Magnetic Recyclable Cu/ZnFe$_2$O$_4$ for Catalytic Reduction of Nitroarenes and C-N Bond Formation Reactions

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Abstract
A magnetic recyclable Cu/ZnFe$_2$O$_4$ composite material was successfully prepared with easy-to-obtain raw materials. This obtained copper composite material could be employed as efficient catalyst for both degradation of aromatic nitro compounds and C-N bond formation reactions without extra ligands. The degradation reaction could be accomplished in 4 min with high selectivity. A variety of N-aryl nitrogen-containing heterocycles products could be achieved in good to excellent yields with Cu/ZnFe$_2$O$_4$ as the reusable catalyst and this environmentally friendly heterogeneous catalyst could simply be recovered via magnetic decantation and used again for six cycles without considerable loss in activity. Besides, the morphology, chemical state and magnetic property of Cu/ZnFe$_2$O$_4$ was systematically characterized with FT-IR, TGA, XRD, EDS, SEM, TEM, XPS, ICP-OES and VSM studies.

Graphical Abstract

A magnetic recyclable Cu/ZnFe$_2$O$_4$ composite material has been prepared as efficient catalyst for both degradation of aromatic nitro compounds and C-N bond formation reactions without extra ligands.

Keywords Zinc ferrite composite materials · Magnetic recyclable materials · Copper catalysis · Heterogeneous catalysis · C-N bond formation reactions

1 Introduction

Aromatic nitro compounds which are released from organic or textile industries, have been threatening both environmental safety and human health in recent years. Among them, the p-nitrophenol (p-NP) has been listed as priority toxic pollutants and hazardous wastes by United States Environmental Protection Agency [1, 2]. Therefore, removal or degradation of such toxic compounds from waste water is of great significance. Till date, several methods including
electrochemical oxidation [3], adsorption [4], biodegradation [5], and photocatalysis degradation [6] have been developed and applied to this research field. In particular, the catalytic reduction of nitroaromatic compounds to the corresponding amino compounds is considered one of the most efficient methods because the generated amino compounds are important intermediates in the preparation of various useful products such as dyes, agricultural and pharmaceutical products [7].

The N-aryl nitrogen-containing heterocycles are valuable compounds that are present in numerous bioactive natural products, synthetic drugs, and materials [8]. Accordingly, the prevalence of these compounds has expedited the exploration of efficient and environmentally benign synthetic methods for their preparation. In this context, the copper-catalyzed Ullmann-type C-N bond formation reactions [9] that enable the coupling of aryl halides with nitrogen-containing compounds representing a straightforward, inexpensive approach to such compounds and have received high priority in synthetic community [10]. However, this copper-catalyzed synthesis usually confronts with some restrictions such as harsh conditions, stoichiometric dosage of copper, limited substrate scope, and reasonable to poor yields [11]. To overcome these drawbacks, researchers have employed several organic ligands and copper salts to facilitate this transformation [12]. In spite of the efficiency of these homogenous catalyst systems, due to the unre cyclable of the metal catalyst and the valuable ligands, together with the separation problems of the products expedited the fabrication of copper based heterogeneous catalysts for the C-N bond formation reaction. Anchoring appropriate ligands and copper to the surface of specially developed supports is a viable strategy for the development of heterogeneous catalysts [13]. Several systems have been disclosed based on a series of recyclable supporting materials, such as hydroxyapatites [14], silica-based materials [15], carbon nanotube materials [16], metal oxide materials [17] and polymers [18]. Despite much progress having been achieved in this research field, a great number of problems still remain unsettled, such as the extra synthesis steps for introduction of the ligands, agglomerate problems during the reaction process, reduced activity and reusability caused by the poor stability of the catalyst [19]. Therefore, the development of more efficient and easily available heterogeneous catalysts for the Ullmann-type C-N bond formation reactions is still highly desirable.

Zinc ferrite (ZnFe$_2$O$_4$) possesses large saturation magnetization and low coercivity which lead to its huge application prospects as magnetic recyclable materials [20–22]. Magnetic recyclable catalysts based on zinc ferrite avoiding filtering or centrifugal separation procedures would be a reliable solution for the separation problems in terms of heterogeneous Ullmann-type C-N bond formation reactions. However, the fabrication of available zinc ferrite-supported copper catalyst (Cu/ZnFe$_2$O$_4$) should settle the following intrinsic obstacles: (1) the difficulty of possessing both high catalytic activity and good magnetic properties because of the fact that the catalytic active copper is prone to be wrapped by the surrounding catalytically inactive magnetic materials; (2) the stability of the materials under reaction conditions, such as high temperature, pH, organic corrosion and so on; (3) issues associated with the catalytic activities and catalytic efficiency. Considering the importance of developing efficient heterogeneous catalysts for degradation of aromatic nitro compounds and C-N bond formation reactions, together with our continuous investigation on the application and development of magnetically recoverable materials [23], herein we wish to report a novel Cu/ZnFe$_2$O$_4$ composite material as efficient catalyst for both of the above mention reactions. This developed composite features easy availability, high catalytic performance, and good reusability via a simple magnetic separation method.

2 Experimental

2.1 Synthesis of Cu/ZnFe$_2$O$_4$ Composite Catalyst

The solution of Cu(NO$_3$)$_2$ (67 mmol) dissolved in 100 mL diethanolamine was heated at 150 °C and stirred vigorously for 3 h in nitrogen atmosphere. The color of the mixture turned red as the reaction proceeds. After cool down to room temperature, copper precipitates were obtained by centrifugation [24]. Then, a solution of FeCl$_3$ (9.5 mmol, 1.504 g) and ZnCl$_2$ (4.4 mmol, 0.600 g) in 100 mL deionized water was sonicated for 3 h at room temperature. Then, NaOH (3 M, 30 mL) was added into the mixture. And then the temperature of the ultrasonic water bath was raised to 60 °C for 1 h, finally magnetic suction out ZnFe$_2$O$_4$ [25, 26]. The obtained ZnFe$_2$O$_4$ was dispersed in 100 mL deionized water, and a mixture of the above obtained coppery precipitate in 50 mL ethanol was added. And the resulting mixture was heated at 80 °C and agitated for 50 min. The solid precipitate was then separated with a magnetic bar and washed with distilled water and anhydrous ethanol. Then the Cu/ZnFe$_2$O$_4$ composite was obtained after drying at 160 °C for 2 h.

2.2 Cu/ZnFe$_2$O$_4$ Catalyzed Reduction of p-nitrophenol

p-Nitrophenol (2 mL, 400 ppm in deionized water), freshly prepared NaBH$_4$ solution (2 mg/mL) and Cu/ZnFe$_2$O$_4$ (3 mg, 5 mg, 7 mg, 10 mg) were added to a quartz cuvette, and the reaction was monitored with an ultraviolet–visible spectrophotometer with a wavelength of 200–600 nm.
2.3 Cu/ZnFe$_2$O$_4$ Catalyzed C-N Bond Forming Reactions

A mixture of Cu/ZnFe$_2$O$_4$ (100 mg, 4.8 mol%) and Cs$_2$CO$_3$ (0.6500 g, 2 mmol) was added to the re-sealable test tubes (25 mL) with Teflon septa. The tube was emptied and filled with nitrogen, then the process was repeated three times. EtOH (1.0 mL), 3a (1.5 mmol, 0.3060 g) and 4a (1.0 mmol, 0.0681 g) were added through a syringe under N$_2$, afterwards sealed the reaction tube. Heated the mixture at 120 °C for 12 h, and cool down to room temperature. The organic phase was extracted by CH$_2$Cl$_2$, then dried on anhydrous magnesium sulfate and concentrated by rotary evaporation. Finally, the crude product was purified by column chromatography (ethyl acetate: petroleum ether = 2:1) to afford 5a (0.1356 g, 94% yield).

3 Results and Discussion

3.1 Characterization of the Cu/ZnFe$_2$O$_4$ Catalyst

The Cu/ZnFe$_2$O$_4$ catalyst was synthesized by loading Cu species on zinc ferrite particles through ultrasound assisted co-precipitation. The structure of the fabricated zinc ferrite and Cu/ZnFe$_2$O$_4$ materials were characterized by FT-IR, XRD, TGA, EDS, TEM, SEM, XPS and ICP-OES.

As shown in Fig. 1a, the absorption peak at 556 cm$^{-1}$ is attributed to the Fe–O vibration from the magnetic
The peak near 3440 cm\(^{-1}\) is related to the O–H stretching vibration of coordinated the hydroxyl groups and water molecules in the layers. The peak around 1618 and 1383 cm\(^{-1}\) maybe caused by another absorption band corresponding to the water deformation \cite{28, 29}. The absorption peak at 618 cm\(^{-1}\) indicated the existence of copper nanoparticles \cite{30}. The TGA curves of Cu/ZnFe\(_{2}\)O\(_4\) and ZnFe\(_2\)O\(_4\) were shown in Fig. 1b. The Cu/ZnFe\(_2\)O\(_4\) and ZnFe\(_2\)O\(_4\) showed a mass loss of 7%, which was attributed to the loss of adsorbed water on the sample surface. Furthermore, the mass loss of Cu/ZnFe\(_2\)O\(_4\) and ZnFe\(_2\)O\(_4\) was 2%, which was due to the loss of crystalline water bound within the sample. The mass of Cu/ZnFe\(_2\)O\(_4\) catalyst did not decrease significantly at 700 °C, which indicated that the Cu/ZnFe\(_2\)O\(_4\) catalyst had good thermal stability. The Fig. 1c showed the XRD patterns of Cu/ZnFe\(_2\)O\(_4\), Cu (PDF#03–1015) and ZnFe\(_2\)O\(_4\) (PDF#01–1108). The position and relative intensity of the position and relative intensity of the peaks in the Cu/ZnFe\(_2\)O\(_4\) of XRD pattern were consistent with the standard XRD pattern of ZnFe\(_2\)O\(_4\) \cite{31} and Cu \cite{32}, indicating that the crystal structure of ZnFe\(_2\)O\(_4\) was maintained during Cu loading processes. As shown in Fig. 1d the elemental composition was determined by EDS analysis and the results indicated the existence of O, Cu, Zn and Fe elements and further confirmed the structure of Cu/ZnFe\(_2\)O\(_4\). The SEM and TEM images of the obtained catalysts were showed in Fig. 1e, f, g, h. As shown, Cu/ZnFe\(_2\)O\(_4\) and ZnFe\(_2\)O\(_4\) were still irregular shape and nearly uniform size. The surface of ZnFe\(_2\)O\(_4\) is changed after the introduction of Cu nanoparticles, and needle-like Cu single crystals are loaded on the original smooth surface. In addition, the valence and content of copper were further characterized by XPS and TCP-OES. The bands at the binding energy of 932.6 eV and 952.2 eV belong to Cu\(_{2p3/2}\) and Cu\(_{2p1/2}\) respectively, confirming the copper valence of bulk Cu. The characteristic peak of Cu\(^{2+}\) is due to the oxidation of copper surface by air \cite{33}. (Please refer the supporting information, Figure S4).

### 3.2 Degradation of Aromatic Nitro Compounds with Cu/ZnFe\(_2\)O\(_4\)

After investigation of the morphology of Cu/ZnFe\(_2\)O\(_4\), we continue to study the catalytic performance of Cu/ZnFe\(_2\)O\(_4\). The catalytic activity of Cu/ZnFe\(_2\)O\(_4\) in the reduction process of p-nitrophenol to p-aminophenol was investigated with NaBH\(_4\) as the reducing agent. The decrease in peak intensity at 400 nm is due to the reduction of p-nitrophenol (Fig. 2). At this time, the stability of the peak intensity also indicates the proceeding of the reaction. Figure 2a depicted the UV spectra of the reduction of p-nitrophenol in terms of different reaction time. Figure 2b and c showed the kinetic experiments of the reduction reactions. The kinetic equation of the reduction reaction can be described as follows:

\[
\ln\left(\frac{C_t}{C_o}\right) = kt
\]

The \(C_t\) is concentration of p-nitrophenol during the reaction, and \(C_o\) is concentration of p-nitrophenol at the beginning of the reaction. When the catalyst dosage is increased from 3 to 10 mg, the k value increased from \(1.31 \times 10^{-2}/s\) to \(4.02 \times 10^{-2}/s\), the time required for the complete reduction of p-nitrophenol also decreased from 330 to 120 s. As the concentration increases, the reaction rate also increases. The rate of reaction depends on concentration of p-nitrophenol, which conforms to the characteristics of a quasi-first order reaction. These studies demonstrated that in the reduction of p-nitrophenol, the Cu/ZnFe\(_2\)O\(_4\) exhibited good catalytic activity.

Subsequently, in order to further explore the application of this catalyst in the reduction of aromatic nitro compounds, an array of substrate scope experiments was carried out under the optimum reaction conditions. As shown in Fig. 2d, all of the investigated nitro aromatic compounds are reduced to the corresponding amino compounds with high yields (Supplementary data, Table S1). A series of functional groups were well tolerated including halogen, hydroxyl, carboxyl, alkoxy groups. Notably, both of the two nitro groups of 1,3-dinitrobenzene substrate could be reduced to amino groups in this transformation in 98% yield.

### 3.3 Cu/ZnFe\(_2\)O\(_4\) Catalyzed C-N Bond Formation Reactions

#### 3.3.1 Optimization of C-N Bond Formation Reaction Conditions

To further estimate the application possibility of Cu/ZnFe\(_2\)O\(_4\) in catalysis, the C-N bond formation reactions were further investigated with iodobenzene (3a) and imidazole (4a) as the model substrates. Table 1 shows the reaction condition optimization studies. Generally, the nitrogen-based ligands play significant roles in the interaction with metal centers and affect the efficiency of the catalyst system in the C-N bond formation reactions \cite{34}. Therefore, we firstly screened some commercially available ligands on the reactivity, including 1,10-phenanthroline, p-toluenethiol, thiourea, and proline (entries 1–4). To our delight, we found that the reaction proceeded well without any extra ligand and the yield reached 94% with Cs\(_2\)CO\(_3\) as the base in ethanol at 120 °C (entry 5). Disappointingly, further screening the base failed to elevate the reaction yield, including K\(_2\)CO\(_3\), NaOH, and KOH (entries 6–8). Increasing the catalyst dosage (entry 10), elevating the reaction temperature (entry 11), and prolonging the reaction time (entry 12) also failed to increase the yield. What’s more, decreasing the temperature (entry 13) or shortening the reaction time (entry 14) the
reactivity deteriorated dramatically. In addition, altering the ratio of $3a$ and $4a$ had almost no influence on the reactivity (entries 15–16). Moreover, the reaction could not occur in the absence of the catalyst (entry 17). Hence the optimal conditions for this reaction were as follows: 100 mg of Cu/ZnFe$_2$O$_4$, 120 °C, 1.5 equiv. iodobenzene and 2.0 equiv. of Cs$_2$CO$_3$ in ethanol for 12 h.

3.3.2 Substrate Scope and Recyclability Investigations

Under the optimal conditions, the substrate scope of aryl halides and nucleophilic reagents were investigated and the results were shown in Table 2. In general, various aryl halides were transformed into the coupling products with 50–94% yields. The functional groups including methyl, methoxy, nitro, cyanide, bromine and trifluoromethyl were tolerated in this reaction. Then the steric effects were investigated via the exploration of substrates bearing methyl group at different position of aryl iodides (entries 2–4). Results demonstrated that the reaction proceeded mildly and delivered the products with high yields. Subsequently, the electronic effects of p-phenyl substituents were conducted (entries 5–9). Substrates bearing both electron-donating groups and electron-withdrawing groups could be converted to the products in 83–89% yield. Notably, when subjected 1-bromo-4-iodobenzene to this catalytic reaction, the iodine was substituted instead of bromine and delivering the corresponding 1-(4-bromophenyl)-1H-imidazole product 5f which could be further transformed to other useful organics through coupling reactions (entry 6). Interestingly, the 1,4-di(1H-imidazol-1-yl)benzene could be furnished when increased the dosage of imidazole to 2.0 equivalent. What’s more, other nucleophiles such as (benzo)imidazoles, benzotriazoles, pyrazols, and pyrroles were also tolerated in this reaction resulting in the corresponding products with high yields (entries 10–15). In addition, transformations with arylamines and cyclic amines, such as 1-phenylpiperazine, morpholine, and piperidine were also viable and furnished the products in 60–94% yields (entries 14–20). In order to further explore the substrate scope, several aryl bromides were also employed to this transformation and all of the reactions were happened stably in 50–74% yields (entries
Disappointingly, reaction with aryl chloride as the coupling agent generated the product with only 10% of yield (entry 28).

Since one of the key properties of our catalysts is the magnetic response, the recovery and reuse of catalysts were worth focusing on. We are pleased to find that the catalysts could be quickly and easily recovered from the reaction system with an external magnetic bar. The results showed that the catalyst could be recovered quickly and easily in the reaction system and reused continuously for 6 times without obvious loss of activity (Fig. 3a). Moreover, we found that Cu/ZnFe$_2$O$_4$ presented good magnetic properties through the vibrating sample magnetometer studies (VSM). It could be seen from the VSM study that the synthesized Cu/ZnFe$_2$O$_4$ is ferromagnetic material and presented strong magnetism albeit slightly lower than ZnFe$_2$O$_4$, with a magnetic moment up to 15 emu/g (Fig. 3b). In order to investigate the stability of this material, several characterization studies of the reused catalyst were conducted, including FT-IR, TGA, XRD, SEM, and TEM (SI, Figure S1-S3). All these results were consistent with the data of unused Cu/ZnFe$_2$O$_4$, thus demonstrating that the surface and internal structure of this catalyst stayed intact after 6 times of reuse, proving its good stability.

According to the literature report [35], a conceivable mechanism of this C-N coupling reaction was illustrated in Fig. 4. Firstly, the aryl halides reacted with the copper center decorated on the surface of Cu/ZnFe$_2$O$_4$ catalyst generating the corresponding oxidative adducts A. Then, the heterocyclic substrates replaced the halide ions to

\[
\begin{align*}
\text{Entry} & \quad \text{Base} \quad \text{Cu/Fe$_2$O$_4$ (mg)} \quad \text{Temp. (°C)} \quad \text{Time (h)} \quad \text{Yield (%)} \\
1^c & \quad \text{Cs$_2$CO$_3$} \quad 100 \quad 120 \quad 12 \quad 93 \\
2^d & \quad \text{Cs$_2$CO$_3$} \quad 100 \quad 120 \quad 12 \quad 94 \\
3^e & \quad \text{Cs$_2$CO$_3$} \quad 100 \quad 120 \quad 12 \quad 94 \\
4^f & \quad \text{Cs$_2$CO$_3$} \quad 100 \quad 120 \quad 12 \quad 70 \\
5 & \quad \text{Cs$_2$CO$_3$} \quad 100 \quad 120 \quad 12 \quad 94 \\
6 & \quad \text{K$_2$CO$_3$} \quad 100 \quad 120 \quad 12 \quad 21 \\
7 & \quad \text{NaOH} \quad 100 \quad 120 \quad 12 \quad 23 \\
8 & \quad \text{KOH} \quad 100 \quad 120 \quad 12 \quad 20 \\
9 & \quad \text{Cs$_2$CO$_3$} \quad 75 \quad 120 \quad 12 \quad 75 \\
10 & \quad \text{Cs$_2$CO$_3$} \quad 125 \quad 120 \quad 12 \quad 94 \\
11 & \quad \text{Cs$_2$CO$_3$} \quad 100 \quad 130 \quad 12 \quad 94 \\
12 & \quad \text{Cs$_2$CO$_3$} \quad 100 \quad 120 \quad 15 \quad 94 \\
13 & \quad \text{Cs$_2$CO$_3$} \quad 100 \quad 110 \quad 12 \quad 47 \\
14 & \quad \text{Cs$_2$CO$_3$} \quad 100 \quad 120 \quad 9 \quad 88 \\
15^g & \quad \text{Cs$_2$CO$_3$} \quad 100 \quad 120 \quad 12 \quad 92 \\
16^h & \quad \text{Cs$_2$CO$_3$} \quad 100 \quad 120 \quad 12 \quad 94 \\
17 & \quad \text{Cs$_2$CO$_3$} \quad 0 \quad 120 \quad 12 \quad - \\
\end{align*}
\]

\(^a\)Reaction conditions: 3a (1.5 mmol), 4a (1.0 mmol), Cs$_2$CO$_3$ (2.0 mmol), EtOH (1.0 mL) in N$_2$

\(^b\)Isolated yields

\(^c\)1,10-phenanthroline (0.5 mmol)

\(^d\)p-toluenethiol (0.5 mmol)

\(^e\)thioanisole (0.5 mmol)

\(^f\)proline (0.5 mmol)

\(^g\)3a (1.0 mmol)

\(^h\)3a (2.0 mmol)
deliver the intermediate B and kicked out one molecular of hydrogen halide (HX). Finally, the coupling product could be obtained through the reductive elimination processes around the copper center and regenerated the catalyst to complete the catalytic cycle.

3.3.3 Catalytic Performances Comparison for Cu/ZnFe₂O₄ with Various Catalysts

The catalytic performance of aromatic nitro compounds degradation and C-N bond formation reactions were
further evaluated through comparison with other previously reported catalysts, and the results are listed in Tables 3. By comparison with the catalysts previously reported, we can see that the Cu/ZnFe₂O₄ catalyst can promote the reaction completed in 2 min with a dosage of only 10 mg showing high efficiency in the degradation of aromatic nitro compounds. Besides, through comparison of the catalytic performance in the Cu-based heterogeneous catalyzed C-N bond formation reactions, it could be concluded that the Cu/ZnFe₂O₄ catalyst performed good catalytic activity with a broad substrate scope.

### 4 Conclusions

To sum up, a magnetic recoverable Cu/ZnFe₂O₄ composite material was successfully prepared and well characterized. This copper composite material performed well in the reduction of nitroaromatics to amino aromatics with sodium borohydride as the reductant. For the Ullmann-type C-N bond formation reactions, Cu/ZnFe₂O₄ could also exhibit good catalytic activity and delivering the corresponding N-aryl nitrogen-containing heterocycles products with good to excellent yields. What’s more, this heterogeneous catalyst could be easily separated and reused for several consecutive times with a magnetic bar without significant loss of their catalytic efficiency. Further investigations on applications of this magnetic recyclable copper composite material are underway in our laboratory.
Table 2 (continued)

|   |   |   |   |
|---|---|---|---|
| 5a | 5b | 5c | 5d |
| 50 | 70 | 72 | 61 |
| 5e | 5f | 5g | 5h |
| 74 | 63 | 62 | 5a |

| 5m | 5a |
|---|---|
| 74 | 10 |

*Reaction conditions: 3 (1.5 mmol), 4 (1.0 mmol), Cs₂CO₃ (2.0 mmol), Cu/ZnFe₂O₄ (100 mg, 4.8 mol%), EtOH (1.0 mL), 120 °C under N₂ for 12 h

*Isolated yields

*When 2.0 equiv. of imidazole was employed, the 1,4-di(1H-imidazol-1-yl)benzene could be achieved in 65% yield.

Fig. 3  a Recyclability of the Cu/ZnFe₂O₄ catalyst. b VSM study of ZnFe₂O₄ and Cu/ZnFe₂O₄
Magnetic Recyclable Cu/ZnFe₂O₄ for Catalytic Reduction of Nitroarenes and C-N Bond…

Supplementary Information

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Declarations

Conflict to interest The authors declare that they have no conflict to interest.

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