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Original Research

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Aromatic Sulfonamides: S-N bond formation using MNPs-Benzo[d]imidazole-Cu Magnetic Catalyst

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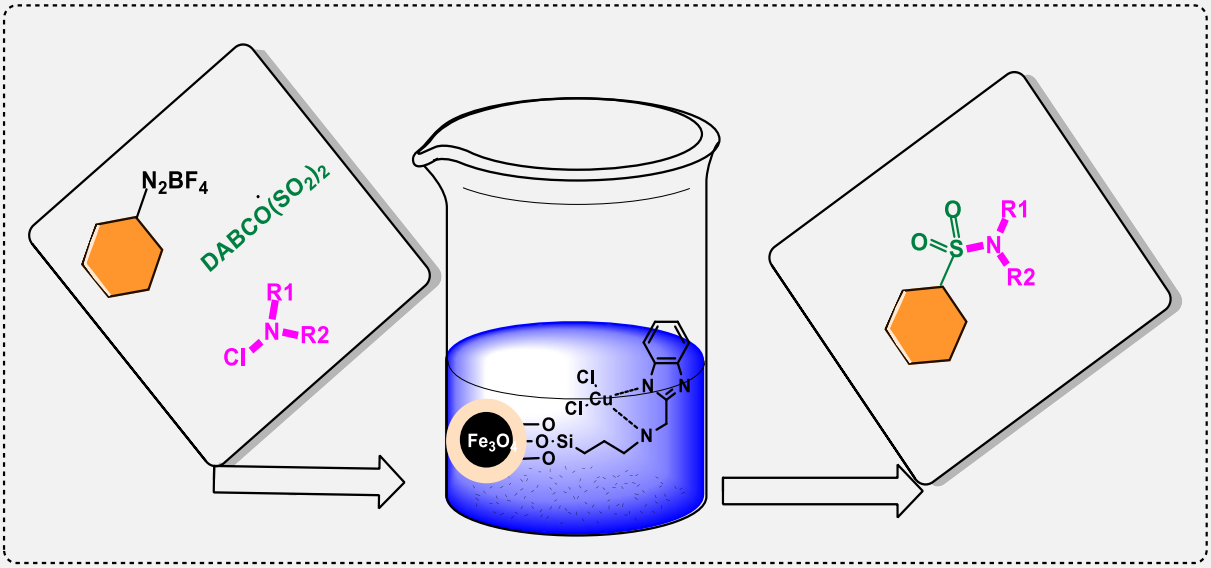
ABSTRACT: A sulfonamide is an important scaffold in medicinal and synthetic organic chemistry, as it forms the basis of many sulfa drugs. Synthesis of sulfonamide has recently been made possible by the development of new methodologies. An aminosulfonylation of copper using aryldiazonium tetrafluoroborate, DABCO·$(SO_2)_2$, and N-chloroamines is described in this paper. Under mild conditions, this coupled reaction provides a simple and efficient method for preparing a wide range of sulfonamides. Based on a mechanistic investigation, this back-to-back reaction integrates radical catalysis and transition metal catalysis. The structure of MNPs-Benzo[d]imidazol-Cu nanocatalyst is well analyzed by several spectroscopic techniques. High reusability and good characterization of MNPs-Benzo[d]imidazol-Cu nanocatalyst, high purity and yields of products, aqueous conditions, and simple operation are several considerable advantages of this catalytic system. A magnet can be used to separate the synthesized catalyst and reuse it for six consecutive cycles.



KEYWORDS: Sulfonamide, DABCO·$(SO_2)_2$, N-chloroamine, MNPs-Benzo[d]imidazol-Cu.

Introduction

There are several important structural motifs in compounds that contain S-N bonds, which play a crucial role in biological activities, pesticide activity, and material properties [1]. Pharmaceutically interesting molecules and biologically active compounds often contain sulfonamides, one of the most important functional groups in S-N compounds [2]. Modern pharmaceuticals and agrochemicals still contain the sulfonamide functional group, which was first introduced into medicines in the 1930s [3]. Since their introduction, sulfonamides have been widely used in drug development, but their fundamental principles

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J. Synth. Chem. 2023, 2, 227-239

have remained essentially unchanged since their introduction, because they rely on starting materials with pre-installed sulfur-functionalities [4]. An extensive spectrum of biological applications can be attributed to sulfonamides, a vital group of pharmaceutical compounds. Aside from this, they are useful intermediates for the synthesis of organic compounds [5]. A wide range of medicinal applications are available for sulfonamides with medicinal properties, such as anticonvulsants and HIV protease inhibitors. It is usually possible to produce sulfonamides through the reaction between sulfonyl chlorides and ester-derived amines [6]. While sulfonyl chlorides are relatively stable, they have some disadvantages, such as the difficulty of handling them and the inability to store them for long periods of time [7]. The instability of some of them makes them unavailable commercially [8]. N-aryl sulfonamides are commonly synthesized under diverse catalytic conditions using compounds such as aryl siloxanes, halides, boronic acids, etc. It is well-known that N-aryl sulfonamides can be synthesized using Cu-based catalysts [9]. High cost and toxicity are two of the most important problems associated with these catalysts [10]. In order to synthesize N-aryl sulfonamides, researchers have developed more efficient, cost-effective, low-toxicity, and environmentally friendly catalysts [11]. From various primary sulfonamide derivatives and boronic acids, copper-based catalysts have been extremely effective in synthesizing N-aryl sulfonamides. Catalysts based on copper offer a number of advantages, including low cost, high yield, and nontoxicity [12].

The primary advantage of heterogeneous catalysts over other catalytic systems is that they can be easily separated from reaction mixtures, which is the most important feature for researchers [13]. The choice of the right support for functionalizing metal nanoparticles (NPs) or complexes is therefore an important challenge in the preparation of heterogeneous catalysts [14,15]. By decorating homogenous organometallic complexes and metal or metal oxide nanoparticles on their surface, heterogeneous compounds can be applied as efficient supports for heterogeneous heterogeneous catalyst catalysts [16]. Graphene, natural and synthetic polymers, and other heterogeneous catalysts must be synthesized on a variety of suitable supports [17]. Aside from electronic devices and biomedicine, Fe_3O_4 NPs are also used as catalysts in different kinds of organic reactions because they are non-toxic and superparamagnetic, making them easy to separate from the reaction medium [18–20]. Currently, metal complexes are being explored for a wide range of applications, especially for catalysis [21]. Liquid ligands use nitrogen more widely than any other atom as an electron donor. A compound known as metformin (Met) is one of the most effective ligands available [22].

It is reported in this work that primary sulfonamides can be synthesized using aryl diazonium tetrafluoroborate, DABCO $(SO_2)_2$, and N-chloroamines. A magnetic heterogeneous catalyst was synthesized using MNPs-Benzo[d]imidazol-Cu as an efficient, new, and green support. In this regard,. As a new and magnetic catalyst, MNPs-Benzo[d]imidazol-Cu was synthesized using (1H-benzo[d]imidazol-2-yl)methanamine in ethanol for the N-arylation of primary sulfonamides.

■ Results and Discussion

Sulfonamides were prepared by synthesizing $CuCl_2$ on Fe_3O_4 magnetic nanoparticles supported by (1H-benzo[d]imidazol-2-yl)methanamine. Experimentally, MNPs-Benzo[d]imidazol-Cu nanocatalysts were fabricated as shown in **Scheme 1**. To form the final MNPs-Benzo[d]imidazol-Cu catalyst, (1H-benzo[d]imidazol-2-yl)methanamine was reacted with $CuCl_2$ after immobilization on magnetic nanoparticles. MNPs-Benzo[d]imidazol-Cu nanocomposites have been identified using different techniques.

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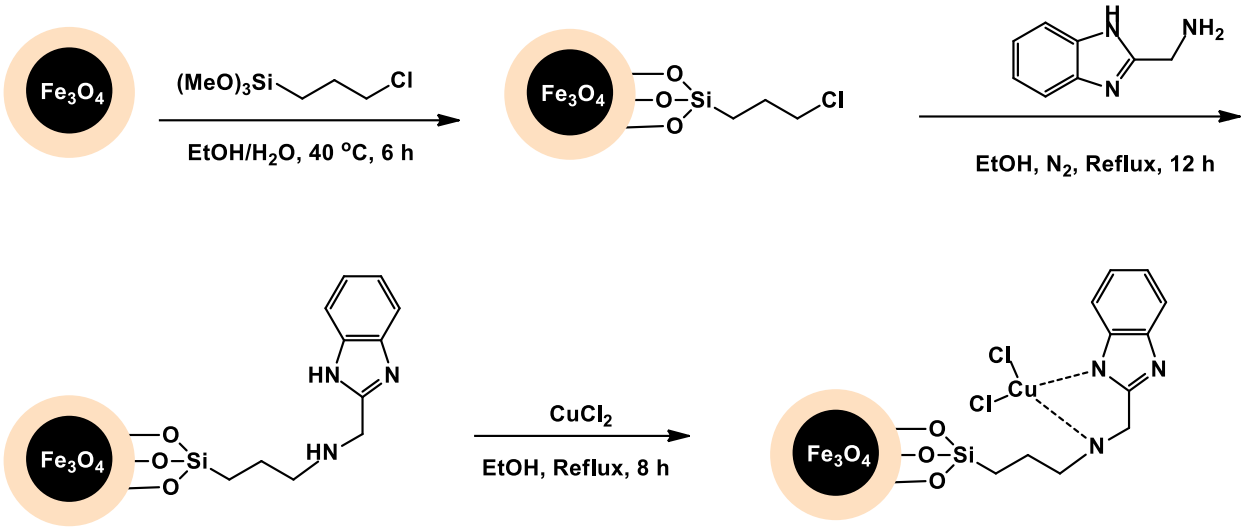
J. Synth. Chem. 2023, 2, 227-239



Scheme 1. An experimental analysis of the fabrication of MNPs-Benzo[d]imidazole-Cu nanocatalysts.

FT-IR spectroscopy

Figure 1 shows good agreement between the FT-IR spectroscopy of MNPs-Benzo[d]imidazol-Cu nanocatalyst and the optical measurements. FT-IR spectra of copper nanoparticles showed peaks at 3433 cm^{-1} which corresponds to stretching of H-O, 2922 cm^{-1} for stretching of C-H, and 1625 cm^{-1} for stretching of C-N. Copper nanoparticles show the greatest stretch between 571 and 560 cm^{-1}. Nanocatalyst of MNPs-Benzo[d]imidazol-Cu were thus synthesized.

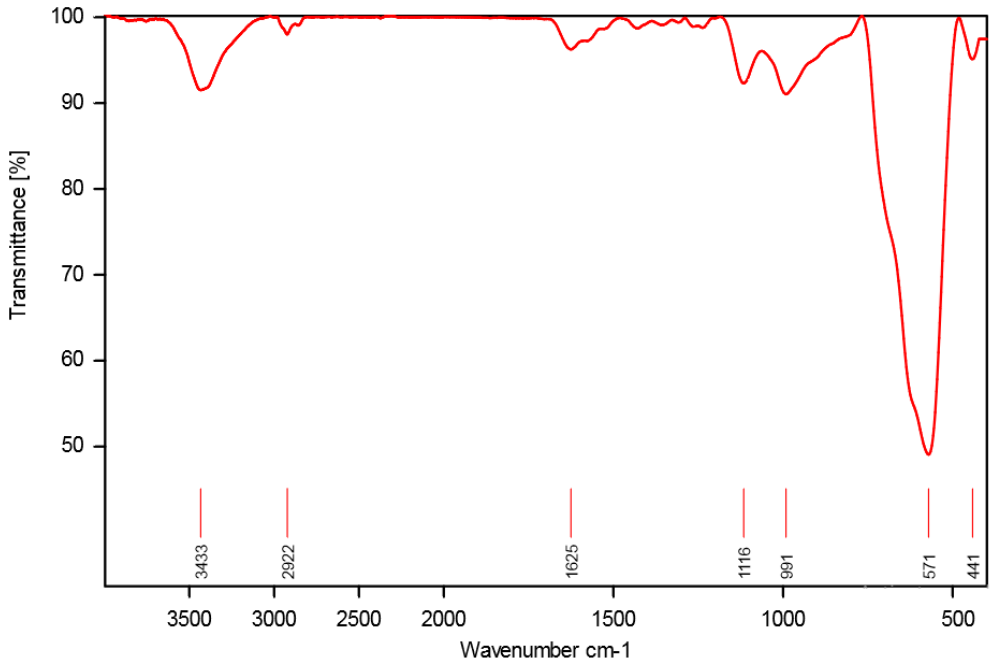


Figure 1. FT-IR spectroscopy of MNPs-Benzo[d]imidazol-Cu nanocatalyst.

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J. Synth. Chem. 2023, 2, 227-239

X – Ray diffraction (XRD) analysis

XRD spectroscopy was used to analyze the synthesized MNPs-Benzo[d]imidazol-Cu nanocatalyst. In **Figure 2**, MNPs-Benzo[d]imidazole-Cu nanocatalysts are shown in XRD patterns. It is evident from the X-ray diffraction pattern of the Cu sample level that it is highly crystallinized, with a distinct diffraction angle of 42.05°, 51.05° and 75.15° that correspond to the characteristic cubic face centers of copper nanoparticles indexed by (111), (200) and (220) respectively. A capping agent present in MNPs-Benzo[d]imidazol-Cu may stabilize copper nanoparticles, resulting in these sharp Bragg peaks. Generally, solids exhibit broadened peaks in XRD patterns due to particle size effects. Additionally, the broader peak reveals that experimental conditions affect the nucleation and growth of crystals. A wide range of literature has reported the size of Cu-NPs as 40-100nm. Based on the Debye-Scherrer equation, we estimate the size of synthesized Cu-NPs to be 55 nm based on their spherical shape and e(1H-benzo[d]imidazol-2-yl)methanamine synthesis. We found that (1H-benzo[d]imidazol-2-yl)methanamine is capable of synthesizing Cu-NPs with high surface areas and high surface areas to volume ratios.

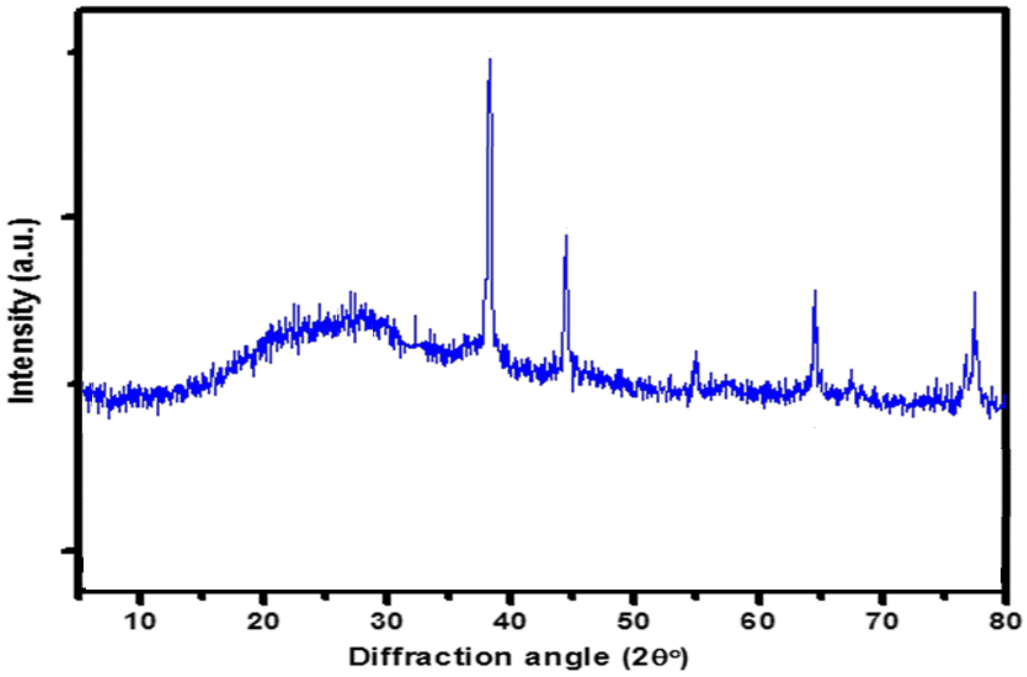

Figure 2. XRD graph of formed MNPs-Benzo[d]imidazole-Cu nanocatalysts.

ICP analysis was used to determine the Cu content in the nanocatalyst structure, which confirmed the amount of Cu immobilized on the Fe_3O_4 nanoparticles was 14.58× 10-5 mmol.g^{-1}.

The reaction of phenyldiazonium tetrafluoroborate, 1,2-dichloroethane (DCE), and Nchloromorpholine in EtOH/water was first investigated, but only small amounts of product 3a were detected by TLC (**Table 1, Entry 2**). In an interesting finding, when the reaction was carried out at 80 °C, 12% yield of the desired sulfonamide 3a was obtained. For this reaction, we employed a series of copper catalysts (**Table 1, entries 3–6**), inspired by copper-catalyzed amination reactions. As a result of using MNPs-Benzo[d]imidazole-Cu

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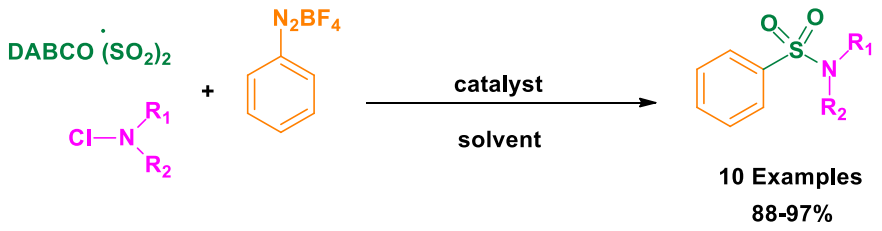
J. Synth. Chem. 2023, 2, 227-239

as a catalyst, the yield increased to 58% (**Table 1, entry 7**). Increasing the size of its mixture resulted in a much higher yield than lowering it (**Table 1, entry 8**), likely due to its dual function as a sulfonyl source and as a reductant. The next step was to screen solvents (**Table 1, entries 12-20**), but no better results were achieved. A lower temperature of 60 °C resulted in a 30% yield. In a sealed tube at 100 °C, a similar yield of 97% was obtained. Our next step was to investigate the scope of the aminosulfonylation reaction with optimized reaction conditions. There were several sulfonamides obtained in moderate to good yields, as shown in **Table 2**. Under the conditions, all aryldiazonium salts, no matter how electron-rich or electron-deficient, worked well. A variety of functional groups, including alkyl, ethers, haloes, ester groups, and even nitro groups, were tolerated during this transformation. Even substrates with ortho-substitutions or polysubstitutions could provide the desired sulfonamides, although at a lower yield.

Table 1. Experimental details for the standardized conditions for synthesis of **product 3a**.



Entry	Catalyst (mol%)	Temp (°C)	Solvent (°C)	Time (h)	Yield% [a]
1	No	80	EtOH	12	--
2	$CuCl_2$ (5 mg)	80	EtOH	1	12
3	CuOAc (5 mg)	80	EtOH	1	43
4	$CuBr_2$ (10 mg)	80	EtOH	1	59
5	CuCl(5mg)	80	EtOH	1	42
6	MNPs-Benzo[d]imidazole(20 mg)	80	EtOH	1	72
7	MNPs-Benzo[d]imidazole-Cu(5 mg)	100	EtOH	1	58
8	MNPs-Benzo[d]imidazole-Cu(10 mg)	100	EtOH	1	82
9	MNPs-Benzo[d]imidazole-Cu(15 mg)	100	EtOH	1	62
10	MNPs-Benzo[d]imidazole-Cu(20 mg)	100	EtOH	1	79
11	MNPs-Benzo[d]imidazole-Cu(20 mg)	60	PEG	1	30
12	MNPs-Benzo[d]imidazole-Cu(20 mg)	No	PEG	1	Trace
13	MNPs-Benzo[d]imidazole-Cu(20 mg)	100	DMSO	1	71
14	MNPs-Benzo[d]imidazole-Cu(20 mg)	100	H_2O	1	65

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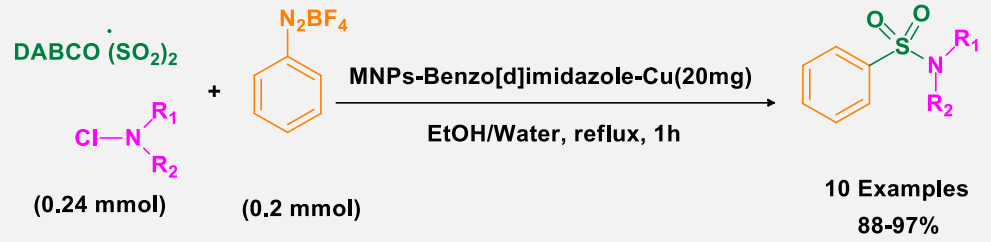
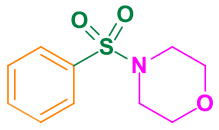
J. Synth. Chem. 2023, 2, 227-239

15	MNPs-Benzo[d]imidazole-Cu(20 mg)	**100**	**EtOH/water**	**1**	**97**
16	MNPs-Benzo[d]imidazole-Cu(20 mg)	100	Toluene	1	52
17	MNPs-Benzo[d]imidazole-Cu(20 mg)	100	CH_3CN	1	87
18	MNPs-Benzo[d]imidazole-Cu(20 mg)	100	THF	1	90
19	MNPs-Benzo[d]imidazole-Cu(20 mg)	100	Solvent-free	8	15
20	MNPs-Benzo[d]imidazole-Cu(20 mg)	100	DMF	1	71

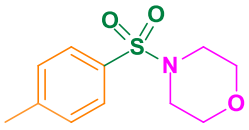
[a] Isolated Yields

Table 2. Synthesis of sulfonamides under the optimized conditions.

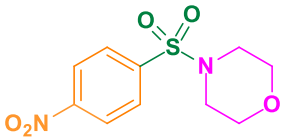




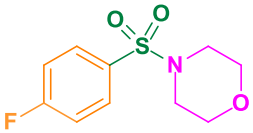
3a (97%)



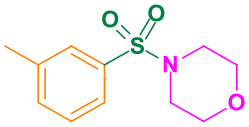
3b (94%)



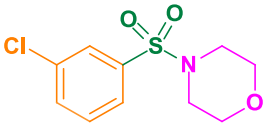
3c (92%)



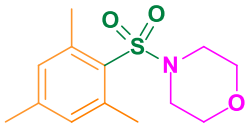
3d (91%)



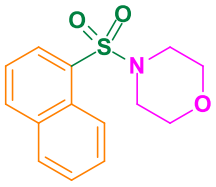
3e (85%)



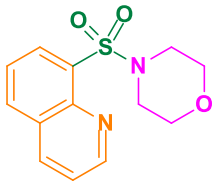
3f (86%)



3g (89%)



3h (90%)



3i (89%)

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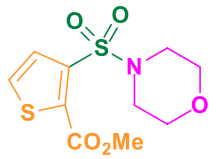
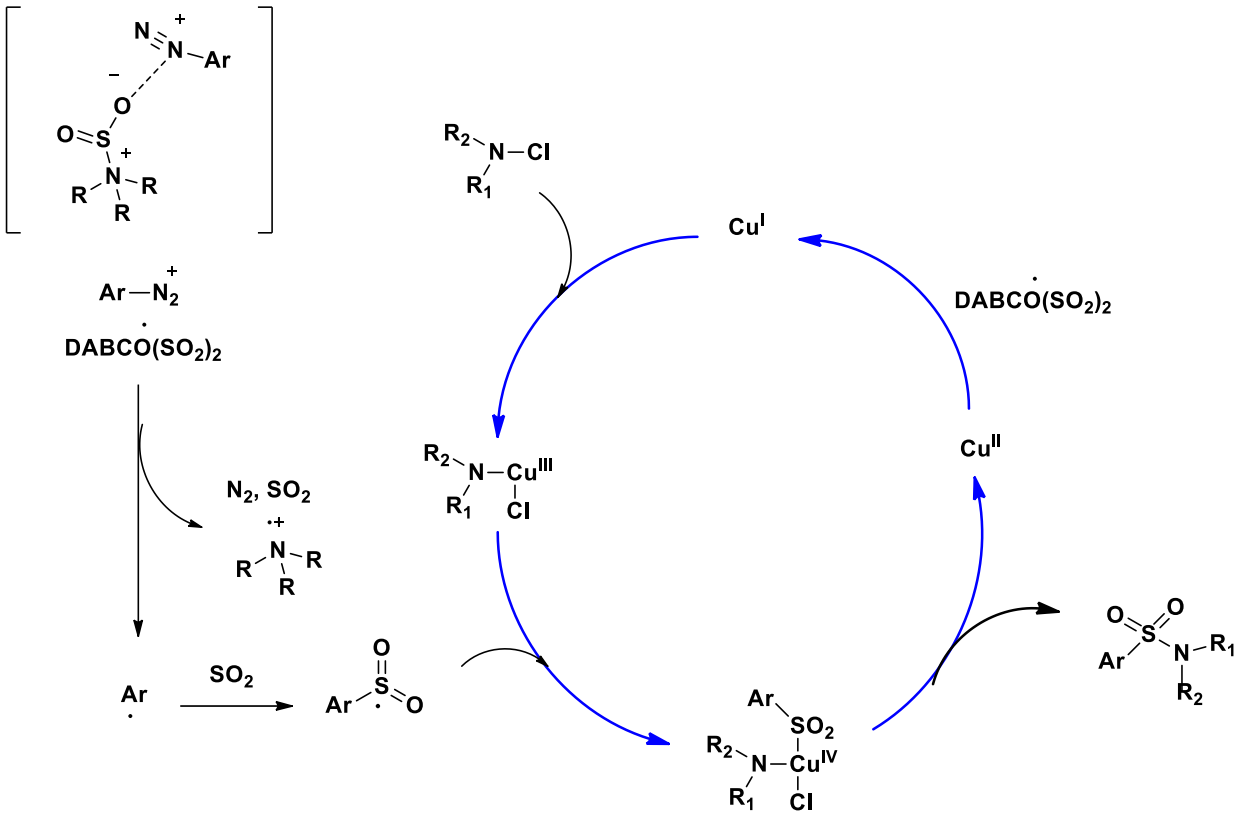
J. Synth. Chem. 2023, 2, 227-239



3j (91%)

[a] Isolated yields

As a result of the reported sulfonamide synthesis methods, we propose a plausible mechanism for the one-pot reaction between aryl diazonium tetrafluoroborate, DABCO $(SO_2)_2$, and N-chloroamines catalyzed by MNPs-Benzo[d]imidazole-Cu nanocomposite, as shown in **Scheme 2**.



Scheme 2. A Plausible mechanism for synthesis of sulfonamide catalyzed by MNPs-Benzo[d]imidazole-Cu.

Recyclability is essential to the usefulness of a heterogeneous catalyst. In addition to its high price, copper plays an important role in limiting catalysts. This limit can be reduced to some extent since copper can be recycled and reused several times. According to **Figure 3**, after six reuses of the same compound, less than one unit of reduction was achieved. The MNPs-Benzo[d]imidazole-Cu maintained high activity and selectivity for six consecutive cycles.

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J. Synth. Chem. 2023, 2, 227-239

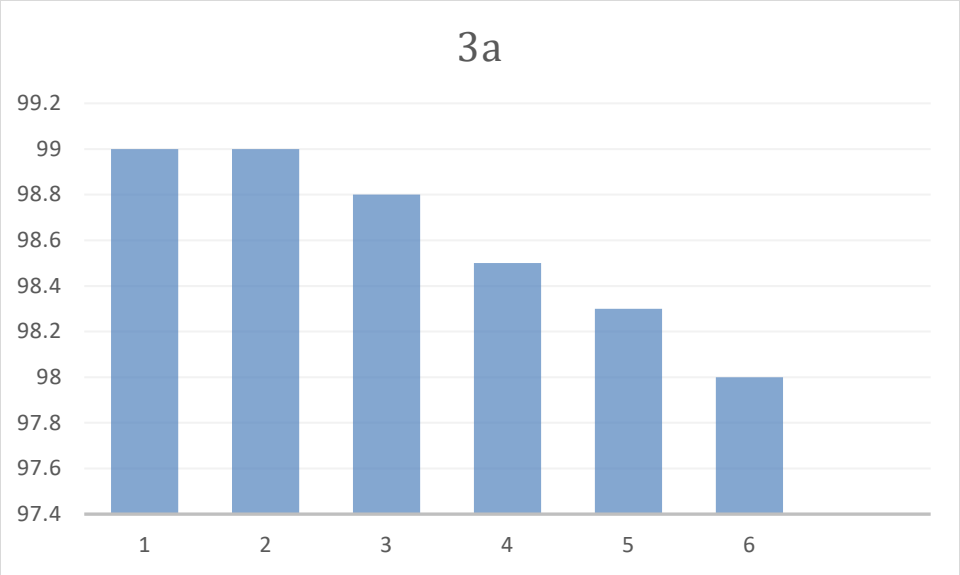

Figure 3. Reusability of MNPs-Benzo[d]imidazole-Cu catalyst in the synthesis of product 3a.

Conclusion

In summary, we have developed a three-component copper-catalyzed reaction for the synthesis of sulfonamides using aryldiazonium tetrafluoroborates, DABCO·$(SO_2)_2$, and N-chloroamines. In sulfur dioxide insertion reactions, N-chloroamines are used as amino sources, which take place under mild conditions and are compatible with a wide variety of substrates. It is also possible to use amines rather than N-chloroamines by in situ chlorination in one pot and two steps. There is a possibility that a radical process and transition-metal catalysis are involved in the process. As a result of this reaction, it can be concluded that a combination of metal catalysis and radical processes is a powerful strategy for this type of reaction.

Experimental

A complete set of starting materials was purchased from Merck and Aldrich Companies. The IR spectra were recorded using an RXI infrared spectrometer made by Perkin-Elmer. A Broucker FT-NMR spectrometer operating at 400 MHz was used to record 1H NMR spectra. Silica gel polygram SIGL/UV254 plates were used to monitor the purity of substrates and reactions using TLC.

Preparation of the Magnetic Fe_3O_4-Nanoparticles

Under N_2 atmosphere, $FeCl_3.6H_2O$ (5.838 g, 0.0216 mol) and $FeCl_2.4H_2O$ (2.147 g, 0.0108 mol) were dissolved in 100 ml of deionized water. In the following 30 minutes, 10mL of NH3 were added, followed by rapid mechanical stirring. It was then heated to 80°C for 30 minutes after stirring the black dispersion rapidly for 30 minutes. After obtaining a black precipitate, it was washed with distilled water until neutral, then twice with ethanol, then allowed to dry at room temperature.

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J. Synth. Chem. 2023, 2, 227-239

Preparation of the Fe_3O_4@SiO_2

The reaction mixture was sonicated for 30 minutes with Fe_3O_4 MNPs (2 g) then added 200 ml of 2-propanol. Mixtures were stirred at room temperature with magnetic stirrers. The suspension was continuously stirred for 36 hours with PEG (5.30 g), water (20 mL), ammonia solution (10 mL, 28 wt.%), and tetraethyl orthosilicate (TEOS). A magnet was used to isolate the product (Fe_3O_4@SiO_2), which was then washed twice with ethanol and distilled water.

Preparation of MNPs-CPTMS

Afterwards, 3-(trimethoxysilyl) propan-1-amine (2.5 mL) was added to 250 mL of ethanol/water (1:1) to disperse MNPs-CPTMS nanoparticles (1.5 g). For six hours, the reaction mixture was stirred under N_2 atmosphere at 40 ^oC. A magnetic decantation method was used to separate the nanoparticles after they were dispersed in ethanol using sonication five times. A room-temperature drying process was performed on the nanoparticle product (MNPs-CPTMS).

Preparation of MNPs-Benzo[d]imidazole

To the MNPs-CPTMS (1.3 g), 1H-benzo[d]imidazol-2-ylmethanamine was added (1.5 g) under N_2 atmosphere for 10 h. The prepared MNPs-Benzo[d]imidazole nanocomposite was separated by magnetic decantation, followed by three ethanol washes. After drying at room temperature, the product was packaged.

Preparation of the MNPs-Benzo[d]imidazole-Cu

A solution of MNPs-Benzo[d]imidazole (2.5 g) in absolute ethanol (50 mL) was added to a solution of $CuCl_2$ (5 mmol), which was stirred under reflux for eight hours. After synthesizing the nanosolid (MNPs-Benzo[d]imidazole-Cu), it was separated by magnetic decantation. At room temperature under vacuum, the nanomagnetic catalyst was washed and dried several times with absolute ethanol.

General Protocol for Synthesis of Sulfonamide

It was performed with phenyldiazonium tetrafluoroborate (0.2 mmol), DABCO $(SO_2)_2$, N-chloromorpholine (0.24 mmol), EtOH/H_2O (2.0 mL), under N_2 protection using MNPs-Benzo[d]imidazole-Cu catalyst (0.01 mmol). After that, it was magnetically stirred at 80°C. Through thin layer chromatography (TLC), the progress of the reaction was monitored. After that, 25 ml of CH_2Cl_2 was used to extract the reaction mixture. Over anhydrous magnesium sulfate, the materials were dried.

Supporting Information:

4-((3-Chlorophenyl)sulfonyl)morpholine (3f): 1H NMR (400 MHz, $CDCl_3$) δ 7.75 (s, 1H), 7.74-7.60 (m, 2H), 7.55 (t, J = 8.0Hz, 1H), 3.76 (t, J = 4.8 Hz, 4H), 3.04 (t, J = 4.8 Hz, 4H); 13C NMR (100 MHz, $CDCl_3$) δ 134.95, 132.64, 130.45, 127.29, 125.19, 65.91, 44.80.

4-(Mesitylsulfonyl)morpholine (3g) : 1H NMR (400 MHz, $CDCl_3$) δ 6.92 (s, 2H), 3.70 (t, J = 4.3 Hz, 4H), 3.15 (t, J = 4.3 Hz, 4H), 2.61 (s, 6H), 2.30 (s, 3H); 13C NMR (100 MHz, $CDCl_3$) δ 143.66, 140.38, 130.98, 128.79, 66.98, 43.75, 21.12, 20.89.

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J. Synth. Chem. 2023, 2, 227-239

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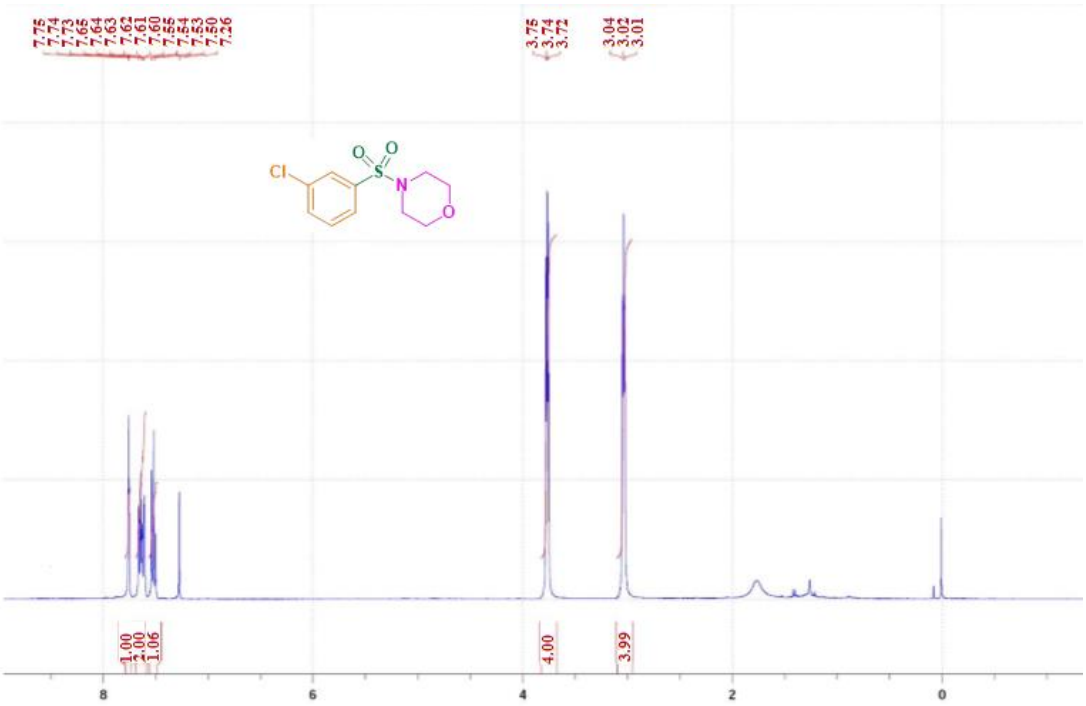

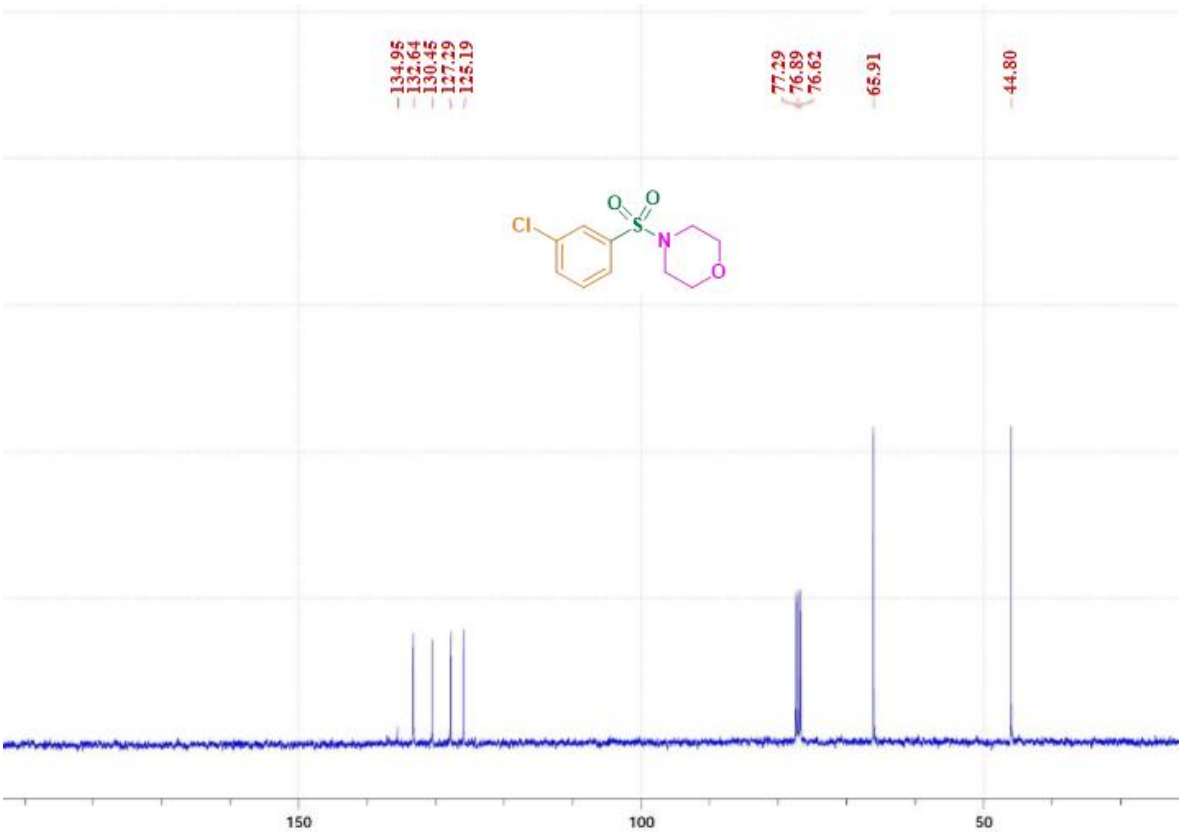

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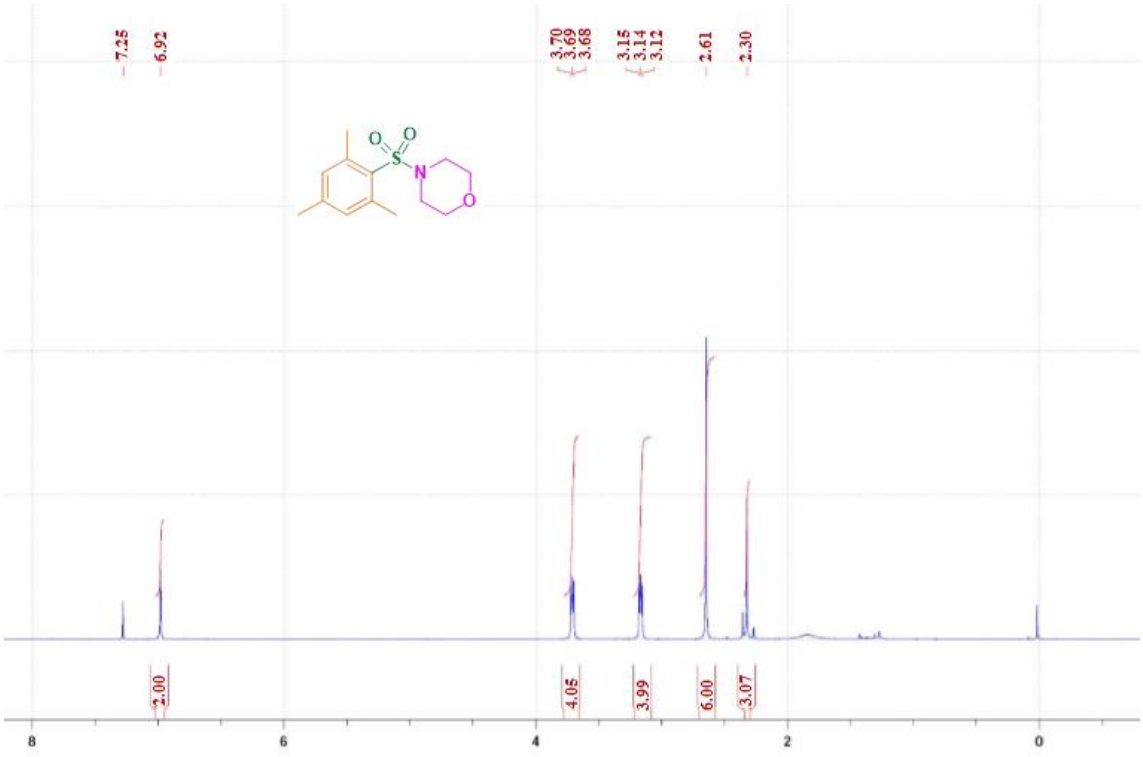
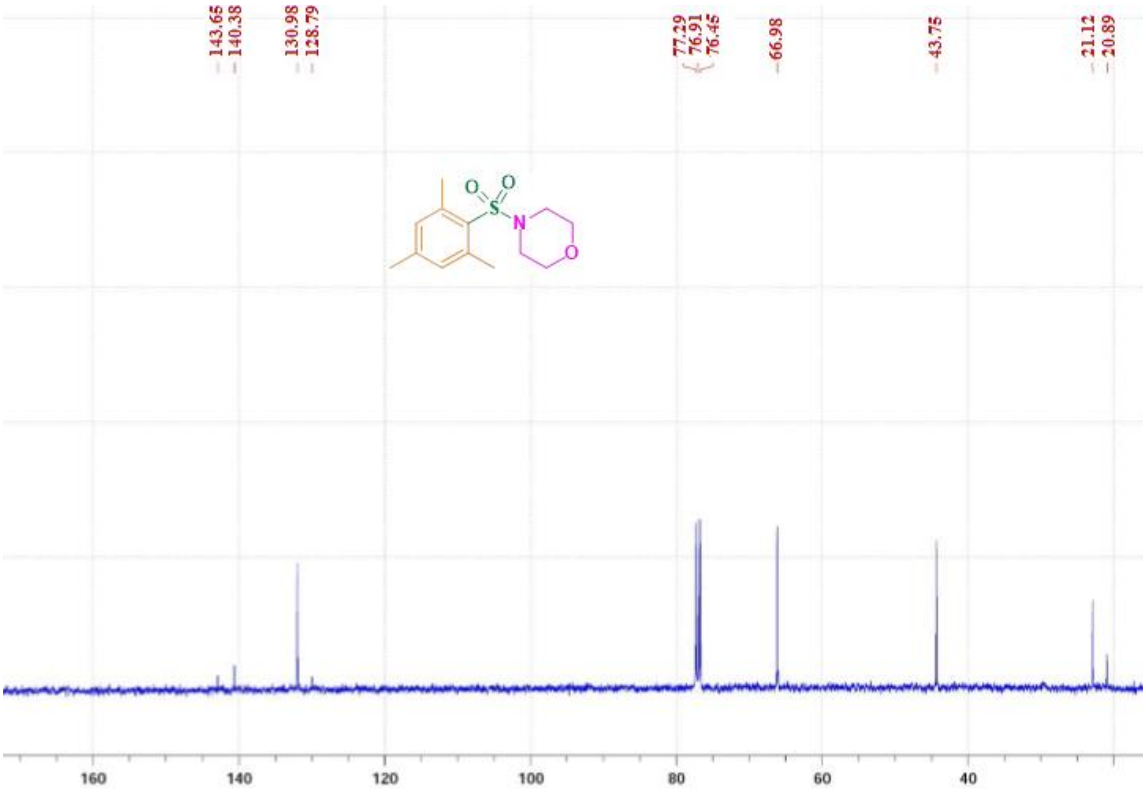
J. Synth. Chem. 2023, 2, 227-239





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J. Synth. Chem. 2023, 2, 227-239