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Research Article

Biological and Molecular Chemistry

# Investigating the Synthesis of Nanocatalyst $Fe_3O_4$@Arginine-Pd(0) and its Application in the Synthesis of Sulfonamide Derivatives with Antimicrobial Properties

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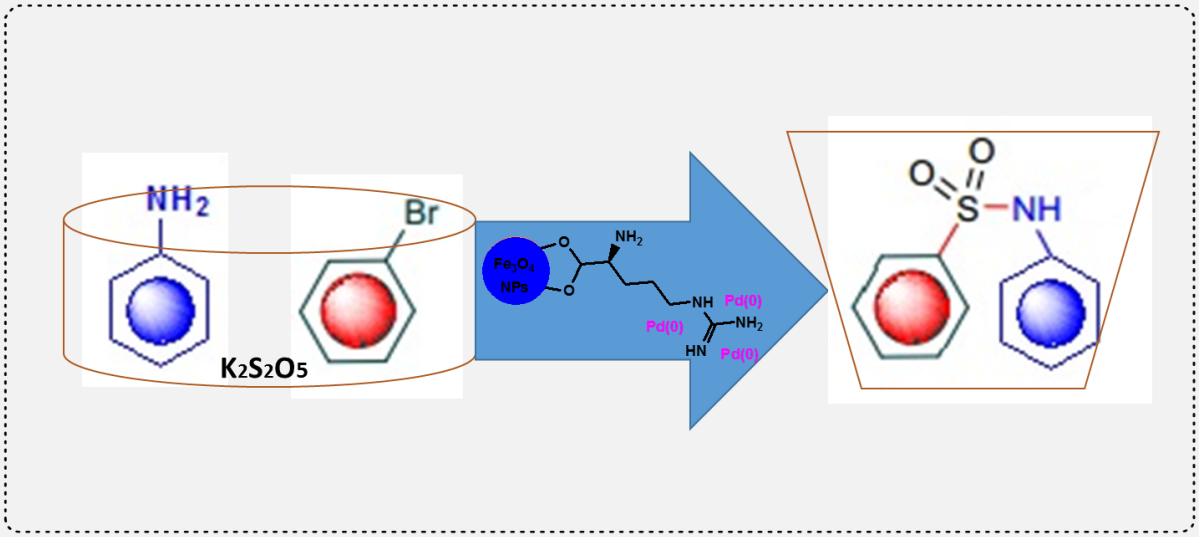
**ABSTRACT:** Sulfonamide drugs were the first effective antibacterials that were used systemically and paved the way for the antibiotic revolution in medicine. Sulfonamide is a functional group that is the basis of several groups of drugs. The main antibacterial sulfonamides are synthetic (non-antibiotic) antimicrobial agents that contain the sulfonamide group. Some sulfonamides also lack antimicrobial activity. In this project, sulfonamides were synthesized from the reaction of bromoaryl and aniline derivatives in the presence of catalyst $Fe_3O_4$@Arginine-Pd(0) in the green solvent of n-hexane at a temperature of 60 °C. The orientation of this catalyst was obtained by FT-IR and TGA analysis, which confirms the successful synthesis of this catalyst. This catalyst has the ability to be regenerated and it works for at least 8 consecutive cycles without reducing the catalytic activity.




**KEYWORDS:** Sulfonamide, Antibacterials, non-Antibiotic, $Fe_3O_4$@Arginine-Pd(0), Bromoaryl.

## Introduction

Sulfonamides are a group of medicinal compounds that show a wide spectrum of biological activities. Today, more than 30 drugs including this agent group have been used in clinics [1]. Among the general features of sulfonamides, we can mention their medicinal and biological properties, their use in synthesis and their use as ligands [2]. Sulfonamide is a compound that contains the functional group $NS(=O)_2$. The

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laboratory synthesis of these compounds is possible in various ways due to their wide range [3]. Sulfonamides form an important group of compounds in medicine and medicinal chemistry with several biological applications. Among known pharmacological agents, sulfonamide drugs have been used for the first time to treat and prevent bacterial infection in humans [4]. Sulfonamides play a very important role as a key component in a number of biologically active molecules [5]. Sulfonamides show a wide range of medicinal properties, including antimicrobial, antibiotic, anti-chronic bacterial infections and anti-fungal, anti-convulsant, diabetes and gout control, anti-tumor, anti-viral, cancer control, anti- Malaria, treatment of brain tumors, anti-Salek, anti-AIDS virus, control and treatment of blood pressure through potassium control, anti-sterility of men and medicinal action against hepatitis C and A [6]. Sulfonamide drugs have been used in the treatment of rheumatic arthritis and control of enzyme activities. The most important medicinal property of sulfonamides is their antibacterial properties[7]. The antibacterial activity of sulfonamides comes from a relatively simple fact that is related to the structural similarity of para-aminobenzoic acid with the related sulfonamide [8]. Enzymes in pathogenic bacteria confuse the sulfonamide compound with para-aminobenzoic acid, which is an important metabolic product [9]. So that in a metabolic conflict, sulfonamide competes with para-aminobenzoic acid to obtain reactive sites on the enzyme [10]. The medicinal efficiency of a sulfonamide and its effectiveness depend on the nature of the group attached to the amino nitrogen [11]. This group should give good acid strength to sulphonamide hydrogen and acid strength is only one of the interfering factors [12]. Sulfonamide compounds in their protonated form have more biological activity because they act in neutral or alkaline biological environments such as the intestine [13]. The solubility of these drugs in water is very low and sometimes it can be deposited in the kidneys, in which case patients are advised to take the drug with a lot of water [14]. Studies show that when the PKa of a sulfonamide drug is around 10, it is more likely to be deposited in the kidneys, and sulfonamide drugs with a PKa of around 5-6 are more favorable [15]. Of the hundreds of sulfonamide compounds that have been synthesized so far, only a few of them have the right combination of high antibacterial activity and low toxicity to humans, which are the characteristics of any effective drug, and almost all of them have a nitrogen-bound sulfonamide group belonging to It is a heterocyclic ring [16]. Sulfonamides can be used as protecting groups for amino groups. In addition, it has been reported in the preparation of -N-sulfonyl aldiamines, -N-sulfonyl pyrroles, indoles and carobazoles and the formation of C-C bond in aqueous medium using sulfonamides [17]. Useful organic herbicides and pesticides have also been prepared from sulfonamides. In order to improve color stability on cellulose fibers, sulfonamide derivatives have been used in the structure of diazo dyes [18]. Sulfonamides have been used as intermediates in the preparation of other organic molecules. The first sulfonamide was produced in 1906, which was used indefinitely in the paint industry, and its medicinal properties were not known [19]. Later, the commercial sulfonamide Prontosil, which was a prodrug, was discovered. This medicine was able to effectively affect a range of bacterial infections inside the body [20]. It should be noted that this drug had no effect on the bacteria inside the test tube and its antibacterial activity was limited to the biological environment [21]. Experiments with Prontosil started in 1932 in the laboratories of the Bayer company and were tested on animals under the supervision of Domak, a research doctor. For this reason, this doctor won the Nobel Prize in Medicine and Physiology in 1939 [22] (**Figure 1**).



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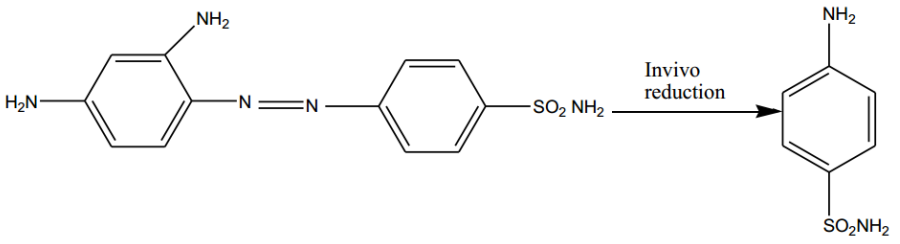


Prontosil Sulfanilamide

**Figure 1.** Conversion of prontosil to sulfonamide.

To increase the activity and selectivity of catalysts, the manufacturers of heterogeneous nanocatalysts are trying to increase their lifetime and recycle and reduce the energy consumption of nanocatalysts [23]. As a result of the low active catalytic surface of many catalysts, they are unable to efficiently catalyze reactions. By immobilizing metals on the substrate, the surface and efficiency of the catalyst increases [24]. Due to the uniform structure and ordered nature, properties such as size, shape, functional groups, and functional groups of mesoporous solid substrates, organic silica nanomaterials are organic-inorganic hybrids. Porous organic silica nanomaterials are used more widely as a substrate for various catalysts [25].For this purpose, in this article, we describe the synthesis of a new palladium catalyst containing Schiff donor base ligands immobilized on modified magnetic nanoparticles. To synthesize a new catalyst for the synthesis reaction of antimicrobial sulfonamides, we combined palladium on arginine substrate to increase thermal stability, magnetic separation, and increase catalytic activity.

## ■ Results and Discussion

We show the efficiency of a novel Pd nanomagnetic catalyst for the synthesis of sulfonamides via the reaction between amine and aryl bromide. As shown in **Scheme 1**, $Fe_3O_4$@Arginine-Pd(0) nanocomposites can be prepared schematically. The structure of $Fe_3O_4$@Arginine-Pd(0) nanocatalyst was determined using various spectroscopic techniques.



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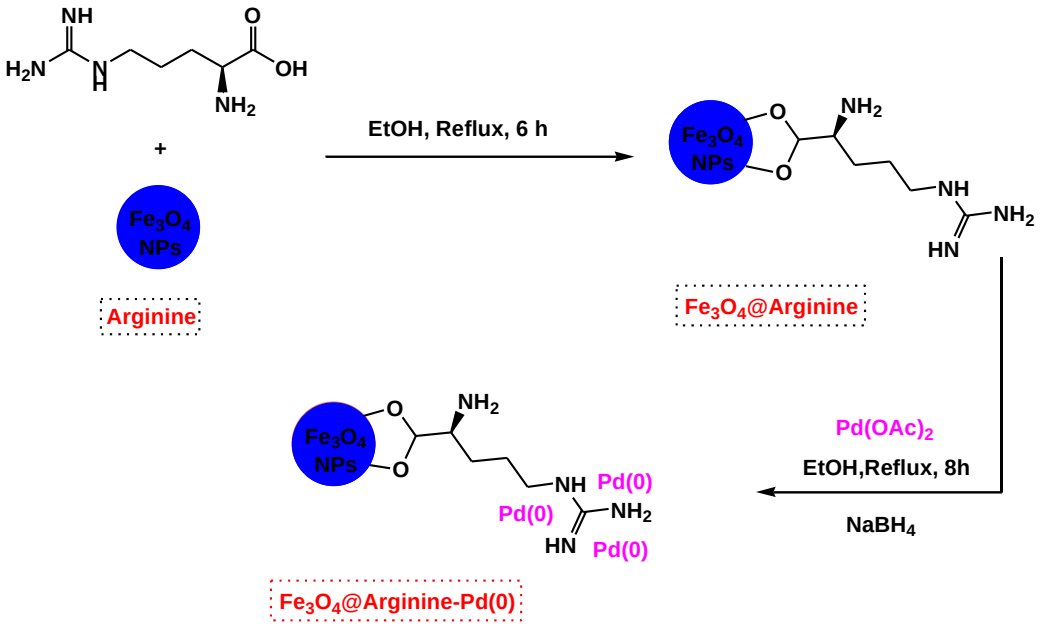


**Scheme 1.** Experimental details of the fabrication of $Fe_3O_4$@Arginine-Pd(0) nanocatalyst.

**FT-IR spectra**

FTIR spectrum of $Fe_3O_4$@Arginine-Pd(0) nanocatalyst is shown in **Figure 2.** The strong and broad peak in the range of 3000-3300 $cm^{-1}$ was assigned to the stretching vibration of N-H bonds, to N-H groups involved in H-bonding or to the presence of O-H groups due to water absorption. C=N stretching vibration can be seen at 1600 $cm^{-1}$. The peaks of 100-1300 $cm^{-1}$ are attributed to the stretching vibration of C-N bonds.

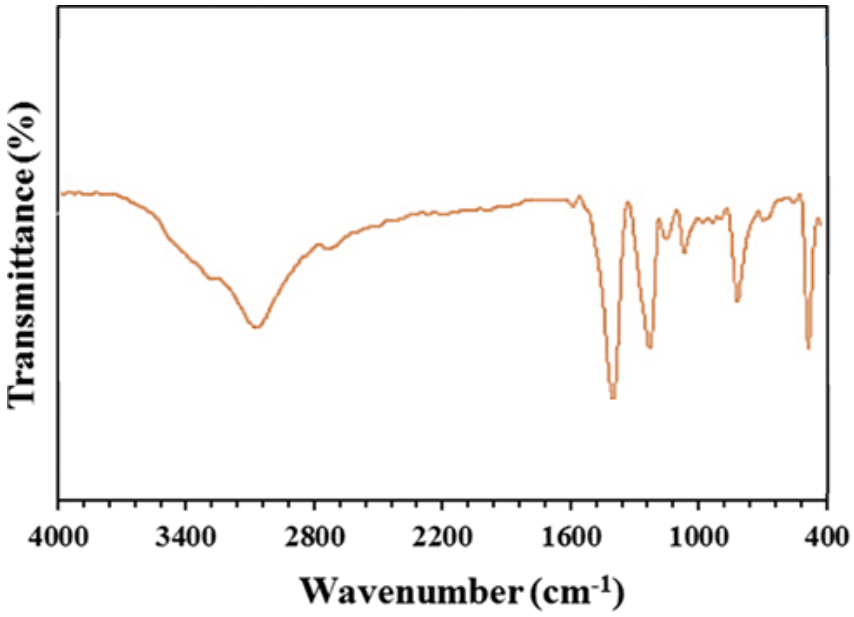


**Figure 2.** FT-IR spectra of $Fe_3O_4$@Arginine-Pd(0) nanocatalyst



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**TGA analysis**

**Figure 3** shows the thermal stability of synthesized $Fe_3O_4$@Arginine-Pd(0) in the range of 0-800 °C. As can be seen, with the increase in temperature from 100 to 200 °C, the weight ratio gradually decreased, which is most likely related to the removal of water absorbed on the surface of $Fe_3O_4$@Arginine-Pd(0). Another weight loss is observed in the range of 200–400 °C, which is attributed to the dissociation of L-arginine from the structure. Finally, another weight loss in the range of 400 to 700 °C is due to the decomposition of other functional groups on the surface of this nanocatalyst.

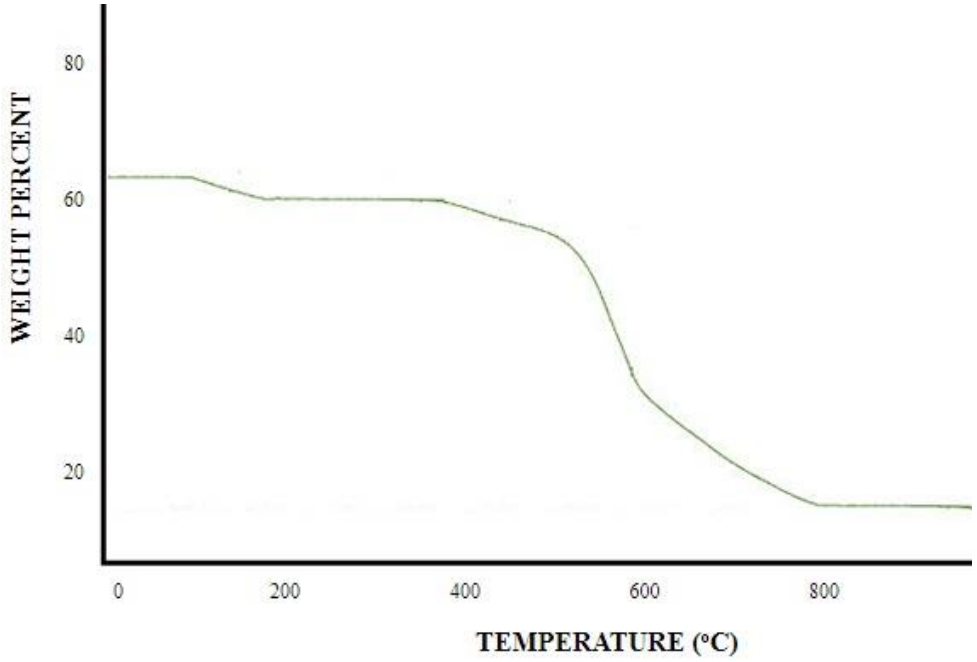


**Figure 3**. TGA analysis of $Fe_3O_4$@Arginine-Pd(0) nanocatalyst

To find the optimal solvent, the reaction of bromobenzene with aniline has been carried out in different solvents, the results of which are given in **Table 1**. The reaction in polar and non-polar solvents had different results. The n-hexane solvent has been chosen as a solvent for other investigations due to its lower amounts of by-products and high efficiency. Moreover, this reaction did not produce any product under catalyst-free conditions. The reaction was carried out with different amounts of catalyst, the best result being 15 mg. Also, different temperature conditions were tested for this reaction. The best temperature for this reaction is 60 °C. Therefore, the reaction of bromobenzene with aniline in green solvent n-hexane and temperature of 80 degrees has been selected as superior.



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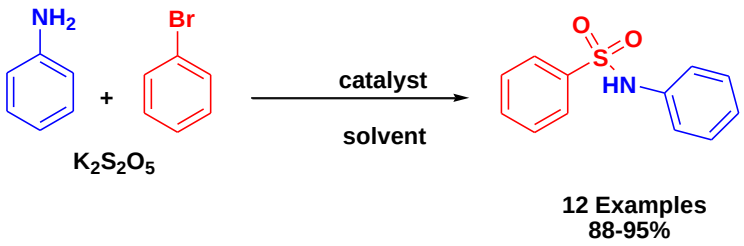
**Table 1.** Optimization parameters for the reaction of bromobenzene with aniline in the presence of $Fe_3O_4$@Arginine-Pd(0) nanocatalyst[a]




| Entry | Catalyst (mg) | Solvent | Tem (°C) | Yield (%) [a] |
|---|---|---|---|---|
| 1 | -- | n-Hexane | 60 | No |
| 2 | 5 | n-Hexane | 60 | 58 |
| 3 | 10 | n-Hexane | 60 | 81 |
| **4** | **15** | **n-Hexane** | **60** | **95** |
| 5 | 15 | n-Hexane | 40 | 53 |
| 6 | 15 | PEG | 60 | 63 |
| 7 | 15 | EtOH/Water | 60 | 74 |
| 8 | 15 | DMSO | 60 | 41 |
| 9 | 15 | DMF | 60 | 35 |
| 10 | 15 | $C_2Cl_2$ | 60 | 69 |
| 11 | 15 | EtOH | 60 | 77 |
| 12 | 15 | $H_2O$ | 60 | 80 |

[a] Isolated yields

After obtaining the optimal conditions, the reaction with different derivatives of aryl bromide and amine electron-donating and killing groups was investigated (**Table 2**). As shown in the table, the reaction was carried out under mild conditions that provided the corresponding products in good to high yields. Different electron donating and withdrawing groups were well accepted on aniline and aryl bromide. Furthermore, the products with electron withdrawing groups reacted well under these conditions and produced their corresponding sulfonamides with high efficiency.



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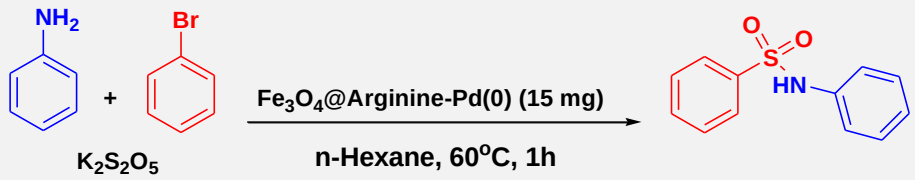
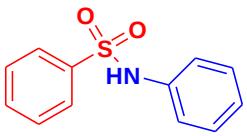
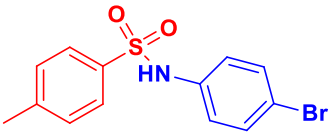
**Table 2.** Scope of reaction of p-toluenesulfonyl chloride and amine in the presence of $Fe_3O_4$@Arginine-Pd(0) nanocatalyst [a]

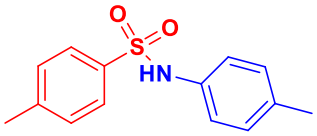


4a (95%)

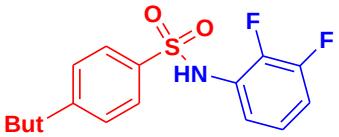



4b (90%)

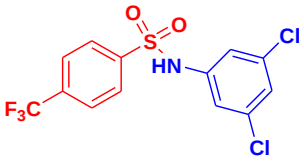


4c (89%)

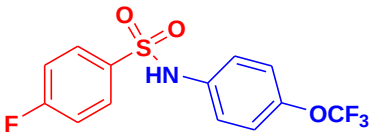

4d (88%)



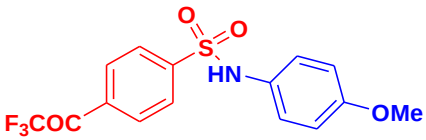

4e (91%)



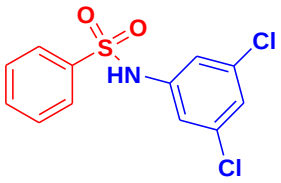

4f (93%)




4g (94%)

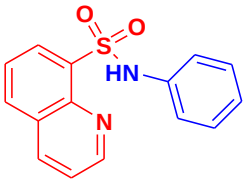



4h (93%)

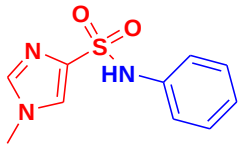



4i (89%)


4j (93%)




4k (92%)




4l (90%)

[a] Isolated yields

**Scheme 2** shows the proposed mechanism for the formation of sulfonamides using $Fe_3O_4$@Arginine-Pd(0) magnetic catalyst.



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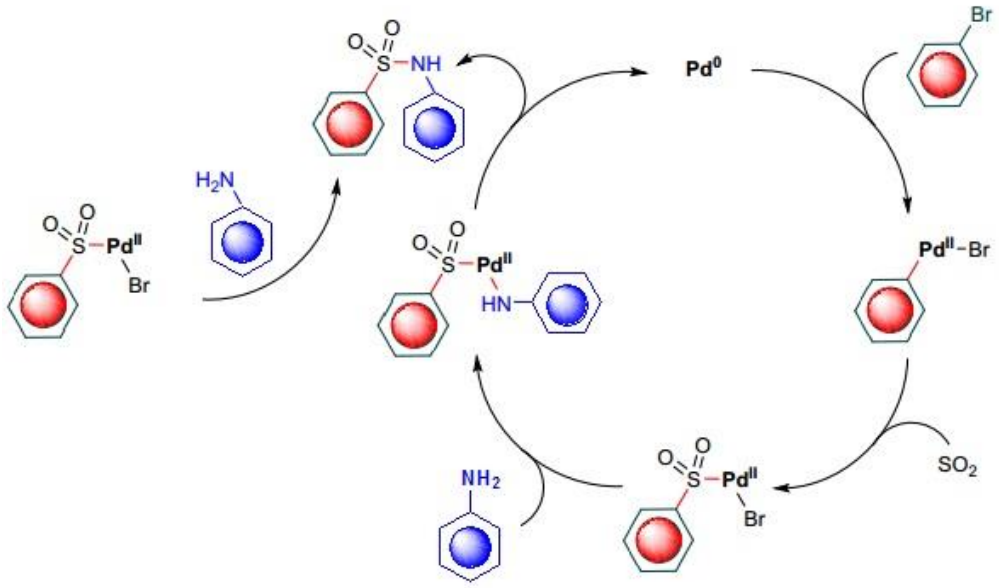


**Scheme 2 .**Proposed mechanism for the preparation of products 4a-l.

The capacity of a heterogeneous catalyst for recycling is essential for its effectiveness. One of the main disadvantages of Pd is its high cost, but since it can be recycled and used repeatedly for this purpose, this limitation can be reduced to some extent. As **Figure 4** shows, Catalyst $Fe_3O_4$@Arginine-Pd(0) has the capability of at least 8 cycles of reduction in which there is a significant reduction. Therefore, it has maintained its high activity and selectivity.

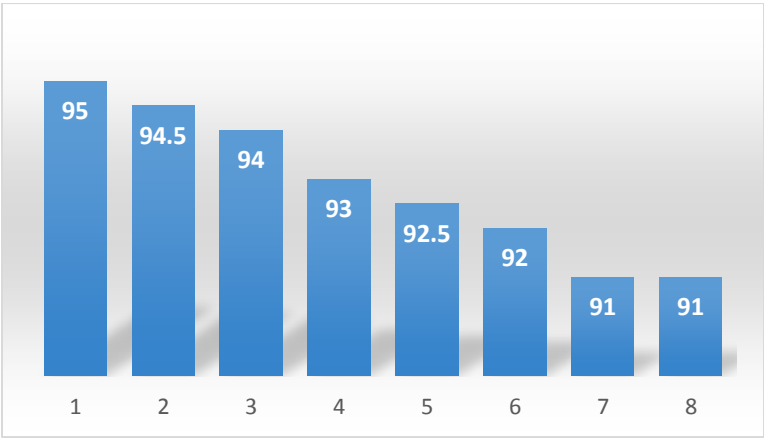


**Figure 4**. Reusability of $Fe_3O_4$@Arginine-Pd(0) catalyst in the synthesis of product 4a.

## ■ Conclusion

In this research, the biological effects of sulfonamide compounds were investigated in the presence of the magnetic catalyst-$Fe_3O_4$@Arginine-Pd(0). New sulfonamide compounds were synthesized and identified. According to the studies that show that the presence of electron-donating and electron-withdrawing



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functional groups in these compounds play an important role in antimicrobial activities. In general, the results obtained from this research show that this catalyst shows a high level of stability and can be It was used at least 8 times without loss of catalytic activity. The recycling test and The hot filtration test clearly confirmed that $Fe_3O_4$@Arginine-Pd(0) was indeed heterogeneous in nature.

## Experimental

### Chemical synthesis

Sigma-Aldrich or Merck chemical companies provided the chemicals and solvents used in this study without further purification. The ICP analysis was performed using an inductively coupled plasma analyzer (Varian Vista-Pro). The surface area and pore volume of the samples were determined at liquid nitrogen temperature. Under vacuum, the samples were degassed at 200 C for 5 h before measurements. $^1$H-NMR and $^{13}$C-NMR spectra were made on a 500 MHz Bruker Advance III spectrometer in $CDCl_3$ as solvent. The course of the reaction was followed by thin layer chromatography (TLC) on silica gel polygrams SIL G/UV 254 plates.

### Preparation of $Fe_3O_4$@Arginine-Pd(0) catalyst

First, magnetic iron nanoparticles were synthesized using previous methods [26]. In the next step, $Fe_3O_4$@Arginine-Pd(0) nanocomposite was successfully synthesized through the reaction of $Fe_3O_4$-NPs with arginine in ethanol solvent under reflux conditions for 6 hours. Finally, palladium acetate (II) $(PdOAc)_2$ was successfully immobilized on the $Fe_3O_4$@Arginine-Pd(0) ligand in ethanol solvent under reflux conditions for 8 hours in the presence of $NaBH_4$.

### General procedure for the preparation of compounds

For sulfonamide synthesis starting from aryl bromide (240 mg, 1.0 mmol, 1.0 equiv), $K_2S_2O_5$ (267 mg, 1.2 mmol, 1.2 equiv), $Fe_3O_4$@Arginine-Pd(0) (15 mg, 0.02 mmol, 0.02 equiv) and amine (123 mg, 1.0 mmol, 1.0 equiv) were prepared at 80 °C. Purification was performed by column chromatography on silica gel to give the desired compound 4a (361 mg, 0.80 mmol, 95% ) to be provided.

### Supporting Information

N-(4-methoxyphenyl)-4-(2,2,2-trifluoroacetyl)benzenesulfonamide (4h): Yellow solid, mp 98 - 100$^o$C. $^1$H NMR (500 MHz, CDCl3): δ 3.84 (s, 3H, OMe), 6.90 (d, 2H, J= 8.6 Hz, CHAr), 6.95 (d, 2H,J= 8.8 Hz, CHAr), 7.54 (d, 2H, J= 7.5 Hz, CH Ar), 7.64 (t, 2H J= 7.9 Hz, CHAr), 7.94 (d, 2H,J= 7.5 Hz, CHAr). $^{13}$C{1H} NMR (126 MHz, CDCl3): δ 55.9, 114.8, 120.1 , 121.3, 125.6, 129.2,129.7, 130.5, 133.0, 140.5, 148.6, 161.9.

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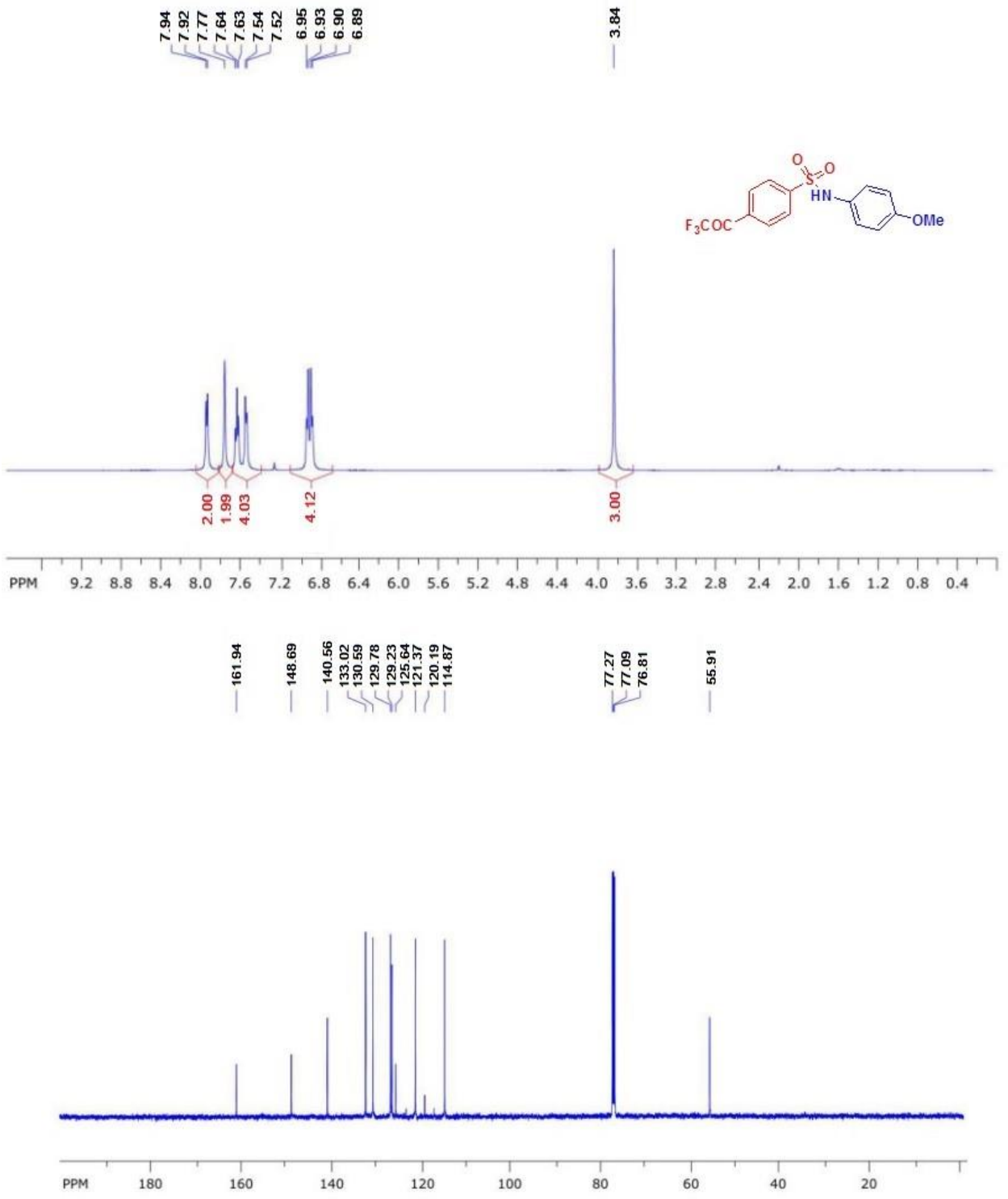
**$^{1}$H and $^{13}$C NMR Spectra of Sulfonamide:**



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