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FULL PAPER

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KCC-1 aminopropyl-functionalized supported on iron oxide magnetic nanoparticles as a novel magnetic nanocatalyst for the green and efficient synthesis of sulfonamide derivatives

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A new magnetic nanocatalyst (Fe_3O_4@KCC-1-npr-NH_2) was synthesized directly through the reaction of Fe_3O_4@KCC-1 with (3-aminopropyl) triethoxysilane (APTES) using a hydrothermal protocol. Prepared nanocomposite was used as a magnetically reusable nanocatalyst for an efficient synthesis of a broad range of sulfonamide derivatives in water as a green solvent at room temperature and the products are collected by filtration with excellent yields (85–97%). The nanocatalyst could be remarkably recovered and reused after ten times without any significant decrease in activity. This mild and simple synthesis method offers some advantages including short reaction time, high yield and simple work-up procedure.

KEYWORDS

fibrous nano-silica, green synthesis, magnetic nano-catalyst, sulfonamides

1 | INTRODUCTION

Sulfonamides are well-known class of organic compounds found in numerous natural products, materials and pharmaceutical compounds which commonly employed as chemotherapeutic and preventive agents against several diseases (Figure 1).[1–3] Pharmaceutical and biological importance of sulfonamides are in the fields of the antibacterial, antifungal, antiinflammatory and anticonvulsant activities, as well as several enzymes inhibition including glycogen phosphorylase, carbonic anhydrase, serine protease, cholesterol acyltransferase, cyclooxygenase and matrix metalloproteinase.[4–9] In addition, it has been reported that sulfonamide derivatives act as a herbicide, dye[10,11] and plaguicides.[12] The conventional method and practical way for the synthesis of sulfonamide derivatives involves the reaction between sulfonyl chlorides and amine nucleophiles, using a base.[13] Also, one-pot reactions of monosubstituted or un-substituted sulfonamides using metal-catalyzed,[14,15] oxidation of sulfinimides or sulfonamides,[16,17] oxidative coupling reactions between an amine and a sulfonate salts have been developed.[18] These methods have some disadvantages including, non-environmentally friendly reagents, harsh conditions and limitation in scope.

Recently, metal oxides such as nano ZnO,[19] CuO[20] and CdO[21] have been used as effective heterogeneous catalysts in various sulfonylation reactions from amines. Despite the effective progress, there remains need to development of metal catalysts for the synthesis of sulfonamide compounds particularly using highly efficient heterogeneous catalysts. Although metal oxide catalysts display as both Lewis acid and base, surface area of metal oxide materials and nature of metal cation have widely employed to their catalytic properties.[22,23] Ferric oxide catalysts are cheap metal oxide which has been used as a common catalyst in numerous organic transformations.[14] Moreover, surface functionalized mesoporous

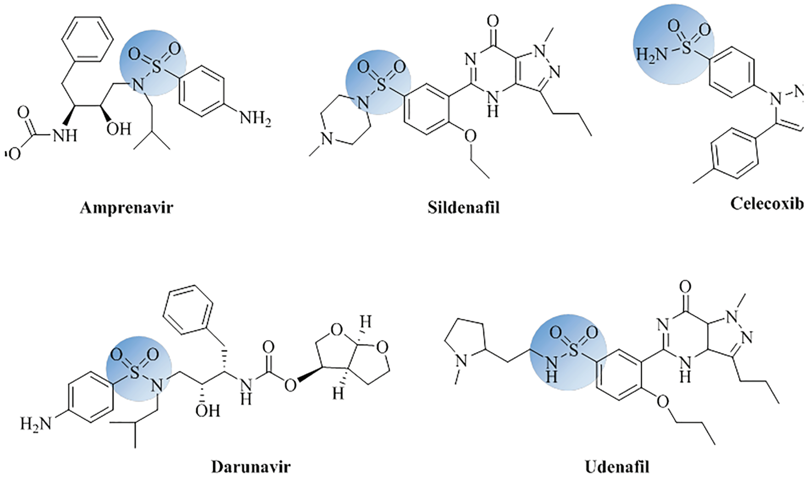


FIGURE 1 Structure of important sulfonamide-based drugs

materials have appeared as one of the most significant research areas in the concerning of advanced functional materials. Particularly, Polshettiwar et al., reported fibrous nano-silica (KCC-1), with the high surface area (typically >700 m^2/g), broad pore size distribution, large pore sizes,[24,25] ease of surface modification, low density, stability and low toxicity with good biocompatibility.[26–28] This Dendritic fibrous nanosilica showed special activities as heterogeneous catalysis.[29]

Herein, we report a simple and facile method for the synthesis of sulfonamides using Fe_3O_4@KCC-1-npr-NH_2 as a reusable and efficient nanocatalyst in water as a green solvent at room temperature in excellent yields. This approach involves various amines as nitrogen donor and paratoluenesulfonylchloride as the sulfur source for the preparation of sulfonamides.

2 | EXPERIMENTAL

2.1 | Materials and methods

All of materials and solvents were purchased from Merck, Sigma Aldrich and Fluka in high purity and used without further purification. Melting points were measured in open capillariesusing an Electrothermal MEL-TEMP apparatus (model 9200). X-ray diffraction (XRD) patterns of KCC-1 based materials were recorded by Siemens D 5000 X-Ray diffractometer (Texas, USA) with a Cu K_α anode (λ = 1.54 A°) operating at 40 kV and 100 mA. The Scanning Electron Microscopy (SEM) images and Energy Dispersive X-Ray (EDX) were recorded with FEG-SEM MIRA3 TESCAN, Czech Republic) at 1000 kV. Transmission Electron Microscopy (TEM) analysis was conducted on a Carl Zeiss LEO 906 electron microscope operated at 100 kV (Oberkochen, Germany). Brunauer–Emmett–Teller (BET) was recorded by Micromeritics NOVA 2000 (Florida, USA) apparatus at 77 K using nitrogen as the adsorption gas. The particle size distribution and zeta potential values were determined using Malvern particle size analyzer (Malvern, UK). The purity determination of the products and reaction monitoring were accomplished by TLC on silica gel poly gram SILG/UV 254 plates.

2.2 | General procedure for the synthesis of Fe_3O_4 magnetic nanoparticles

The synthesis procedure of hydrophilic nanoparticles Fe_3O_4 is explained as follows:

Briefly, $FeCl_3.6H_2O$ (3.25 g) was dispersed along with sodium acetate (NaAc, 6.0 g) and trisodium citrate (1.3 g) in the ethylene glycol (100 ml) with stirring. The collected yellow solution was transferred in a Teflon-lined stainless-steel autoclave with 200 ml capacity and the heating process of autoclave was continued about 10 hr at 200 °C. After cooling at room temperature the black product was rinsed several times with water and ethanol.[30]

2.3 | General procedure for the synthesis of Fe_3O_4@KCC-1 magnetic nanoparticles

At first stage the mixture of 0.1 g of the Fe_3O_4 NPs and 1.8 g of urea in 15 ml of water were sonicated about 30 mins. Then, 1.0 g of cetyl trimethyl ammonium bromide (CTAB), 1.0 g of n-butyl alcohol 1.0 g and 20 g of cyclohexane were added respectively and stirred for 30 mins. At this stage tetraethyl orthosilicate (TEOS) (1.00 g) was added drop wise and stirred with mechanical stirrer for 24 hr at 70 °C. Then, the product was washed with ethanol and water a few times for further process and dried in vacuum at 80 °C for 12 hr. Finally, the synthesized material was calcined at 550 °C for 5 hr in air to remove CTAB as template.

2.4 | General procedure for the synthesis of nanocatalyst (Fe_3O_4@ KCC-1-npr-NH_2)

For this purpose, Fe_3O_4@KCC-1 (0.2 g) was added in toluene (100 ml) and sonicated for 30 min. Then, (3-aminopropyl) triethoxysilane (APTES) (500 μL) were added in the mixture and the solution was refluxed for 24 hr under N_2. After that, the product was centrifuged, and was further purified by washing with ethanol and toluene several times and dried at 80 °C. Scheme 1 shows synthesized procedure of Fe_3O_4@ KCC-1-npr-NH_2 as an advanced nanocatalyst.

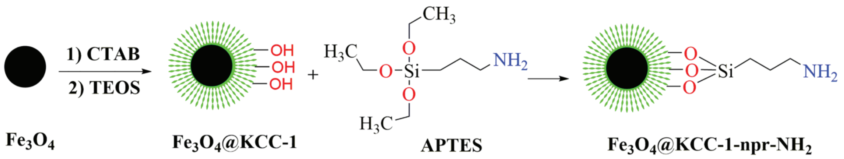
2.5 | General procedures for the synthesize of sulfonamides (3a-o)

A mixture of paratoluenesulfonylchloride (1 mmol) (1) and various amines (1 mmol) (2a-m) and Fe_3O_4@KCC-1-npr-NH_2 (0.1 mg) in water (3 ml) was magnetically stirred for 30–60 min at ambient temperature. Then, the purely mixture monitored by TLC and after completion of the reaction, the catalyst Fe_3O_4@ KCC-1-npr-NH_2 MNPs were separated by strong external magnet. The obtained precipitate was filtered and washed with cool EtOH to afford the pure product and then dried well under vacuum pump.

3 | RESULTS AND DISCUSSION

The synthesis of Fe_3O_4@ KCC-1-npr-NH_2 magnetic nanocatalyst involved several steps (Scheme 1). To investigation of the morphology of Fe_3O_4@KCC-1-npr-NH_2 MNPs, the FESEM images were recorded (Figure 2a). Moreover, the structure and size of the Fe_3O_4@ KCC-1-npr-NH_2 MNPs were evaluated utilizing transmission electron microscopy (TEM). As shown in Figure 2b, the uniform fibers of the magnetic KCC-1 with high surface area have several Si-OH groups that could grow from the center to outside. TEM images revealed the porous, fibrous and dendritic structure of those magnetic nanomaterials which the fibrous system is as a result of using the CTAB for the MNPs design while the fibrous-sphere reveals the formation of Fe_3O_4@KCC-1. The size of the Fe_3O_4 MNPs is about 15 nm as can be seen in Figure 2 (a and b). Also, Fe_3O_4@KCC-1-npr-NH_2 MNPs have a particle size range of 20 nm (Figure S1 (see supporting information)). In addition, EDX results indicate the atomic structure of the produced compound that the KCC-1 is composed only with Si and O. Moreover, functionalization of KCC-1 with APTES, the weight percent of N, O and C are increased which confirmed successful functionalization of KCC-1 by npr-NH_2 (Figure S2 (see supporting information)). Though, the carbon is arising from the SEM grid and CTAB as a pattern agent NH_2 (Figure S3 (see supporting information)).

The powder X-ray diffraction patterns of Fe_3O_4@KCC-1-npr-NH_2 are shown in Figure 3. The XRD pattern of Fe_3O_4@KCC-1-npr-NH_2 NPs was performed from 5.0°



SCHEME 1 Schematic for the synthesis of Fe_3O_4@KCC-1-npr-NH_2

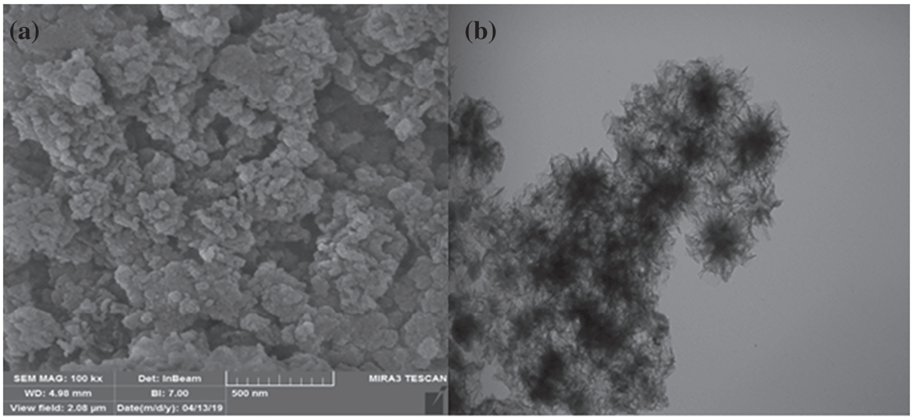


FIGURE 2 SEM (a) and TEM (b) images of Fe_3O_4@KCC-1-npr-NH_2

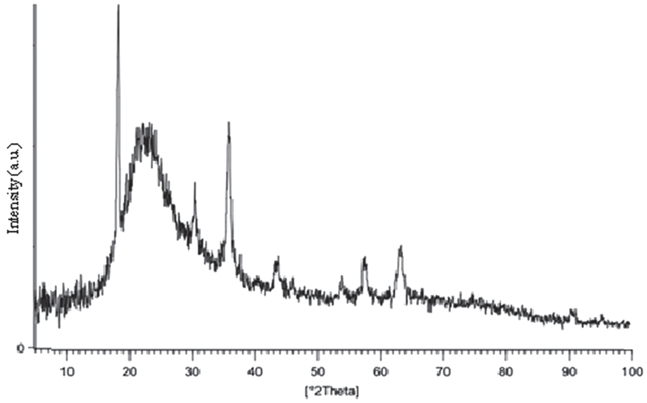


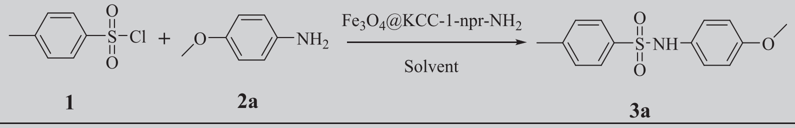
FIGURE 3 XRD analysis of Fe_3O_4@KCC-1-NH_2

(2θ) to 100.0° (2θ) to investigate the crystallinity of the synthesized nanomaterial in order to obtain additional information about their molecule structures. As can be observed in Figure 3, sample possess the diffraction peaks at (220), (311), (400), (422), (511) and (440), which are in similarity with the data for a standard Fe_3O_4 sample, as reported in this work. The wide peak between 20° and 30° is related to the amorphous silica and proved the effective coating of the silica on Fe_3O_4 core. It is important to point out that the XRD templates of the Fe_3O_4@KCC-1-npr-NH_2 NPs are similar to the fibrous mesoporous nanosilica with Fe_3O_4 core.[31]

The N_2 adsorption–desorption isotherms of Fe_3O_4 @ KCC-1-npr-NH_2 are shown in (Figure S4 (c) (see supporting information)). The BET and BJH analyses of the Fe_3O_4 @ KCC-1-NH_2 MNPs were used to determine the porous essence of the synthesized nanocatalyst Figure S2. (c) (see supporting information). The specific surface area and porosity of the nanomaterials were determined using the adsorption isotherm and calculated by BET. Also, BJH technique was used to evaluate the pore volume of the KCC-1, KCC-1-npr-NH_2 and Fe_3O_4 @ KCC-1-npr-NH_2 (Figure S2 (a-c) (see supporting information)). The surface area of KCC-1, KCC-1-npr-NH_2 and Fe_3O_4@ KCC-1-npr-NH_2 was observed about 617, 367 and 87 m^2 g^{-1}, and also the average pore size is 5.8 nm. The pore volumes, pore size, and surface area of KCC-1, KCC-1-npr-NH_2 and Fe_3O_4 @ KCC-1-npr-NH_2 are clearly proved by the reported results.[32,33]

Zeta potentials of Fe_3O_4@KCC-1-npr-NH_2 was checked at pH 7.5 to control the surface charge to determine the possible surface modification. The zeta potential of the Fe_3O_4@KCC-1-npr-NH_2 shows positive charges which verify the anchoring amine and Fe_3O_4 groups on the surface of the fibrous system, respectively.[32] Also, DLS result of the Fe_3O_4@KCC-1-npr-NH_2 approves again the successful functionalization of KCC-1 with npr-NH_2 and Fe_3O_4 which the first peak related to Fe_3O_4 and the second peak

TABLE 1 The effect of solvent and temperature for the synthesis of sulfonamides



Entry	Solvent	Temp. (°C)	Yield (%)
1	EtOH	r.t.	75
2	H_2O	r.t.	97
3	H_2O	60	97
4	H_2O	reflux	97
5	n-Hexane	r.t.	23
6	CCl_4	r.t.	29
7	THF	r.t.	38
8	CH_3CN	r.t.	44
9	CH_2Cl_2	r.t.	39
10	$CHCl_3$	r.t.	47
11	DMF	r.t.	30
12	Methanol	r.t.	66
13	i-PrOH	r.t.	59

Reaction conditions: 4-toluenesulfonyl chloride (1 mmol), 4-methoxy aniline (1 mmol), Fe_3O_4@ KCC-1-npr-NH_2 (0.1 mg), water (4 ml), r.t. for 30 min. Isolated yield (%).

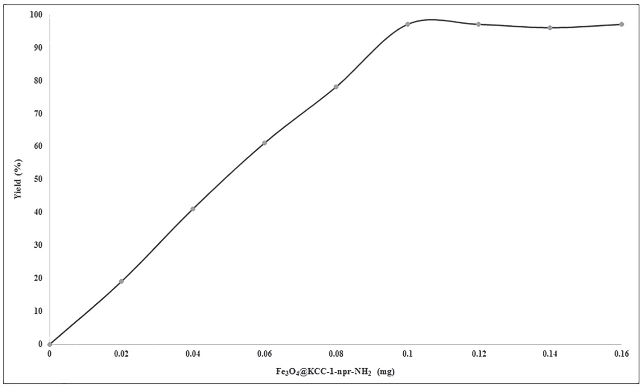


FIGURE 4 Optimization of the conditions for synthesis of sulfonamides: 4-toluenesulfonyl chloride (1 mmol), 4-methoxy aniline (1 mmol), Fe_3O_4@ KCC-1-npr-NH_2 (0.1 gm), water (4 ml), *r. t.* for 1 hr

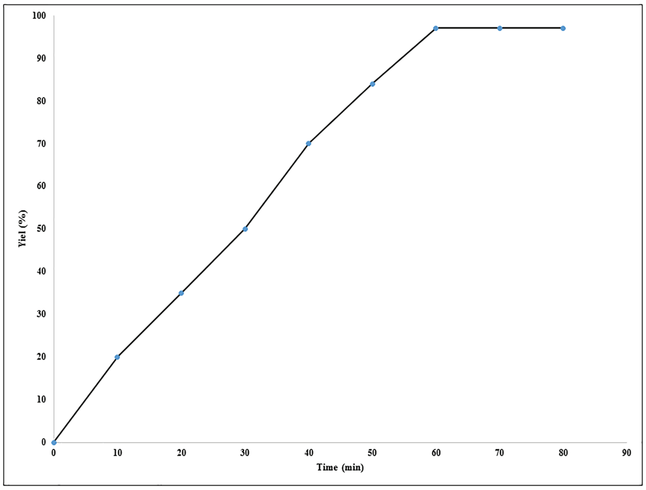
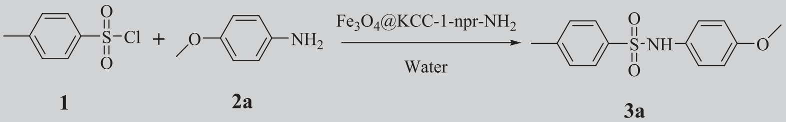


FIGURE 5 Effect of time on yield of sulfonamides: 4-toluenesulfonyl chloride (1 mmol), 4-methoxy aniline (1 mmol), Fe_3O_4@ KCC-1-npr-NH_2 (0.1 gm), water (4 ml), *r.t.* for 1 hr

related to KCC-NH_2 (Figure S5 (see supporting information)).

After characterization of Fe_3O_4@ KCC-1-npr-NH_2 the catalytical performance of this nanomatrial was tested for the synthesis of various sulfonamides. In order to optimize the sulfonation reaction conditions and to obtain good catalytic activity, the synthesis of sulfonamides obtained from 4-toluenesulfonyl chloride (1) and 4-methoxy aniline (2a) was used as a model reaction and investigated under different reaction parameters including amount of the catalyst, time, temperature, and solvent type. Initially, the effect of most commonly used organic solvents and water on the synthesis of sulfonamide compounds using the Fe_3O_4@KCC-1-npr-NH_2 as a nanocatalyst was investigated. It is obtained that, the solvent type has a significant effect the performance of the nanocatalyst. For example, n-Hexane and CCl_4 which are non-polar solvents, gave sulfonamides in a lower yield than other solvents (Table 1, entry 5 and 6). Also, some aprotic solvents including THF, CH_3CN, CH_2Cl_2, $CHCl_3$ and DMF lead to low efficiency (Table 1, entry 7–11). But, the protic solvents improved reaction performance. Methanol and iPrOH gave sulfonamides in average yields (Table 1, entries 12 and 13). In contrast, the use of Ethanol gave a better yield, while the yield was considerably increased up to 97% when water was used as a green solvent in the presence of Fe_3O_4@KCC-1-npr-NH_2 MNPs. Also, the temperature showed no impact in the synthesis of sulfonamides in the presence of Fe_3O_4@KCC-1-npr-NH_2 MNPs. In this regard, the sulfonamides were obtained with excellent isolated yield at room temperature and results clearly indicated that reaction completion isn't related to reaction temperature and even conventional heating under reflux conditions in ethanol and water (Table 1) have no important effect on the yield of desired compounds. On the other hand, high tempera-

TABLE 2 Influence of different nanocatalysts for the synthesis of sulfonamides



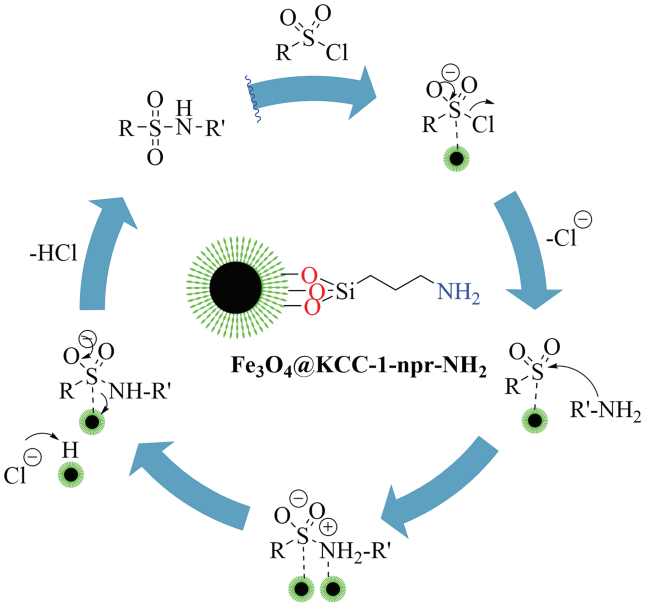
Entry	Catalyst	Yield (%)
1	KCC-1	-
2	KCC-1-npr-NH_2	-
3	Fe_3O_4	10
4	Fe_3O_4@KCC-1	20
5	Fe_3O_4@KCC-1-npr-NH_2	97

Reaction conditions: 4-toluenesulfonyl chloride (1 mmol), 4-methoxy aniline (1 mmol), Fe3O4@ KCC-1-npr-NH_2 (0.1 mg), water (4 ml), r.t. for 30 min. Isolated yield (%).

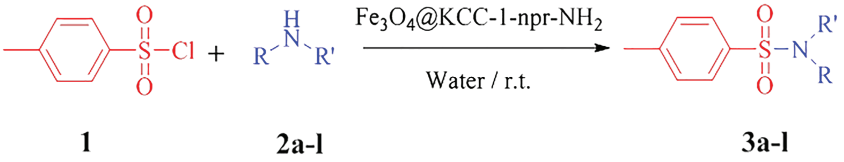
(Table 1, entry 2–4). However, the reaction at 30–60 min in the presence 0.0001 g of Fe_3O_4@KCC-1-npr-NH_2 is optimum condition for the synthesis of sulfonamides.

Finally, the amount of nanocatalyst on the reaction efficiently was evaluated. According to obtained results, the variation in the Fe_3O_4@KCC-1-npr-NH_2 MNPs amount had a key role on the reaction efficiency. The optimum amount of Fe_3O_4@KCC-1-NH_2 MNP was 0.1 mg, which obtained the desired product in 97% yields (Figure 4). It is important to point out that, we could achieve to excellent yields of sulfonamides synthesize using this nanocatalyst in 60 min (Figure 5).

To further investigate the efficiency of the nanocatalyst, we compared the catalytic performance of the newly nanocatalyst with different control experiments and the results are shown in Table 2. Originally, a standard reaction was accomplished using KCC-1, KCC-1-npr-NH_2, Fe_3O_4 and KCC-1-Fe_3O_4; and the results confirmed that the desired product was poorly formed (Table 2, entries 3 and 4) after 1 hr of reaction time in

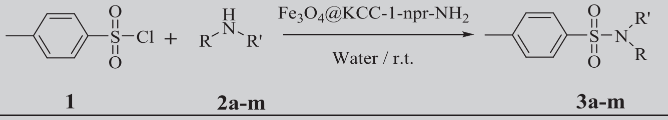


SCHEME 3 Possible mechanism for the synthesis of sulfonamides using Fe_3O_4@KCC-1-npr-NH_2



SCHEME 2 Synthesis of sulfonamides (3a-l) in the presence of KCC-1-NH_2-Fe_3O_4@KCC-1-npr-NH_2 NPs.

TABLE 3 Fe_3O_4@KCC-1-npr-NH_2 catalyzed synthesis of sulfonamides



Entry	R	R′	Product. No.	Time (min)	Yield (%)[a] [Ref.]
1	p-OMePh	H	3a	30	97 [20]
2	Morpholine	H	3b	30	96 [20]
3	Ph	H	3c	30	97 [20]
4	p-NO_2Ph	H	3d	60	85 [20]
5	P-MePh	H	3e	35	90 [20]
6	p-BrPh	H	3f	40	93 [20]
7	o-MePh	H	3 g	50	88 [20]
8	p-CF_3Ph	H	3 h	60	87 [20]
9	Cyclohexane	H	3i	55	90 [20]
10	N,N-diMethylPh	H	3j	50	94 [20]
11	p-CNPh	H	3 k	60	86 [20]
12	Bu	H	3 l	40	92 [20]
13	Et	Et	3 m	30	90 [34]

Reaction conditions: 4-toluenesulfonyl chloride (1 mmol), various amines (1 mmol), Fe_3O_4@ KCC-1-npr-NH_2 (0.1 mg), water (4 ml), r.t. for 30–60 min.
[a]Isolated yield (%).

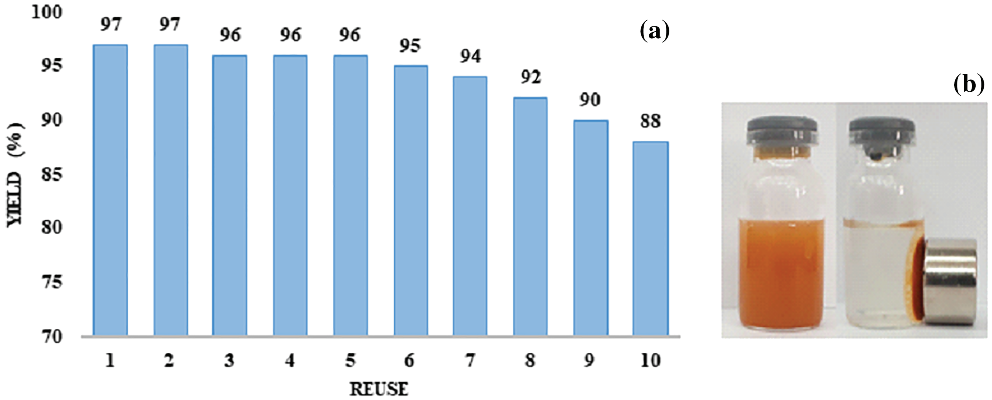


FIGURE 6 (a) Reusability of Fe_3O_4@ KCC-1-npr-NH_2 MNPCs and (b) separation of the nanocatalyst by external magnet

TABLE 4 Comparison of different catalysts for synthesis of N-(phenyl)-p-toluenesulfonamide (**3c**)

Entry	Catalyst	Conditions	Time (min)	Yield (%)	Ref.
1	Nano ZnO	50 °C	60	94	[19]
2	CuO	r.t.	120	88	[20]
3	CdO nanopowder	Reflux	10	95	[21]
4	Zn-Al hydrotalcite	Ultrasound	10	95	[35]
5	Natrolite zeolite	Ultrasound	15	95	[36]
6	Fe_3O_4@KCC-1-npr-NH_2	r.t	30	97	This work

any amount. When, Fe_3O_4@KCC-1-NH_2 was used as the nanocatalyst, a reaction was performed and completed (Table 2, entry 5).

Ultimately, the reaction conditions were optimized. To carry out this approach, we specially evaluated this methodology utilizing 4-toluenesulfonyl chloride (1) and a variety of different amines (2a-m) in the presence of Fe_3O_4@KCC-1-NH_2 MNP in water under optimized reaction conditions (Scheme 2). As can be seen in Table 3, the type of substituents on the aromatic ring and electronic effects did not show extremely evident effects in terms of yields under the reaction conditions. As can be seen in Table 2, it is clear that the aromatic amines containing electron-donating groups and electron-withdrawing which were used in this research, were reacted well to afford the desired sulfonamides in excellent yields with high purity. In addition, secondary amine such as diethylamine also react effectively to prepare the desired product in excellent yield (Table 3, entire 13). Also, the proposed mechanism for the synthesis of sulfonamides using Fe_3O_4@KCC-1-npr-NH_2 MNP is presented in Scheme 3.

The recovery and reuse of catalyst materials are highly preferable. In this regard, the recyclability of the Fe_3O_4@KCC-1-npr-NH_2 MNP was investigated using the model reaction 4-toluenesulfonyl chloride and various amines under identical reaction conditions. After the completion of reaction, the recovered catalyst from the reaction mixture was washed with chloroform and dried at room temperature and reused for subsequent reactions. It is obvious that the heterogeneous and magnetic property of the Fe_3O_4@KCC-1-NH_2 MNP facilitates the effective recovery of the nanocatalyst from the reaction mixture by external magnet during the work-up procedure so that the catalyst could be recycled and reused up to ten consecutive trials without remarkable loss of its catalytic activity (Figure 6 (a and b)) These results indicated that the proposed nanocatalyst was stable and could tolerate the sulfonation reaction conditions.

In order to show the special efficiency of the Fe_3O_4@KCC-1-npr-NH_2 MNP in comparison with different catalysts which used for similar reactions, we summarize numerous results for the synthesis of N-(phenyl)-p-toluenesulfonamide (3c) in Table 4. Our study has some advantages in compare with other mention studies including high yield of synthetic compound, reasonable time reaction and easy catalyst recovery. In this regard, some of other reported have shorter reaction time.

4 | CONCLUSIONS

In summary, a novel magnetic nanocatalyst (Fe_3O_4@KCC-1-npr-NH_2) was prospered and used for the synthesis of sulfonamide derivatives in water as a green solvent in excellent yield. This nanocatalyst could be recovered and reused at least ten times with no considerable decrease in its activity and selectivity. Proposed method, has some advantages, containing short reaction times, mild reaction conditions, reusability of the solid catalyst, high yields and convenient workup process. In addition, the desired sulfonamides were collected by filtration without utilizing any chromatographic separation or organic solvent for extraction confirming a high mass efficiency.

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