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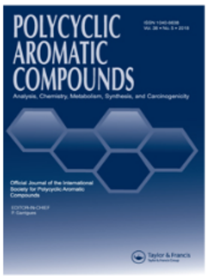

Polycyclic Aromatic Compounds

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MNPs@PBI-$CuBr_2$ Nanocomposite Catalyzed One-Pot Three-Component Reaction of 2-Aminobenzoazoles, Sulfonyl Chloride and Aryl Iodides

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ABSTRACT

Sulfonamides are widely used in the treatment of many diseases due to their biological and pharmacological activities. In this paper, we prepared Cu(II) complex immobilized on the surface of magnetic Fe_3O_4 nanoparticles functionalized with 2-Pyridine Benzoimidazole ligand [**MNPs@PBI-$CuBr_2$**] and its catalytic activity in the synthesis of N-aryl sulfonamides containing benzoxazole scaffolds through one-pot three-component reaction of 2-aminobenzoazoles, sulfonyl chloride and aryl iodides under ecofriendly conditions. The structure of the as-prepared MNPs@PBI-$CuBr_2$ nanomaterial was well characterized by a number of spectroscopic techniques such as FTIR, SEM, EDX, TEM, XRD, TGA, VSM, MAP, XPS and ICP-OES. The MNPs@PBI-$CuBr_2$ nanocatalyst was simply separated by magnetic separation and reused for seven times with constant activity. The TEM, XRD pattern and ICP-OES spectroscopy of catalyst after recycling were studied and the results were similar to before of utilization.

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KEYWORDS
MNPs@PBI-$CuBr_2$ nanocatalyst; N-aryl sulfonamides; ecofriendly conditions; magnetic separation

Introduction

The development of new, ecofriendly and efficient catalytic systems for the preparation of chemical compounds is an important issue in the modern organic chemistry.[1] The widespread use of catalysts in chemistry in the current century has led researchers to design high activity and recyclable catalysts.[2,3] Heterogeneous catalysts have less environmental effects due to advantages such as retrievability, reducing the number of preparation steps and product separation.[4,5] An efficient catalyst that has a high specific surface and a large number of active sites, in this respect, nanoparticles have these characteristics, they are among the best options for catalysts.[6–8] Uncoated nanoparticles are often unstable and stick together during the catalytic reaction. In order to produce stable nanoparticles and maintain their catalytic properties, it is necessary to stop the growth of nanoparticles and stabilize their surface.[9,10] Protection is done by increasing stabilizing factors or stabilizing them on solid materials with a high specific surface area. Magnetic nanoparticles or iron oxide nanoparticles (iron oxides in maghemite or magnetite crystal structure) are the most magnetic nanoparticles to date.[11] Magnetic nanoparticles are an ideal support for connecting

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homogeneous organic and inorganic catalysts, which lead to the production of heterogeneous catalysts while maintaining the activity and properties of homogeneous catalysts.[12–14] Due to the fact that non-magnetic heterogeneous nanocatalysts are separated from the reaction medium by filtration or centrifugation, these methods are one of the disadvantages of these nanocatalysts.[15,16] Iron oxide (Fe_3O_4) nanoparticles (NPs) are the most important group of magnetic nanoparticles.[17] Magnetic Fe_3O_4 nanoparticles supported metallic catalysts can readily separate from the reaction mixture using an external magnet and reuse for many times with constant activity.[18–20]

One-pot multicomponent reactions (MCRs) have emerged as an efficient tool for benign synthesis by virtue of their convergence, productivity, facile execution, and generation of highly diverse and complex products from easily available starting materials in a single operation.[21–23] The advantages of MCRs over multistep synthesis include atom-economy and step-efficiency, which reduce waste generation, in particular.[24,25]

Sulfonamides are widely used in the treatment of many diseases due to their biological and pharmacological properties.[26–28] Sulfonamides or sulfa drugs are a class of antibiotics that target the bacteria causing the infection.[29] Sulfonamide derivatives are generally broad-spectrum antibiotics that act on a wide range of bacteria and are therefore used in the treatment of many types of bacterial infections (Figure 1).[30–34] These drugs are used in the treatment of many infectious and parasitic diseases (such as malaria) and their mechanism of action is inhibition of nucleic acid synthesis.[34,35]

In this paper, we constructed Cu(II) complex immobilized on the surface of magnetic Fe_3O_4 nanoparticles modified with 2-Pyridine Benzoimidazole ligand [**MNPs@PBI-CuBr$_2$**] and its catalytic behavior in the synthesis of N-aryl sulfonamides containing benzoazole scaffolds through one-pot three-component reaction of 2-aminobenzoazoles, sulfonyl chloride and aryl iodides under ecofriendly conditions.

Result and discussion

As shown in Scheme 1, MNPs@PBI-$CuBr_2$ nanocomposite was successfully constructed through the immobilization of $CuBr_2$ on the surface of magnetic Fe_3O_4 nanoparticles modified with 2-Pyridine Benzoimidazole as ligand.

In norder to study functional group supported on magnetic nanopparticles, the structure of Fe_3O_4 MNPs, MNPs@PBI ligand and MNPs@PBI-$CuBr_2$ nanocomposite analyzed by FT-IR

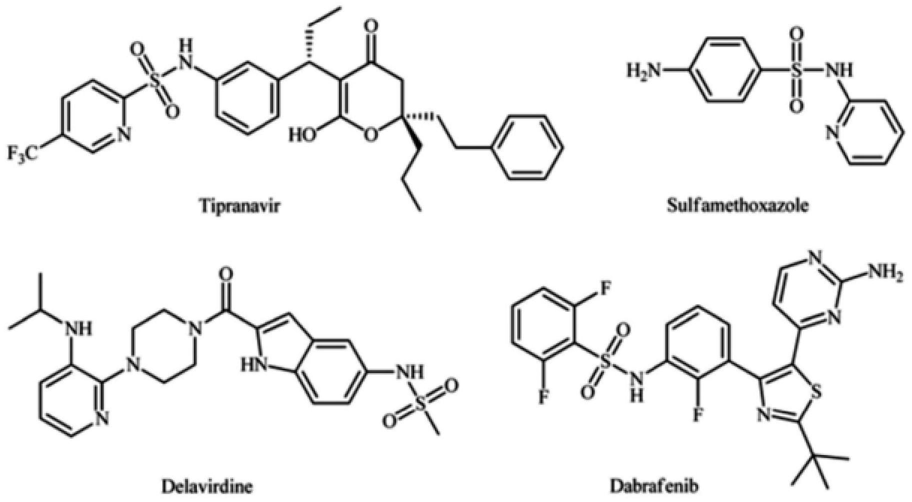
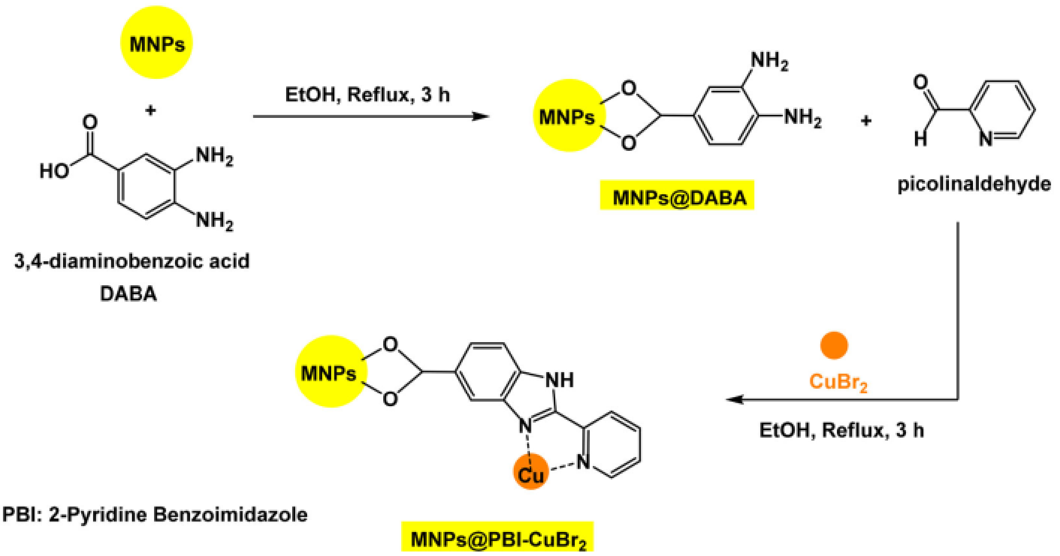


Figure 1. Several N-aryl sulfonamide drugs.

spectroscopy (Figure 2). The absorption in 579 cm^{-1} is related to the metal–oxygen bond peak which confirmed the formation of Fe_3O_4 nanoparticles. The presence of broad peack at 3402 cm^{-1} corresponded to OH groups on the magnetic surface of Fe_3O_4 MNPs.[36] In the FT-IR spectrum of MNPs@PBI ligand and MNPs@PBI-$CuBr_2$ nanocomposite, new bands were observed at 1650 and 1410 cm-1 assigned to the C=N OR C-N and C-H (aromatic) stretching vibrations, respectively.[36] Also, the characteristic bands at 2855 and 2920 cm^{-1} are assigned to the symmetric and asymmetric stretching of C-H bonds in 2-Pyridine Benzoimidazole. The chelation of copper to MNPs@PBI causes a red shift of imine bond vibration from 1650 to 1620 cm^{-1} as shown in Figure 2, which is evidence for the complex formation of copper by the Schiff base ligand.[37]



Scheme 1. General route for construction of MNPs@PBI-$CuBr_2$ nanocatalyst.

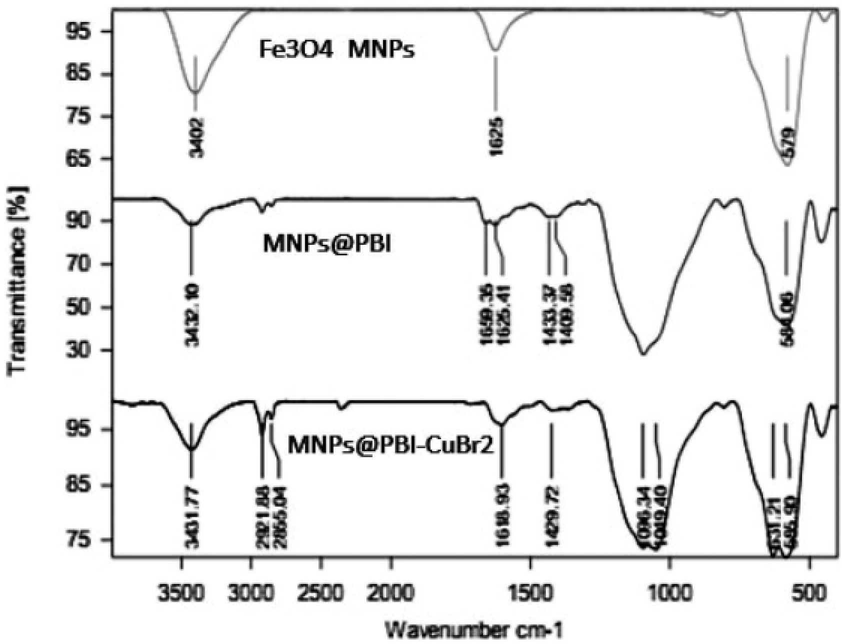


Figure 2. FT-IR analysis of Fe_3O_4 MNPs, MNPs@PBI ligand and MNPs@PBI-$CuBr_2$ nanocomposite.

Size and morphology of MNPs@PBI-$CuBr_2$ catalyst were investigated using TEM and SEM analysis. As exhibited in Figure 3, most of the particles are quasi-spherical with an average diameter at about 15–25 nm, which are in good agreement with the XRD result. The SEM photographs display spherical morphology and uniform nanometer size for most particles (Figure 4).

The successful fabrication of the MNPs@PBI-$CuBr_2$ nanocomposite is confirmed by EDX analysis. EDX analysis displayed the presence of Fe, O, C, N, and Cu species in the structure of MNPs@PBI-$CuBr_2$ catalyst (Figure 5). The magnetic properties of Fe3O4 nanoparticles and MNPs@PBI-$CuBr_2$ nanocomposite have been demonstrated by VSM analysis which shown in Figure 6. The magnetic saturation values of Fe_3O_4 nanoparticles and MNPs@PBI-$CuBr_2$ are 78.2 and 52.1 emu.g-1, respectively. The decrease in the magnetization properties of bare Fe_3O_4 can be attributed to the nonmagnetic copper and ligand shell on the particle surface.

According to previous reports, the XRD pattern of first to third steps shows the standard XRD pattern of crystalline cubic spinel Fe_3O_4 nanoparticles. XRD pattern of the MNPs@PBI ligand and MNPs@PBI-$CuBr_2$ catalyst, standard XRD pattern of crystalline cubic spinel Fe_3O_4 nanoparticles in positions of 2θ angles at 30.1, 35.7, 43.4, 53.7, 57.5 and 62.9 were shown (Figure 7). The

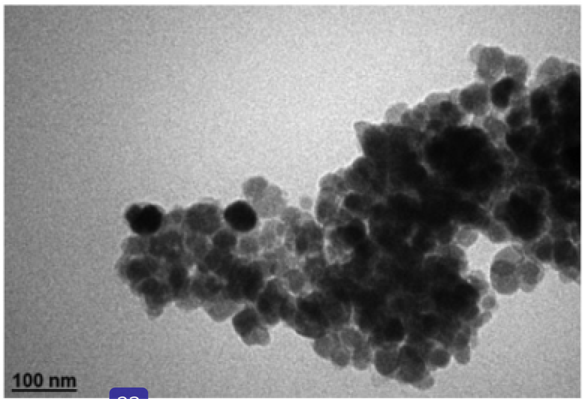


Figure 3. TEM images of MNPs@PBI-$CuBr_2$ nanocomposite.

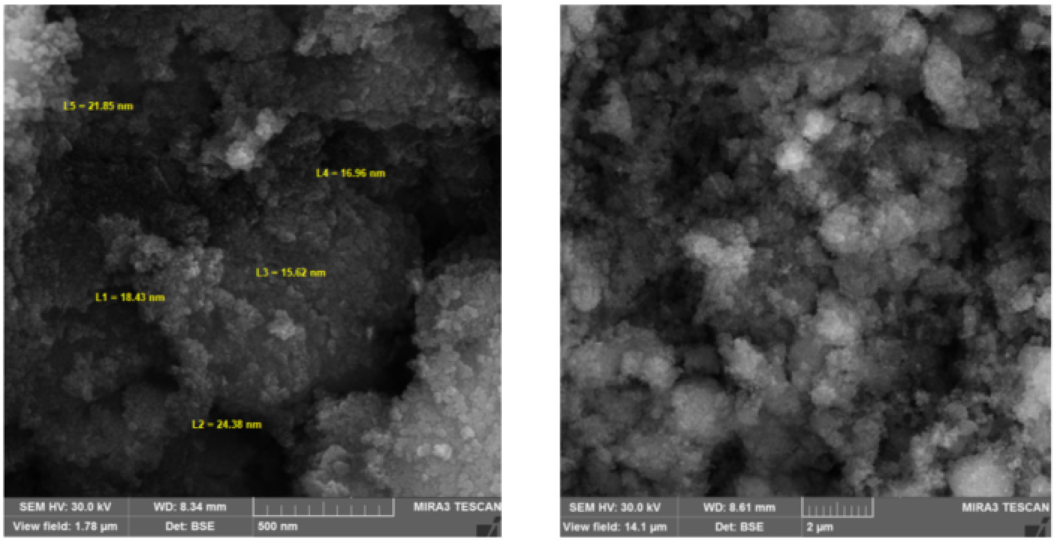


Figure 4. SEM images of MNPs@PBI-$CuBr_2$ nanocomposite.

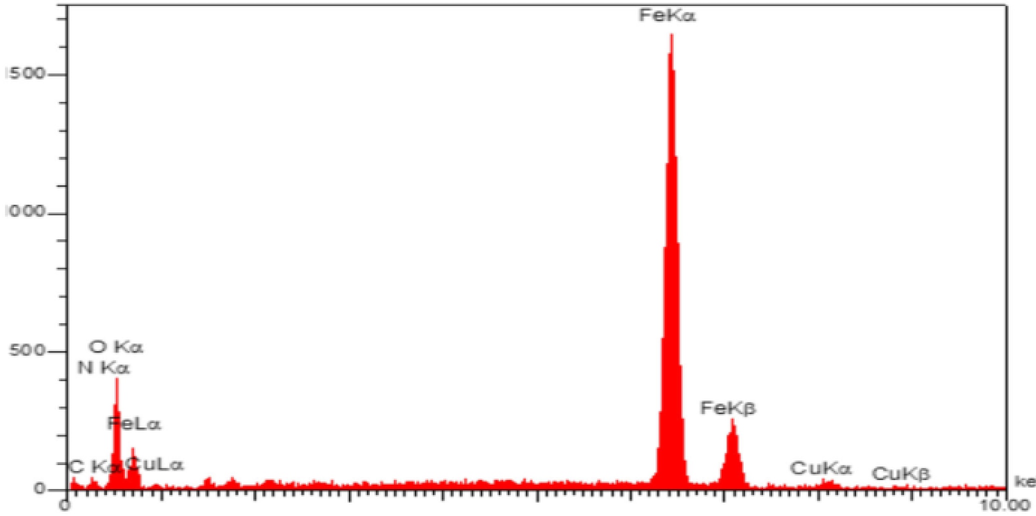


Figure 5. EDX analysis of MNPs@PBI-$CuBr_2$ nanocomposite.

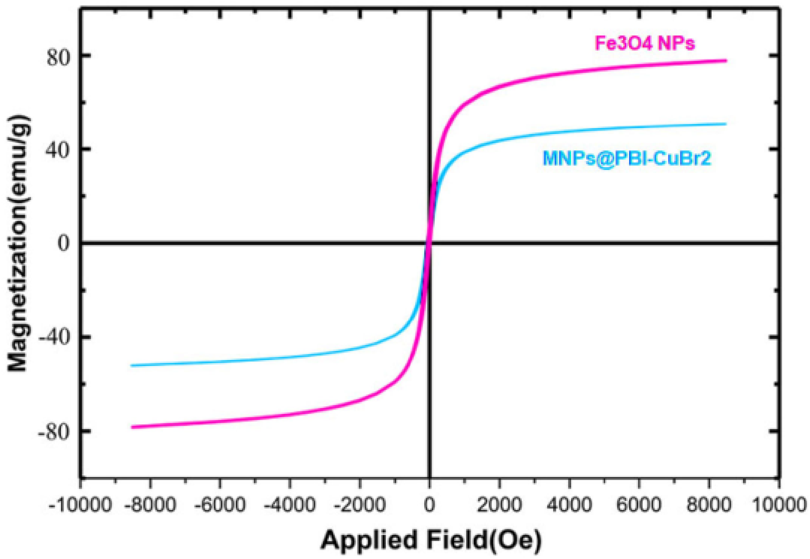


Figure 6. VSM analysis of Fe_3O_4 MNPs and MNPs@PBI-$CuBr_2$.

X-ray mapping of MNPs@PBI-$CuBr_2$ nanocatalyst is shown in Figure 8. The good dispersion of Cu on the surface of the catalyst was confirmed using elemental map images.

Inductively coupled plasma optical emission spectroscopy (ICP-OES) to determine the amount of Cu loaded on modified magnetic was performed and it was found to be 1.22 mmol g^{-1}.

X-ray photoelectron spectroscopy (XPS) is also performed in order to investigate the oxidation state of the copper species present in the structure of MNPs@PBI-$CuBr_2$ nanocomposite (Figure 9). Two characteristic peaks at 934.2 and 953.1 eV are seen that is related to the bonding energies $2p_{3/2}$ and $2p_{1/2}$ respectively. This issue confirmed that only Cu oxidation state (II) exist in the sample.[38]

After comprehensive characterization of MNPs@PBI-$CuBr_2$ nanocomposite, we decide to evaluate its catalytic activity in the synthesis of N-aryl sulfonamides containing benzoxazole

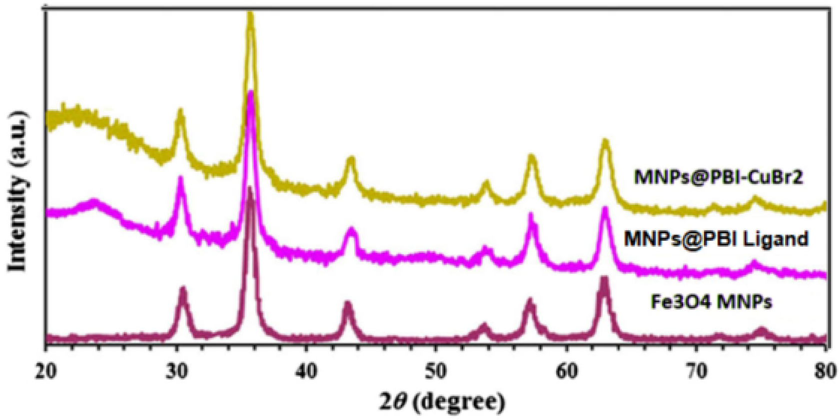


Figure 7. XRD analysis of Fe_3O_4 MNPs, MNPs@PBI Ligand and MNPs@PBI-$CuBr_2$ nanocatalyst.

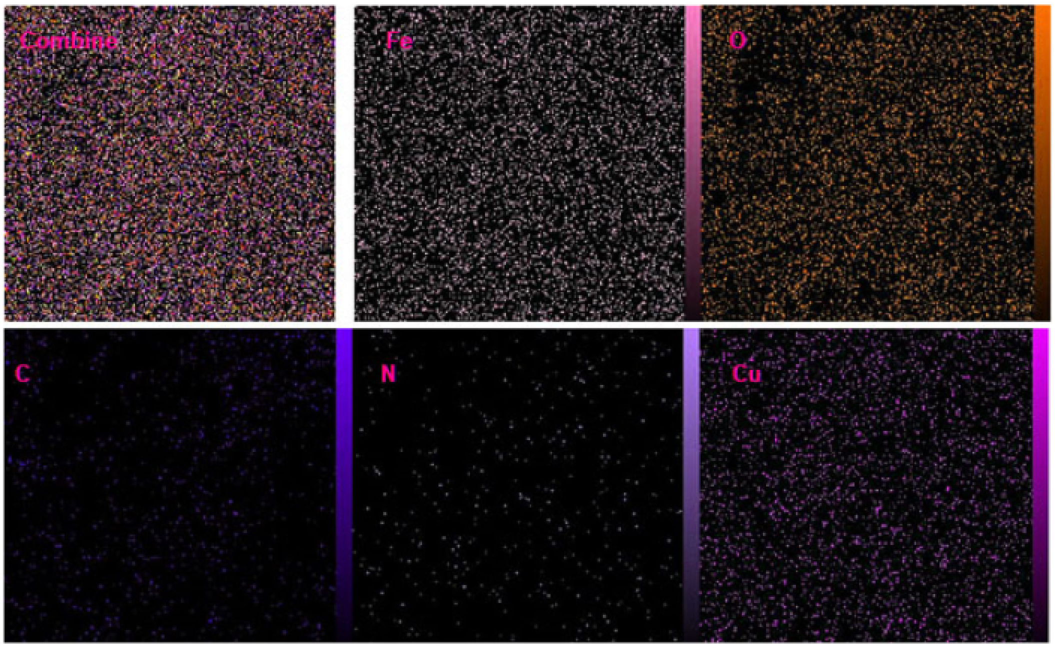


Figure 8. X-ray map analysis of MNPs@PBI-$CuBr_2$ nanocatalyst.

scaffolds through one-pot three-component reaction of 2-aminobenzoazoles, sulfonyl chloride and aryl iodides under ecofriendly conditions. In this respect, we first selected the reaction of 2-aminobenzoxzole (**1**) with iodobenzene (**2**) and sulfonyl chloride (**3**) as the model reaction and evaluated the effect of catalyst, base, solvent and temperature to find the standardized conditions. First, the influence of catalyst loading was studied (Figure 10), the model reaction was not carried out in the absence of MNPs@PBI-$CuBr_2$ nanocatalyst. The highest yield was seen in the presence of 25 mg of MNPs@PBI-$CuBr_2$ nanocatalyst.

Next, the model reaction was catalyzed by 25 mg of MNPs@PBI-$CuBr_2$ nanocatalyst in the presence of different bases in DMF under thermal conditions (Figure 11). A number of customary bases was tested and the highest yield was seen in the presence of KOAc; the model reaction was failed in the absence of any bases.

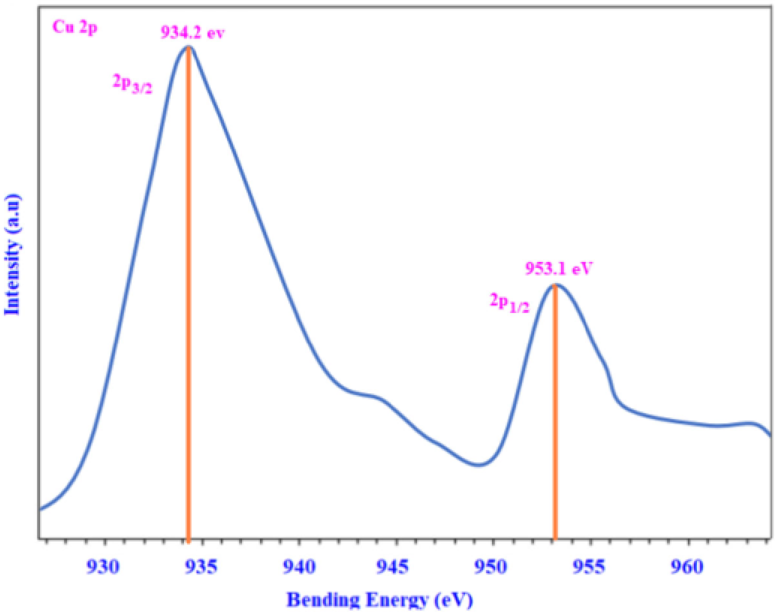


Figure 9. XPS Spectrum of MNPs@PBI-$CuBr_2$ nanocatalyst.

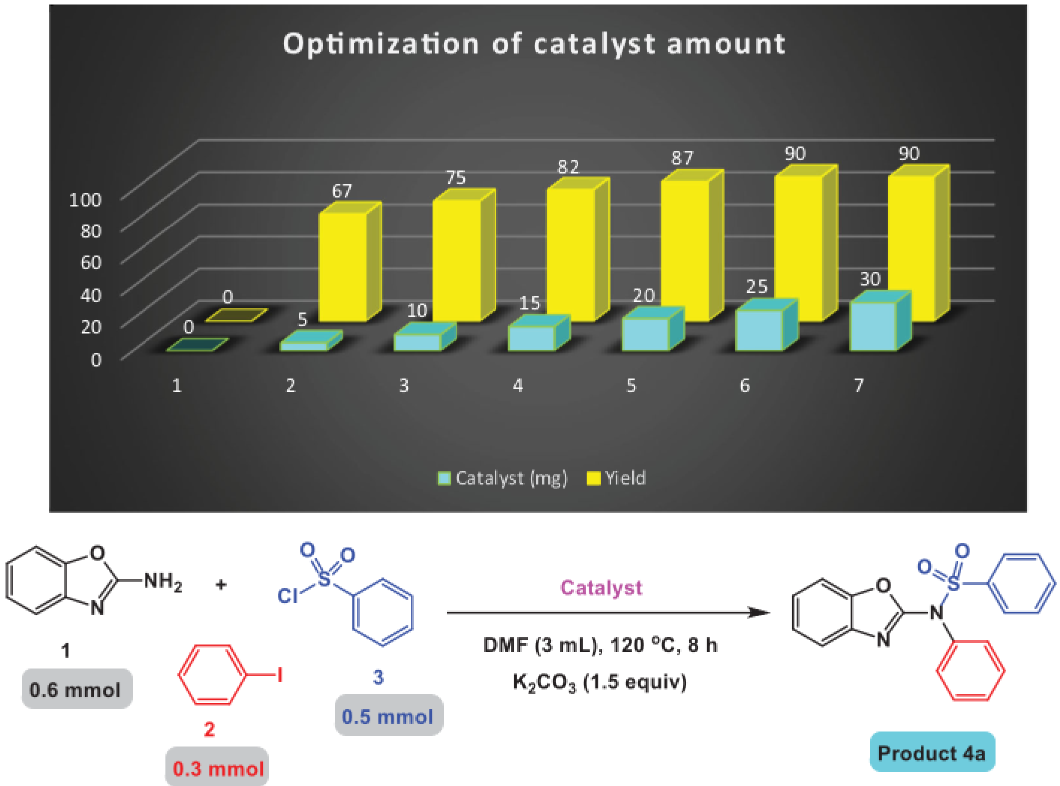


Figure 10. Optimization of the amount of **catalyst** on the model reaction (**Product 4a**).

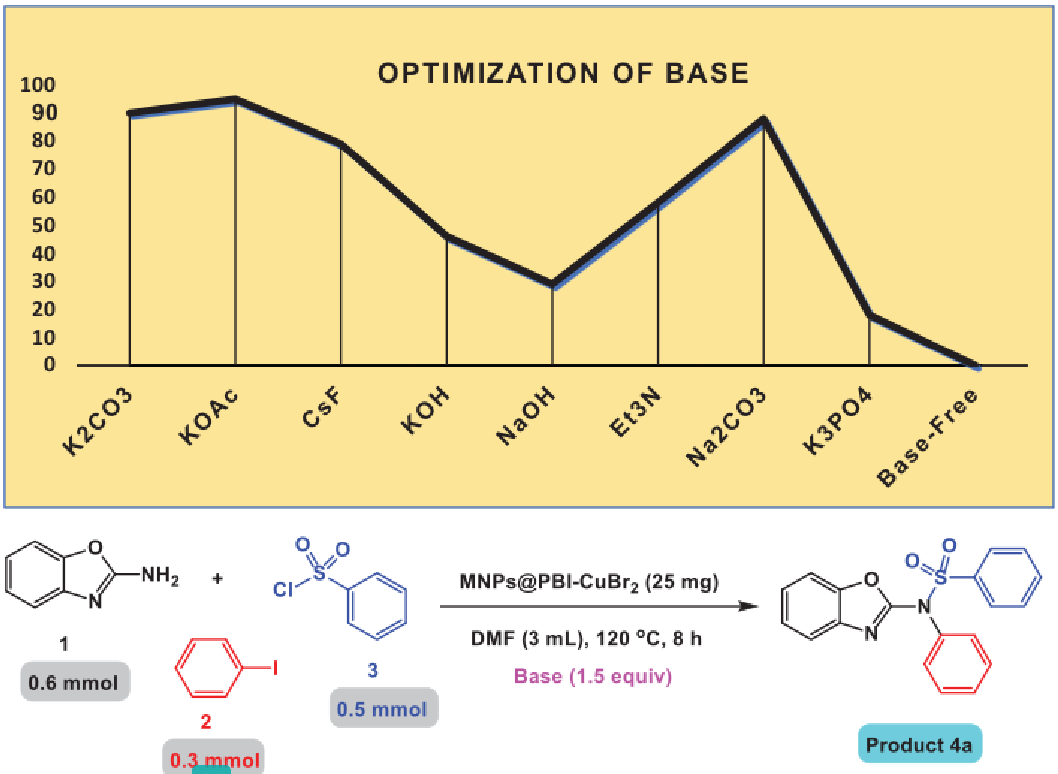


Figure 11. Optimization of the effect of **base** on the model reaction (**Product 4a**).

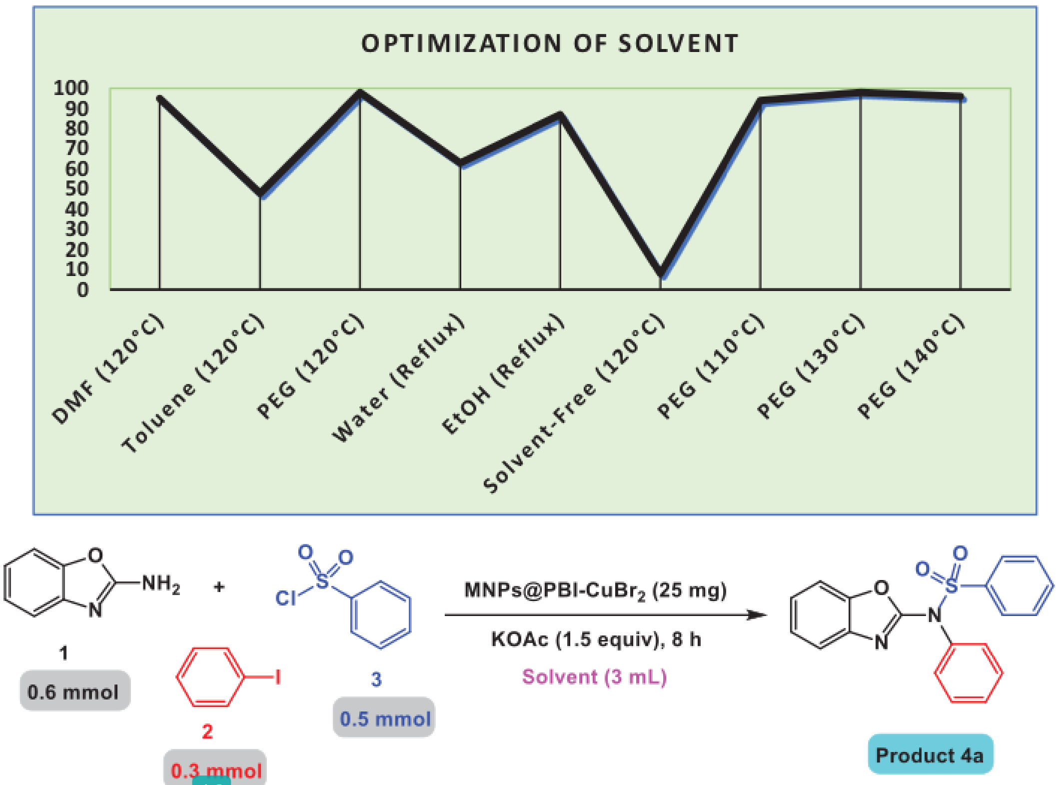
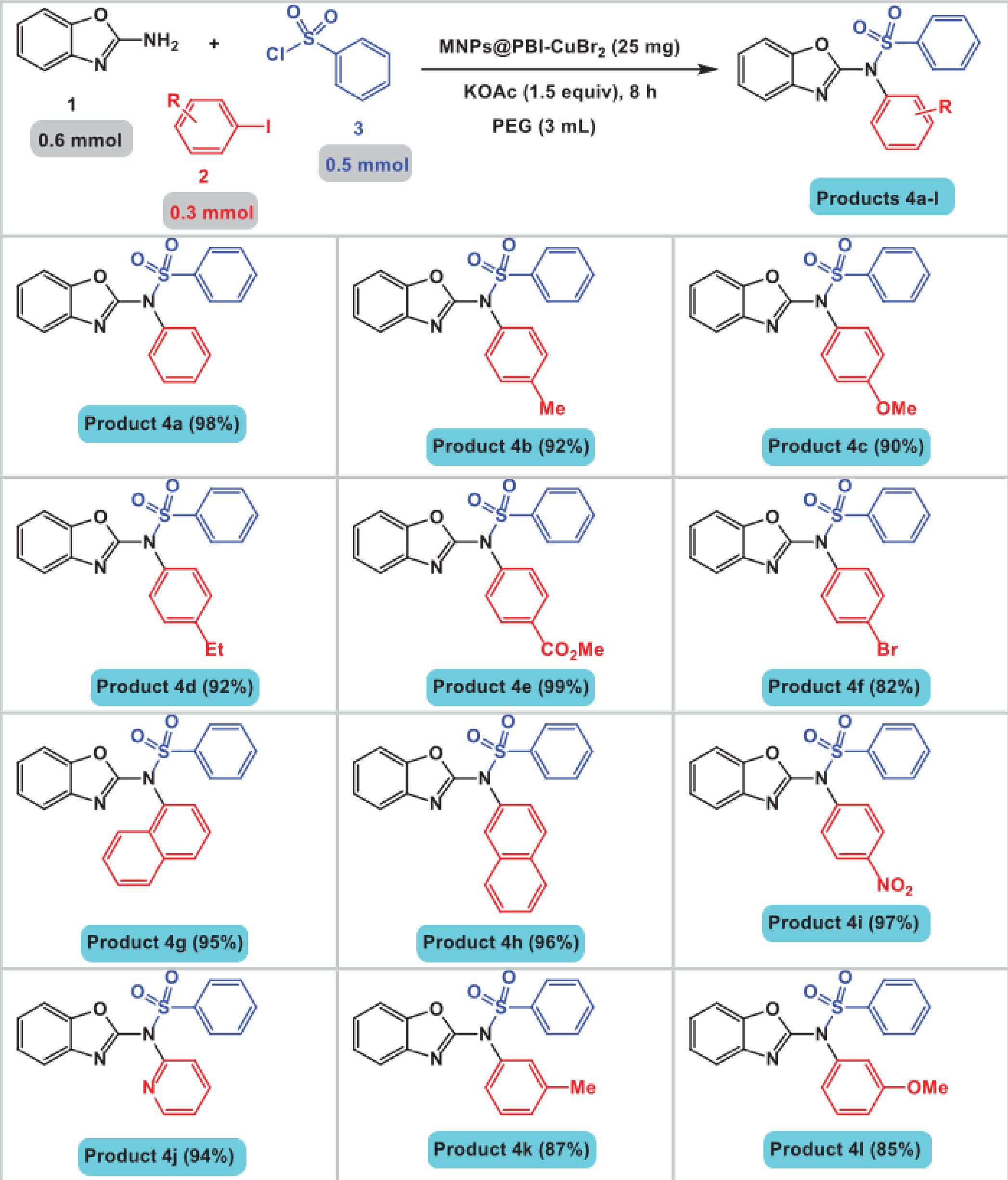


Figure 12. Optimization of the effect of **solvent** on the model reaction (**Product 4a**).

Table 1. Scope of MNPs@PBI-$CuBr_2$ nanocatalyst on the reaction of 2-aminobenzooxazole, sulfonyl chloride and aryl iodides.[a]

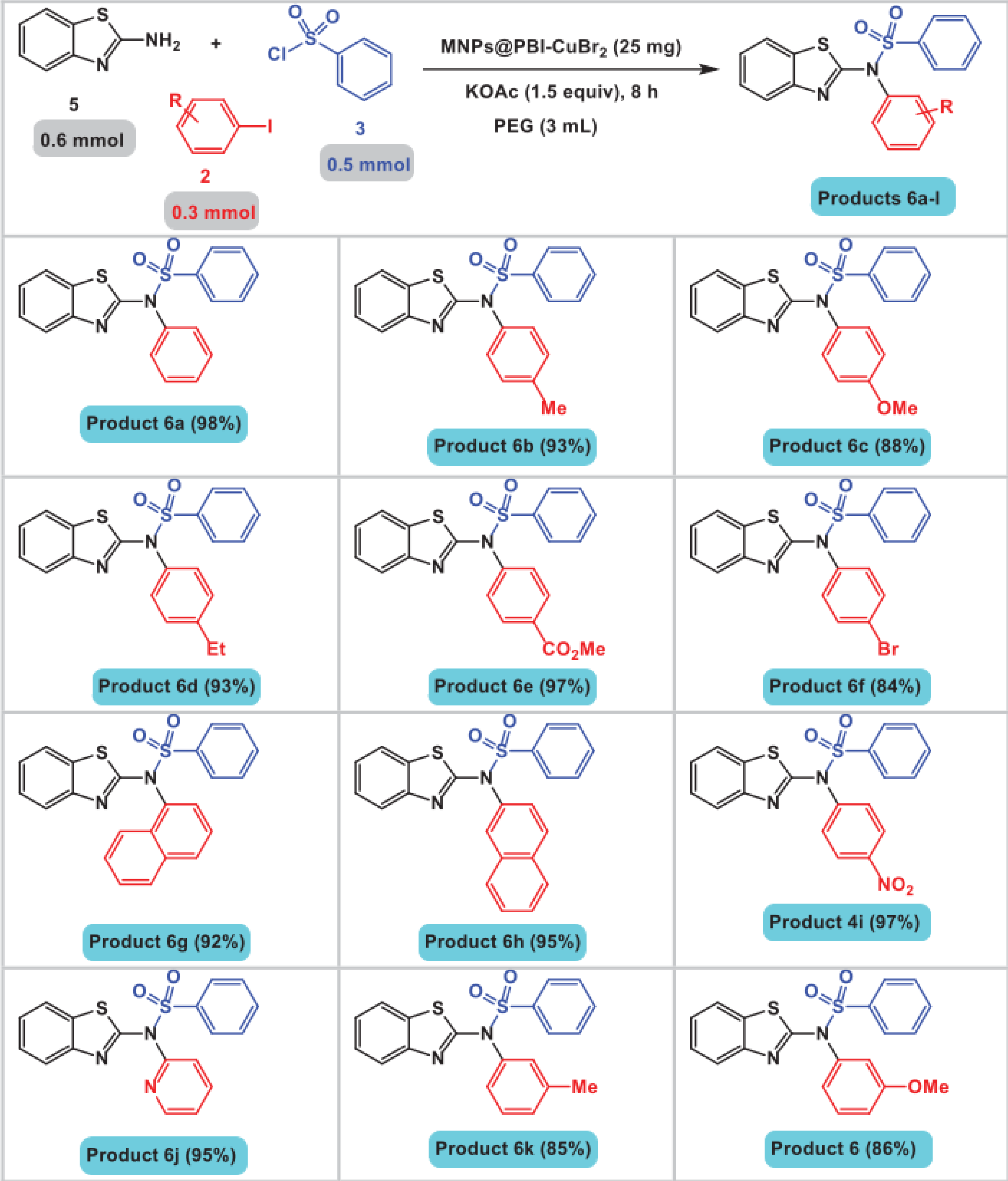


[a]Isolated Yields.

Finally, the model reaction was catalyzed by 25 mg of MNPs@PBI-$CuBr_2$ nanocatalyst and 1.5 equiv of KOAc as base in different solvents to find the best medium conditions (Figure 12). Amongst tested solvents at different temperature, the maximum yield (98%) of the model product was seen in PEG at 120 °C.

Therefore, the utilization of 25 mg of MNPs@PBI-$CuBr_2$ nanocatalyst and 1.5 equiv of KOAc as base in PEG at 120 °C for 8 h was selected as the optimal conditions. With the standardized conditions in hand, the reaction of a broad range of aryl iodides with 2-aminobenzoazoles and

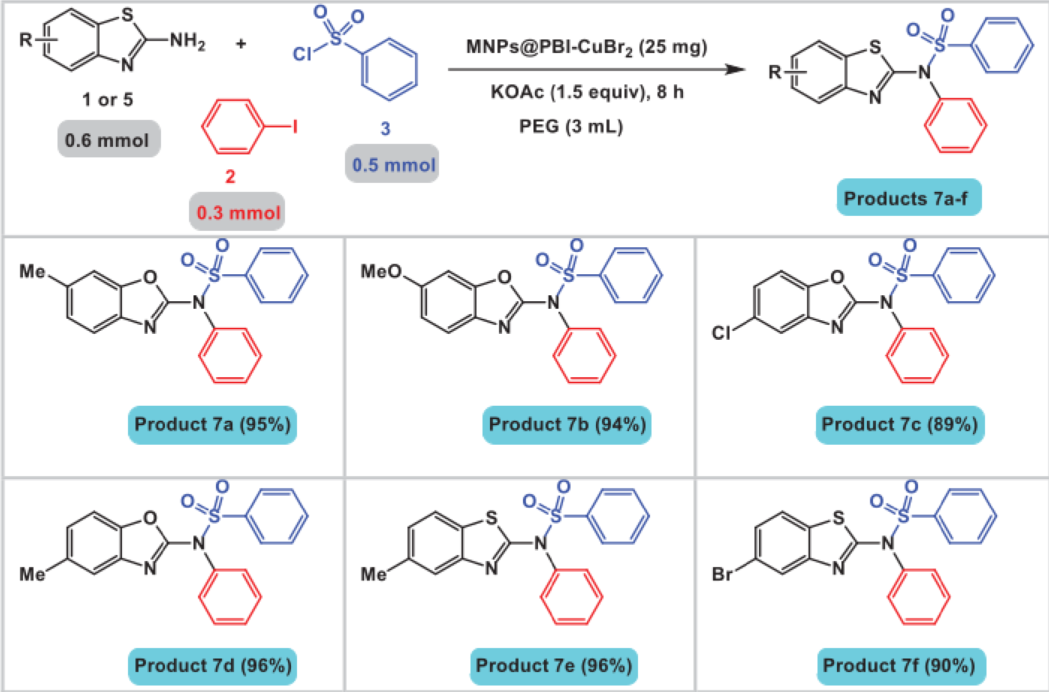
Table 2. Scope of MNPs@PBI-$CuBr_2$ nanocatalyst on the reaction of 2-aminobenzothiazole, sulfonyl chloride and aryl iodides.[a]



[a]Isolated Yields.

sulfonyl chloride in order to synthesize the N-aryl sulfonamides is evaluated which the results are listed in Tables 1 and 2. The results confirmed that MNPs@PBI-$CuBr_2$ is a highly efficient catalyst for the synthesis of N-aryl sulfonamides, because the target products are synthesized with good to excellent yields. Also, the presence of substation on the ring has not significant effect on the efficiency of reactions. Also, we studied the scope of 2-aminobenzoazoles in the reaction with sulfonyl chloride and iodobenzene under the optimized conditions. As shown in Table 3, the target products can be successfully synthesized with high yields.

Table 3. Scope of MNPs@PBI-$CuBr_2$ nanocatalyst on the reaction of 2-aminobenzoazoles, sulfonyl chloride and iodobenzene.[a]



[a]Isolated Yields.

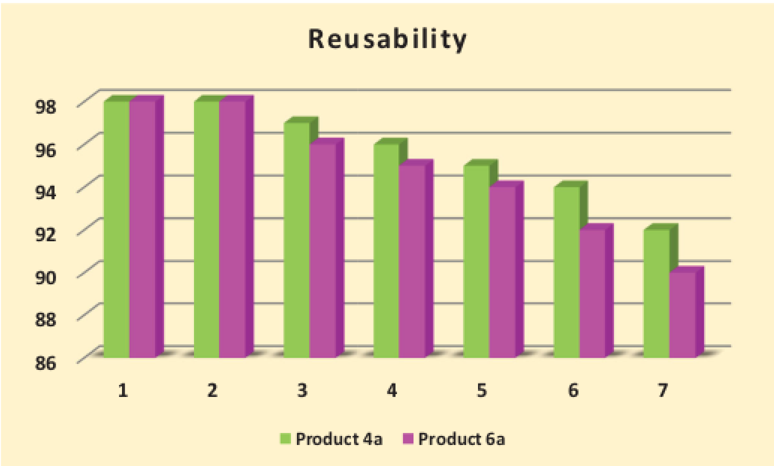


Figure 13. Reusability of MNPs@PBI-$CuBr_2$ nanocatalyst in the synthesis of Products 4a and 6a.

The reusability of catalysts is one of the most important and attractive factors from view point of green chemistry. Therefore, we studied the reusability of MNPs@PBI-$CuBr_2$ nanocatalyst in the synthesis of model reactions (**Products 4a and 6a**). When the reaction was completed, the magnetic nanoparticles were easily removed using a magnet, washed with ethyl acetate several times and then dried to be reused. The recovery feature of the MNPs@PBI-$CuBr_2$ nanocatalyst is shown in Figure 13. The TEM, XRD pattern and ICP-OES spectroscopy of catalyst after recycling were studied and the results were similar to before of utilization. The XRD and TEM analyses

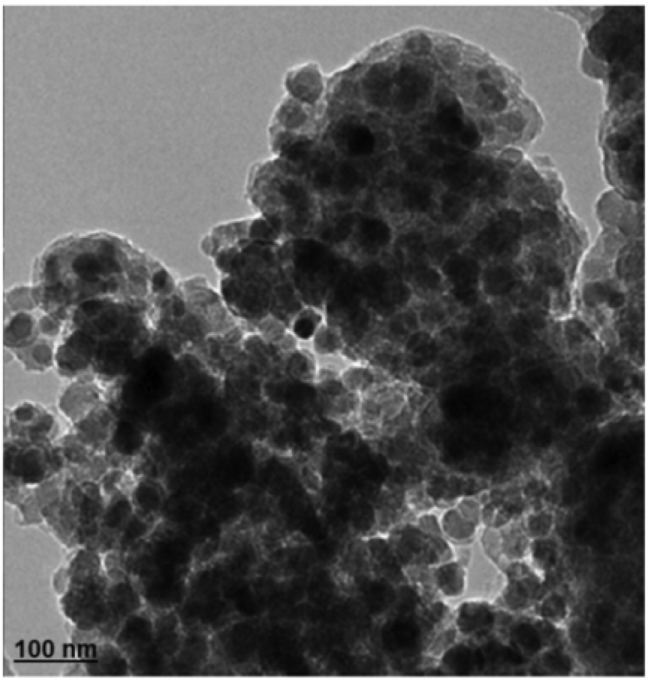


Figure 14. TEM image of the recovered MNPs@PBI-$CuBr_2$ nanocatalyst after 7 runs.

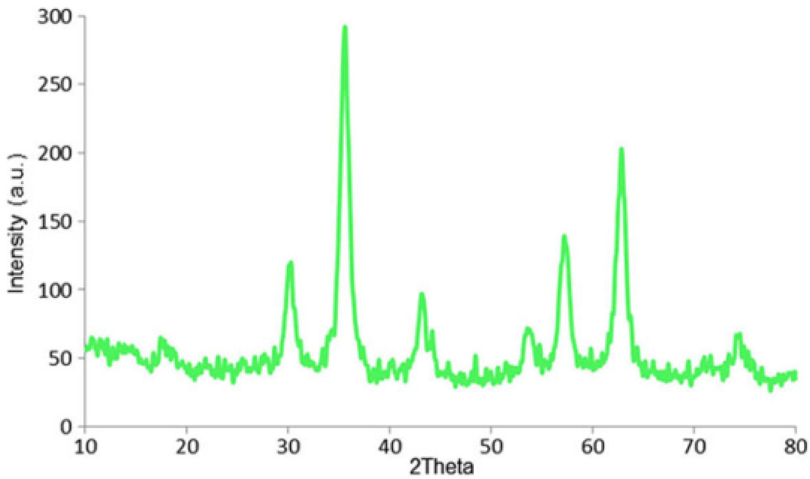


Figure 15. XRD analysis of the recovered MNPs@PBI-$CuBr_2$ nanocatalyst after 7 runs.

Table 4. Comparison of the current method with the previous literature for the synthesis of **product 6a**.

Entry	Conditions	Time	Yield	Reference
1	Pyridine, Ac_2O, 80°C	24 h	71%	[39]
2	Pyridine, EtOH, 60°C	12 h	78	[40]
3	$Cu(OAc)_2$, EtOH, RT	24 h	61	[41]
4	$FeCl_3$, $NaHSO_3$, DMSO, 60°C	12 h	83	[42]
5	**MNPs@PBI-$CuBr_2$, KOAc, PEG, 120°C**	**8 h**	**98**	**This work**

showed that the structure and shape of the recovered MNPs@PBI-$CuBr_2$ catalyst did not change after 7 times of use (Figures 14 and 15). Inductively coupled plasma optical emission spectroscopy (ICP-OES) to determine the amount of Cu loaded on modified magnetic (after 7 times) was performed and it was found to be 1.11 mmol.g^{-1}.

The major advantages of the present protocol over existing methods can be seen by comparing our results with those of some reported procedures, as shown in Table 4. The results for the synthesis of **Product 6a** as representative example, is compared with the best of well-known data from the literature. As shown in Table 4, the present method comparatively affords an absolutely green process, and these results clearly highlight the efficiency of the proposed methodology.

Conclusion

In this article, we clearly shown that $CuBr_2$ immobilized on the surface of magnetic Fe_3O_4 nanoparticles modified with 2-Pyridine Benzoimidazole as ligand (MNPs@PBI-$CuBr_2$) is an ecofriendly and highly efficient reusable nanocatalyst foe the synthesis of N-aryl sulfonamides containing benzoxazole scaffolds through one-pot three-component reaction of 2-aminobenzoazoles, sulfonyl chloride and aryl iodides under ecofriendly conditions. High reusability, simple recovery of catalyst, high yields, mild conditions, performance of reactions in PEG and simple operation are several significant advantages of this methodology. The TEM, XRD pattern and ICP-OES spectroscopy of catalyst after recycling were studied and the results were similar to before of utilization.

Experimental

Chemicals were purchased from Fisher and Merck. The reagents and solvents used in this work were obtained from Sigma-Aldrich, Fluka or Merck and used without further purification. The infrared spectra (IR) of samples were recorded in KBr disks using a NICOLET impact 410 spectrometer. ^{1}HNMR and ^{13}CNMR spectra were recorded with a Bruker DRX-400 spectrometer at 400 and 100 MHz respectively.

Preparation of MNPs@PBI-$CuBr_2$ nanocatalyst

First, magnetic iron nanoparticles were synthesized using previous methods.[43,44] In the next step MNPs-DABA nanocomposite was successfully constructed through the reaction of Fe_3O_4 nanoparticles with 3,4-diaminobenzoic acid at refluxing ethanol for 3 h . The MNPs@PBI-$CuBr_2$ nanocatalyst was fabricated via the immobilization of $CuBr_2$ on the surface of MNPs@PBI ligand (fabricated through the reaction of MNPs-DABA nanocomposite with picolinaldehyde).[45]

General route for MNPs@PBI-$CuBr_2$ catalyzed synthesis of N-arylsulfonamides

A mixture of 2-aminobenzoazoles (0.5 mmol), sulfonyl chloride (0.5 mmol) and MNPs@PBI-$CuBr_2$ (25 mg) in PEG (3 mL) was stirred at 120 °C for 1 h; then, aryl iodide (0.3 mmol) was added to the reaction mixture and stirred for 8 h. The progress of the reaction was monitored by thin layer chromatography (TLC). Then, the reaction was cooled down to room temperature followed by separation of the catalyst by an external magnet. The reaction mixture was then cooled to ambient temperature, diluted with 10 mL of EtOAc. The combined organic extracts were concentrated and the resulting residue was purified by column chromatography on silica gel to obtain the target products.

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Disclosure statement

No potential conflict of interest was reported by the author(s).

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