were studied by VSM. The magnetization curves of Fe<sub>3</sub>O<sub>4</sub>, Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>, and Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>–CPTMS–guanidine–SO<sub>3</sub>H nanoparticles are shown in Fig. 6. The saturation magnetizations of the studied nanoparticles were estimated at 47, 45, and 39 emu g<sup>-1</sup>, respectively. The decreased saturation magnetization of Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>–CPTMS–guanidine–SO<sub>3</sub>H confirmed its successful synthesis.

The concentration of  $SO_3H$  groups on the catalyst surface as estimated by back titration with 0.01 M KOH solution was 1.2 mmol g<sup>-1</sup> catalyst.

Evaluation of the catalytic activity of  $Fe_3O_4@SiO_2$ -CPTMS-guanidine-SO<sub>3</sub>H MNPs in the synthesis of pyrrole derivatives. To evaluate the catalytic activity of the  $Fe_3O_4@SiO_2$ -CPTMS-guani-

dine–SO<sub>3</sub>H nanocatalyst, we performed the synthesis of polysubstituted pyrroles by the reaction of various aniline derivatives,  $\beta$ -diketones/ $\beta$ -ketoesters, and  $\beta$ -nitrostyrene derivatives.

To optimize the reaction conditions, we selected the reaction of *p*-chloroaniline, ethyl acetoacetate, and  $\beta$ -nitrostyrene as the model reaction. The resulting data are presented in Table1. Initially, the effect of the amount of the catalyst was investigated in solvent-free coditions. As seen from Table 1, the reasonable result was obtained with 0.01 g of the catalyst at the reaction time of 3 h. Then, the 3-h reaction was evaluated in various solvents. Solvent-free was found a suitable condition for this reaction. Also, the effect of the temperature was checked, and 50°C was chosed as the optimum temperature for this reaction.

Table 2. Synthesis of functionalized pyrroles 4.



Having optimized the reaction conditions, we synthesized a broad range of pyrrole derivatives. The results are summarized in Table 2.

A possible mechanism of the synthesis of pyrrole derivatives **4**, proposed on the basis of the previously reported ones [23, 26], is shown in Scheme 2. First, the the nanocatalyst activates the carbonyl group of the  $\beta$ -diketone/ $\beta$ -ketoester, and the latter reacts with the amine to form an enamine. Then, the enamine reacts with the  $\beta$ -nitrostyrene by way of the Michael addition to produce intermediate **5**. The subsequent intramolecular cyclization in the latter leads to pyrrole precursor **6** which converts into pyrrole **4** via elimination of HNO and H<sub>2</sub>O.

To demonstrate the efficiency of the  $Fe_3O_4@SiO_2-CPTMS$ -guanidine-SO<sub>3</sub>H nanocatalyst in the syn-

thesis of pyrrole derivatives, we compared the results and conditions of the synthesis of pyrrole **4g** with this system with systems used in previous researches (Table 3).

The reusability of the Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>–CPTMS–guanidine–SO<sub>3</sub>H nanocatalyst was assessed in the synthesis of pyrrole **4f**. The recovered nanocatalyst was recycled five times without significant loss of activity, yield, % (run no.): 95 (1), 93 (3), 90 (3), 85 (4), and 80 (5).

## CONCLUSIONS

In this research, we have developed an efficient approach to the one-pot synthesis of pyrroles. The advantages the developed procedure offers over the previously reported ones include solvent-free conditions, use of a nanocatalyst that is easily



Scheme 2. Proposed mechanism for the synthesis of pyrroles derivatives.

extracted from the reaction mixture, short reaction time, low reaction temperature reaction, and good yields.

## **EXPERIMENTAL**

All reagents and solvents were purchased from Merck, Sigma-Aldrich, and Fluka and used without further purification. The IR spectra were measured on a Bruker Vertex70 spectrometer in KBr. The <sup>1</sup>H NMR spectra were obtained on a Bruker DRX-300 Avance spectrometer in CDCl<sub>3</sub> at 300 MHz. Scanning electron microscopy was performed on a JEOL JEM-2010 instrument. The EDX spectra were measured on a

Bruker XFlash 6130 EDS system. The XRD patterns were obtained on an ItalStructures APD-2000 diffract-tometer ( $\lambda$  1.54 Å). Thermogravimetric analysis was performed on a TA Instruments SDT-Q600 Simultaneous TGA/DSC instrument. Superparamagnetic properties of the catalyst were studied using a vibrating sample magnetometer (MDKFD, Iran).

**Preparation of Fe<sub>3</sub>O<sub>4</sub> MNPs.** A mixture of FeCl<sub>3</sub>·6H<sub>2</sub>O (4.865 g, 0.018 mol) and FeCl<sub>2</sub>·4H<sub>2</sub>O (1.789 g, 0.0089 mol) was dissolved in 100 mL of deionized water, and then 10 mL of 25% NH<sub>4</sub>OH was added to the solution under N<sub>2</sub> atmosphere at 80°C. The reaction mixture was stirred about 30 min with a

Entry	Catalyst	Conditions	Time, h	Yield, %	Reference
1	β-Cyclodextrin	H <sub>2</sub> O, 70–80°C	8	90	[27]
2	Iodobenzene, Oxone	EtOH, reflux	2	78	[24]
3	(Diacetoxyiodo)benzene	EtOH, reflux	4	74	[25]
4	FeCl <sub>3</sub>	CH <sub>3</sub> NO <sub>2</sub> , reflux	14	54	[11]
5	FeCl <sub>3</sub>	CH <sub>3</sub> NO <sub>2</sub> , reflux	8	88	[28]
6	Fe <sub>3</sub> O <sub>4</sub> @SiO <sub>2</sub> -CPTMS-guanidine-SO <sub>3</sub> H	Solvent-free, 50°C	3	95	This work

Table 3. Comparison of the activity various catalysts for the synthesis of pyrrole 4g.

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mechanical stirrer. The black precipitate was separated by magnetic decantation, washed with 5 portions of distilled water, and dried at room temperature [29].

**Preparation of Fe<sub>3</sub>O<sub>4</sub>**(*a*)**SiO<sub>2</sub> MNPs.** A dispersion of 2 g of Fe<sub>3</sub>O<sub>4</sub> MNPs in 20 mL of water was sonicated for 30 min, after which EtOH (50 mL), PEG (5.36 g), H<sub>2</sub>O (20 mL), NH<sub>4</sub>OH (10 mL), and tetraethyl orthosilicate (TEOS) (2 mL) were successively added to the suspension. The mixture was stirred for 38 h at room temperature and then the product was separated by magnetic decantation, washed with EtOH, and dried at room temperature [29].

**Preparation of Fe<sub>3</sub>O<sub>4</sub>@SiO–CPTMS MNPs.** A dispersion of 1 g of Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub> MNPs in 25 mL of water and 25 mL of EtOH was sonicated for 30 min. Then (3-chloropropyl)triethoxysilane (CPTMS) (2.5 mL) was added, and the mixture was stirred at 40°C for 8 h. The precipitate was separated by magnetic decantation, washed several times with EtOH, and dried at room temperature [29].

**Preparation of Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>–CPTMS–guanidine MNPs.** The Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>–CPTMS nanoparticles (1 g) were in 8 mL of toluene for 10 min and then NaHCO<sub>3</sub> (0.008 mol) and guanidine chloride (0.004 mol) were added to the dispersion. The reaction mixture was refluxed for 30 h, cooled down to room temperature, the precipitate was separated, washed several times with EtOH and water, and dried at room temperature [30].

**Preparation of Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>–CPTMS–guanidine–SO<sub>3</sub>H MNPs.** A dispersion of the synthesized Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>–CPTMS–guanidine MNPs (0.5 g) in CH<sub>2</sub>Cl<sub>2</sub> (10 mL) was sonicated for 30 min, after which chlorosulfonic acid (1.5 mL) was added dropwise over the course of 20 min, and the mixture was stirred for 3 h at room temperature. The resulting Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>– CPTMS–guanidine–SO<sub>3</sub>H MNPs were separated by magnetic decantation, washed twice with CH<sub>2</sub>Cl<sub>2</sub>, EtOH and CH<sub>2</sub>Cl<sub>2</sub> in succession, and dried at room temperature [29].

Synthesis of polysubstituted pyrrole derivatives (general procedure).  $\beta$ -Nitrostyrene (0.5 mmol) was added to a mixture of aniline (0.5 mmol) and  $\beta$ -diketone or  $\beta$ -ketoester (0.5 mmol) under solvent-free coditions. The Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>-CPTMS-guanidine-SO<sub>3</sub>H nanocalyst (0.01 g) was then added, and the reaction mixture was stirred for 3 h at 50°C. After the reaction had been complete, the reaction mixture

was cooled to room temperature, the catalyst was separated by means of external magnet, and the product was extracted with dichloromethane and purified by column chromatography on silica gel (n-hexane–ethyl acetate, 8:1).

**1-[1-(4-Bromophenyl)-4-(4-chlorophenyl)-2-methyl-1H-pyrrol-3-yl]ethan-1-one (4h).** Yield 83%, yellow solid, mp 164–166°C. IR spectrum, v, cm<sup>-1</sup>: 1648 (C=O), 1400, 1555 (Ar), <sup>1</sup>H NMR spectrum (CDCl<sub>3</sub>),  $\delta$ , ppm: 2.09 s (3H, CH<sub>3</sub>), 2.40 s (3H, CH<sub>3</sub>), 6.64 s (1H, CH), 7.21–7.65 m (8H, CH).

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