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Article

A $\text{Fe}_3\text{O}_4@\text{Nico}@\text{Ag}$ nanocatalyst for the hydrogenation of nitroaromatics

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ABSTRACT

We report the fabrication and characterization of a magnetically recyclable $\text{Fe}_3\text{O}_4@\text{Nico}@\text{Ag}$ catalyst for reduction reactions in the liquid phase. Fe_3O_4 is a magnetic core and nicotinic acid was used as the linker for Ag. The characterization was done with X-ray diffraction, Fourier transform infrared spectroscopy, scanning electron microscopy, thermogravimetric analysis, vibrating sample magnetometry (VSM), and ultraviolet-visible spectroscopy. VSM measurements proved the superparamagnetic property of the catalyst.

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1. Introduction

Nitroaromatic compounds are dangerous and poisonous to the environment (humans, fishes, and invertebrates) due to the presence of the nitro group. They are widespread environmental contaminants in the soil and groundwater as a result of their wide use for manufacturing dyes, drugs (several analgesic and antipyretic drugs), pesticides, herbicides, fungicides, paints, and explosives [1–6]. In the dye industry, 4-nitroaniline is also an important nitroaromatic compound and a major component of hazardous wastewater. These refractory organics are hard to decompose with conventional ways such as the active sludge method. They are used as the precursor in several chemical synthesis of various azo dyes, poultry medicine, antioxidants, antiseptic agents, pesticides, fuel additives as well as an important corrosion inhibitors [7]. Their disposal in wastewater results in high toxicity, carcinogenicity, and mutagenicity to several organisms [8–12]. However, aminophenol compounds are important for the

preparation of several analgesic and antipyretic drugs such as paracetamol, acetanilide, and phenacetin.

A nicotinic acid complex with a metal, Mn, Co, Ni, Zn, or Cu, has been synthesized and reported in several research works [13]. Due to the electron donor behavior of the N atom of nicotinic acid, it has the ability to attract the metal through the N atom of the pyridine [14,15]. Fe_3O_4 nanoparticles (NPs) have been extensively researched for many years for their supermagnetic behavior and catalytic properties, including their use in many applications such as microwave absorption, medical diagnostics, and catalytic degradation [16–19]. Ag NPs have been studied and found to be an effective catalytic material [20–25]. The chemical grafting of a homogeneous metal complex onto a solid magnetic surface is a versatile method to synthesize a metal complex with a regulated metal-coordination structure having special catalytic properties [26–30].

In recent years, due to their high surface-to-volume ratio and unique electronic and surface properties, nanoparticles

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have gained much attention for catalysis [31,32]. There are many reports in the literature on the application of metal nanoparticles (Au, Ag, Pd, and Pt NPs are the most common) as catalyst for the reduction of nitrophenols to aminophenols in the presence of NaBH_4 [33,34]. Due to the easy separation of magnetic nanoparticles from the reaction medium (due to the superparamagnetic nature of the material), nanocomposites which combine a noble metal with a magnetic material have been extensively studied. Ag and Fe_3O_4 both have catalytic activity and several nanocatalysts have been synthesized and reported, e.g., magnetic photocatalyst $\text{Fe}_3\text{O}_4@\text{C}@\text{Ag}$ [35], $\text{Fe}_3\text{O}_4@\text{C}@\text{Ag}$, and Ag-coated $\text{Fe}_3\text{O}_4@\text{TiO}_2$ [36,37], and Au- Fe_3O_4 heterostructures for nitrophenol reduction [38]. The use of a $\text{Fe}_3\text{O}_4@\text{Nico}@\text{Ag}$ magnetic nanocatalyst for the reduction of 4-nitrophenol and 4-nitroaniline and their mixture has not been reported so far.

2. Experimental

2.1. Chemicals and instruments

$\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$, nicotinic acid, AgNO_3 , NaBH_4 , 4-nitroaniline, 4-nitrophenol, and NH_3 were obtained from Merck and used without further purification. Fourier transform infrared (FT-IR) spectra were recorded in the transmission mode with a PerkinElmer BX FT-IR spectrometer. The powder samples were ground with KBr and pressed into a pellet. FT-IR spectra in the range $4000\text{--}400\text{ cm}^{-1}$ were recorded to investigate the nature of the chemical bonds formed.

The crystalline structure of the nanoparticles was determined with X-ray diffraction (XRD) measurements using a Rigaku D/Max-IIIC instrument with $\text{Cu-K}\alpha$ radiation in the 2θ range of $20^\circ\text{--}70^\circ$. The surface morphology of the composite was analyzed with a JEOL JSM 7001F scanning electron microscope (SEM).

The ultraviolet-visible (UV-Vis) spectrometer used was a Model Shimadzu UV-Vis 2600 in the range of $300\text{--}800\text{ nm}$. The thermal stability was determined by thermogravimetric analysis (TGA, PerkinElmer Instruments model, STA 6000). The TGA thermograms were recorded with 6 mg of powder sample at a heating rate of $10^\circ\text{C}/\text{min}$ in the temperature range of $30\text{--}800^\circ\text{C}$ under a N_2 atmosphere. Vibrating sample magnetometry (VSM) measurements were performed using a vibrating sample magnetometer (LDJ Electronics Inc., Model 9600). The magnetization measurements were carried out in an external field up to 15 kOe at room temperature.

2.2. Preparation of the $\text{Fe}_3\text{O}_4@\text{Nico}@\text{Ag}$ nanocomposite

The $\text{Fe}_3\text{O}_4@\text{Nico}$ nanocomposite was synthesized by the reflux method. Stoichiometric amounts of metal salts, Fe (III) and Fe (II) chlorides with the mole ratio of $\text{Fe}^{3+}/\text{Fe}^{2+}$ equal to 2, were dissolved in 40 mL of distilled water in a three neck round-bottom flask and their homogeneous solution was prepared using magnetic stirring. A stoichiometric amount of nicotinic acid was added to this mixture under vigorous stirring and then concentrated NH_3 solution was added dropwise under

constant stirring to adjust the pH to 10 at which the precipitation of the ferrites takes place. After transferring the flask to the heating mantle, it was refluxed under a N_2 atmosphere at 80°C for 5 h. Finally, the synthesized $\text{Fe}_3\text{O}_4@\text{Nico}$ nanocomposite was separated by a permanent magnet and washed with distilled water and ethanol solution several times to remove impurities. A black powder product was obtained and dried at 80°C for 4 h.

The synthesized $\text{Fe}_3\text{O}_4@\text{Nico}$ nanocomposite (150 mg) was dispersed in 50 mL of deionized water and sonicated for 30 min. This was followed by the addition of 30 mL of AgNO_3 solution (0.2 mmol/L). The solution was vigorously stirred for 30 min and then 0.6 g of NaBH_4 was quickly added and the mixture was allowed to react for 3 h under rapid stirring. The product was separated magnetically and washed several times with deionized water and ethanol to eliminate impurities. The synthesis of the $\text{Fe}_3\text{O}_4@\text{Nico}@\text{Ag}$ nanocomposite is outlined in the Scheme 1.

3. Results and discussion

3.1. Characterizations

The XRD powder pattern of the $\text{Fe}_3\text{O}_4@\text{Nico}@\text{Ag}$ nanocatalyst is given in Fig. 1(a). It indicated the presence of both Fe_3O_4 ((220), (311), (400), (422), (511), (440)) [39] and Ag ((111) and (200)) [40]. All the diffraction peaks matched well with Ag (JCPDS 87-0720) NPs and Fe_3O_4 NPs (JCPDS 75-0033). Using Scherer's formula and the FWHM of the strongest peak of the Fe_3O_4 NPs, the average crystallite size was estimated as 10.1 nm.

The FT-IR spectra of the $\text{Fe}_3\text{O}_4@\text{Nico}$ nanocomposite, Fe_3O_4 , and nicotinic acid are presented in Fig. 1(b). The surface molecules in the adsorbed state were subjected to the field of the solid surface. It is worth noting that the C=O stretching band of the carboxyl group, which was present at 1696 cm^{-1} in the pure nicotinic acid, was absent in the nanocomposite. Tao et al. [41] and Ahn et al. [42] suggested two binding modes for the surface carboxylate bonding. When the carboxylate is bonded symmetrically to the surface, only the symmetric C=O stretching band appears at 1404 cm^{-1} [41,43]. In our case, only one peak at 1403 cm^{-1}

Scheme 1. Illustration for the fabrication of the $\text{Fe}_3\text{O}_4@\text{Nico}@\text{Ag}$ magnetic nanocatalyst.

Fig. 1. XRD pattern (a), FT-IR spectra (b), TG curves (c), and room temperature magnetization curve (d) of Fe_3O_4 @Nicotinic acid@Ag magnetic nanocatalyst (1), Fe_3O_4 @Nicotinic acid (2), and Nicotinic acid (3).

corresponding to the symmetric stretching was observed. Therefore, our conclusion was that both the carboxylic acid oxygen atoms were symmetrically bonded to the Fe_3O_4 nanoparticle surface [44]. The bonding of nitroaniline (NA) to the Fe_3O_4 surface was explained in detail in our previous paper [45].

The following FT-IR stretches can be assigned to the pristine NA: $\nu(\text{C}=\text{O}) = 1700 \text{ cm}^{-1}$, $\nu(\text{C}-\text{O}) = 1300 \text{ cm}^{-1}$, $\nu(\text{C}-\text{N}) = 1326 \text{ cm}^{-1}$ [46]. The characteristic free NA C–N stretching absorption of the pyridine ring ($\nu = 1321 \text{ cm}^{-1}$) was shifted to a higher frequency at 1340 cm^{-1} due to the binding between the N group of NA and the Ag NPs. This suggested that the Ag nanoparticles were formed by the coordination of the pyridine ring N atom [45,47].

The TG curves of pristine nicotinic acid and Fe_3O_4 @Nico@Ag magnetically recyclable nanocatalyst (MRC) are presented in Fig. 1(c). The pristine nicotinic acid has mass losses in two steps up to 180°C (due to the evaporation of adsorbed water) and at 271°C (due to the decomposition of the organic layer) [48]. Above this temperature, nicotinic acid was converted into gaseous products. On the other hand, the product (Fe_3O_4 @Nico@Ag MRC) also has two step mass losses. The first mass loss was at 200°C (due to dehydration). Then the anhydrous compound was stable up to 300°C , and above this temperature the thermal decomposition of organic layer (oxidation of the organic matter) took place. Up to the end temperature, decomposition of the inorganic layer (due to the inorganic content, Fe_3O_4 and Ag) did not occur (Fig. 1(b)). The

TG analysis showed that the Fe_3O_4 @Nico@Ag MRC consisted of 90% inorganic and 10% organic layers.

Figure 1(d) shows the room temperature magnetization curve of Fe_3O_4 @Nico@Ag. The material has no remanence or coercivity at 27°C , which indicated that Fe_3O_4 @Nico@Ag was superparamagnetic at room temperature. The saturation magnetization of the product was 52.4 emu/g .

SEM analysis was performed to evaluate the morphology of the composite nanoparticles, and the results are presented in Fig. 2. The particles of iron oxide were observed to form agglomerates of 20–50 nm in both Fig. 2(a) and (b). EDX analysis was also performed to detect the presence of all the components and the co-presence of Fe, N, C, O, and Ag for the Fe_3O_4 @Nico@Ag MRC and Fe and O for the Fe_3O_4 NPs, which were confirmed. Using the EDX data and assuming a particle size of 20 nm for Fe_3O_4 and 3–4 nm for the Ag NPs, the surface coverage density of Ag NPs on Fe_3O_4 was estimated as 50%.

3.2. Catalytic studies

For the catalytic reduction experiment, 4-nitroaniline solution ($100 \mu\text{L}$, 10 mmol/L) and aqueous freshly prepared NaBH_4 (1 mL , 100 mmol/L) were added to a UV cuvette. The volume of the mixture was adjusted to 3 mL with distilled water. Finally, 1 mg of Fe_3O_4 @Nico@Ag nanocatalyst was used to catalyze the 4-nitroaniline solution, during which the color of the solution vanished, indicating the degradation of the 4-nitroaniline solution. The reduction was monitored with a UV-vis spectrophoto-

Fig. 2. SEM images of Fe₃O₄ NPs (a) and Fe₃O₄@Nicotinic acid@Ag magnetic nanocatalyst (b).

tometer. After the degradation was complete, the Fe₃O₄@Nico@Ag nanocatalyst was separated using a magnet and the process was repeated to investigate the recyclability of the catalyst. A similar procedure was used for the catalytic reduction of 4-nitrophenol. At the end of the reaction, the color of

Fig. 3. Schematic of the reduction of 4-nitrophenol to 4-aminophenol and 4-nitroaniline to *p*-phenylenediamine by the Fe₃O₄@Nico@Ag magnetic nanocatalyst in an excess of NaBH₄.

the solution vanished. The color of each solution changed to colorless, which indicated the degradation of 4-nitrophenol and 4-nitroaniline. The results of the reduction of 4-nitrophenol to 4-aminophenol and 4-nitroaniline to *p*-phenylenediamine by the Fe₃O₄@Nico@Ag magnetic nanocatalyst in an excess of NaBH₄ are presented in Fig. 3.

To investigate the catalytic performance of Fe₃O₄@Nico@Ag, 4-nitrophenol (4-NP) and 4-nitroaniline (4-NA) reduction reactions were carried out as model reactions in the presence of NaBH₄. In the absence of the Fe₃O₄@Nico@Ag magnetic nanocatalyst, as seen in Fig. 4(a) and (b), there was no significant change in the absorption intensity with time. On the other hand, as shown in Fig. 4(c) and 4(d), when the Fe₃O₄@Nico@Ag magnetic nanocatalyst was introduced into

Fig. 4. UV-Vis absorption spectra of 4-NP (a) and 4-NA (b) at different time in the presence of only NaBH₄, and 4-NP (c) and 4-NA (d) reduction by NaBH₄ in the presence of Fe₃O₄@Nico@Ag.

Fig. 5. (a) Plot of rate constant versus time for the reduction of 4-NP, 4-NA, and mixture of 4-NP and 4-NA by NaBH_4 in the presence of the $\text{Fe}_3\text{O}_4@\text{Nico}@\text{Ag}$ magnetic nanocatalyst; (b) Reusability of the $\text{Fe}_3\text{O}_4@\text{Nico}@\text{Ag}$ catalyst for the reduction of 4-NP and 4-NA with NaBH_4 .

the solution, the absorption at 400 nm for 4-NP and 380 nm for 4-NA decreased within 6 and 4 min, respectively, which indicated the formation of 4-aminophenol and *p*-phenylenediamine [49–57].

Since the concentration of NaBH_4 was higher than those of 4-NP and 4-NA, the rate of reduction was independent of the concentration of NaBH_4 , thus the rate constants for the reduction of 4-NP and 4-NA was evaluated by pseudo-first order kinetics. As the absorbance of 4-NP or 4-NA is proportional to its concentration, the ratio of the absorbance at time t (A_t) to that at $t = 0$ (A_0) is proportional to the concentration at time t (C_t) to that of $t = 0$ (C_0), i.e., $A_t/A_0 \propto C_t/C_0$. The reaction rate constant K was calculated from the linear correlation between $\ln(A_t/A_0)$ and time as shown in Fig. 5. The reaction rate constant k was found to be 0.34 and 0.51 min^{-1} for 4-NP and 4-NA, respectively. We have also investigated the catalytic activity of the $\text{Fe}_3\text{O}_4@\text{Nico}@\text{Ag}$ magnetic nanocatalyst for a mixture of 4-NP and 4-NA in the presence of NaBH_4 , which clearly demonstrated that the reduction of both compounds occurred within 7 min as shown in Fig. 6. The rate constant of the reaction, k , obtained from the slope of the straight line was found to be 0.35 min^{-1} for the mixture, which is shown in Table

Fig. 6. Time dependent UV-Vis absorption spectra of a mixture of 4-NP and 4-NA and NaBH_4 in the presence of $\text{Fe}_3\text{O}_4@\text{Nico}@\text{Ag}$.

Table 1

Complete conversion time and rate constants of the $\text{Fe}_3\text{O}_4@\text{Nico}@\text{Ag}$ nanocatalyst catalyzed reduction reactions of 4-NP and 4-NA.

Organic compound	Completion time (min)	Rate constant k (min^{-1})	Correlation coefficient
4-NP	6	0.34	0.9734
4-NA	4	0.51	0.9821
Mixture of 4-NP and 4-NA	7	0.35	0.9954

1. Our results also showed that the reaction rate constant was higher than in previous studies [32,58,59]. The reusability of the $\text{Fe}_3\text{O}_4@\text{Nico}@\text{Ag}$ catalyst was examined. The results are shown in Fig. 5(b). With a high saturation magnetization, $\text{Fe}_3\text{O}_4@\text{Nico}@\text{Ag}$ can be easily separated from the reaction mixture using a magnet. The recycled $\text{Fe}_3\text{O}_4@\text{Nico}@\text{Ag}$ was washed with water and ethanol and then dried for the next cycle. As shown in Fig. 5(b), the catalyst activity did not show any significant change for the 4-NP and 4-NA reduction reactions up to three cycles.

4. Conclusions

A novel magnetic recoverable $\text{Fe}_3\text{O}_4@\text{Nico}@\text{Ag}$ nanocatalyst was synthesized using a combination of reflux and a chemical reduction method. The $\text{Fe}_3\text{O}_4@\text{Nico}@\text{Ag}$ magnetic nanocatalyst was a good catalyst for the reduction of 4-NP to 4-AP and 4-NA to dinitrobenzene at room temperature in the presence of NaBH_4 as the reducing agent. The reduction reaction followed pseudo-first order kinetics. The $\text{Fe}_3\text{O}_4@\text{Nico}@\text{Ag}$ magnetic nanocatalyst exhibited good stability. Its activity showed no significant decrease after reuse for 4 cycles. Therefore this catalyst can be used in wastewater treatment and the conversion of 4-NP to 4-AP in aqueous solution under mild condition.

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Graphical Abstract

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A Fe₃O₄@Nico@Ag nanocatalyst for the hydrogenation of nitroaromatics

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This article reported the synthesis of a magnetic recyclable Fe₃O₄@Nico@Ag nanocatalyst and its use for the hydrogenation of 4-nitrophenol and 4-nitroaniline. The catalyst can be used efficiently for the hydrogenation of nitroaromatics.

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