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Synthesis and Characterization of New nanoparticles compound

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Abstract: The development of competent green chemistry methods for synthesis of metal nanoparticles have become a main limelight of researchers in the field of nanotechnology.Nanoparticles are advanced matarials have recently gained high attention due to their scientific and technological important. They find awide variety of applications such as catalysts with huge activity and specificity,metal semiconductor In this thesis, preparation compound ($CuFe_2O_4@SiO_2$-PA-TFA MNPs) synthesized $CuFe_2O_4@SiO_2$ functionalized with propylamine and trifluoroacetic acid. Heterocycle compounds are an important compound of organic chemistry that exist in the structure of natural products and are responsible for the biological and pharmacological properties of most medicinal compounds. Characterization of $CuFe_2O_4@SiO_2$-PA-TFA MNPs was performed by SEM, EDX, XRD, TGA and VSM analysis. Application of these nanoparticles in the synthesis of tetrahydropyrazolopyridines, shown high activity of the catalyst.

Keywords: Trifluoroacetic, $CuFe_2O_4@SiO_2$-PA-TFA MNPs, propylamine

1 –Introduction:

A cyclic compound containing all carbon atoms in ring formation is referred to as a carbocyclic compound. If at least one atom other than carbon, forms a part of the ring system then it is designated as a heterocyclic compound. Nitrogen, sulfur and oxygen are the most common hetero atoms. Heterocyclic rings may contain another hetero atoms. Extensive range of heterocyclic derivatives are known and the number is increasing rapidly. Heterocyclic ring may comprise of three or more atoms which may be saturated or unsaturated(1) The heterocyclic ring may contain more than one hetero atom which may be similar or dissimilar. A large number of heterocyclic compounds are necessary to life. Various derivatives such as antibiotics, alkaloids, amino acids, vitamins, hormones and a large number of dyes and drugs contain heterocyclic moeity. Heterocycles play a vitals role in agricultural and pharmacological and synthetic fields. There is a large number of synthetic heterocyclic compounds with important applications are a valuable intermediates in synthesis [2-6]. Tetrahydrodipyrazolopyridines another group of pyrazole derivatives with properties including HIF 1-αprolyl hydroxylate inhibitors, dopaminergic, anxiolytic, antiallergic, anti-Leishmania and protein kinase inhibitors [7-18]. The structure of Tetrahydrodipyrazolopyridines is demonstrated in (Figure 1).

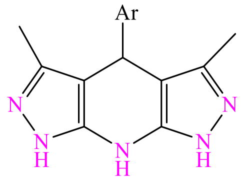

Figure 1: Structure of tetrahydrodipyrazolopyridines

Nano catalysts, new catalysts have been introduced throughout the previous decade. The activity, yield, selectivity, and stability of these nanoparticles are all excellent. As a result, nanocatalysts combine the benefits of homogeneous and heterogeneous systems while also solving the difficulties that plague these catalytic systems. Nanoparticles can also be retrieved and reused several times. Filtration or centrifugation are employed to separate nanoparticles from the reaction mixture, however due to the size of nanoparticles, isolation and recovery of Nano catalyst from the reaction mixture is challenging and time consuming. Magnetic separation was introduced as an efficient approach to overcome the limitations of nanoparticles. An external magnetic field is used to separate magnetic nanoparticles from the reaction medium, which can

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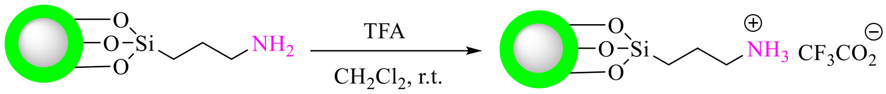
then be recovered and reused various times. low toxicity, High surface area, , high activity, and thermal stability are all characteristics of magnetic nanoparticles (MNPs) [19-21].

2- Materials and Method

Merck chemicals were removed and BDH was employed.With KBr disk, "Testseon Shimadzu TMS as the intarnal standard (Bruker, Ultra Shield 500 MHZ Switzerland).

2.1 Synthesis of $CuFe_2O_4$@SiO_2-PA-TFA magnetic nanoparticles

$CuFe_2O_4$@SiO_2-PA (0.50 g) was dispersed in CH_2Cl_2 (10.0 mL) by sonication for 20.0 min. trifluoroacetic acid (1.50 mL) was added to the mixture and stirred for (3.0 h) for reaction mixture at room temperature. the product ($CuFe_2O_4$@SiO_2-PA-TFA) by an external magnet was extracted and washed several times with CH_2Cl_2 and dried in room temperature (Scheme 1) [22].



Scheme 1: Synthesis of $CuFe_2O_4$@SiO_2-PA-TFA MNPs

3 Results and discussion

The shape and particle size of nanoparticles were determined using scanning electron microscopy (SEM). In Figure 2, SEM pictures of CuFe2O4@SiO2-PA-TFA are shown. These nanoparticles appear to have a spherical form and are nanometer in size, according to these photos.

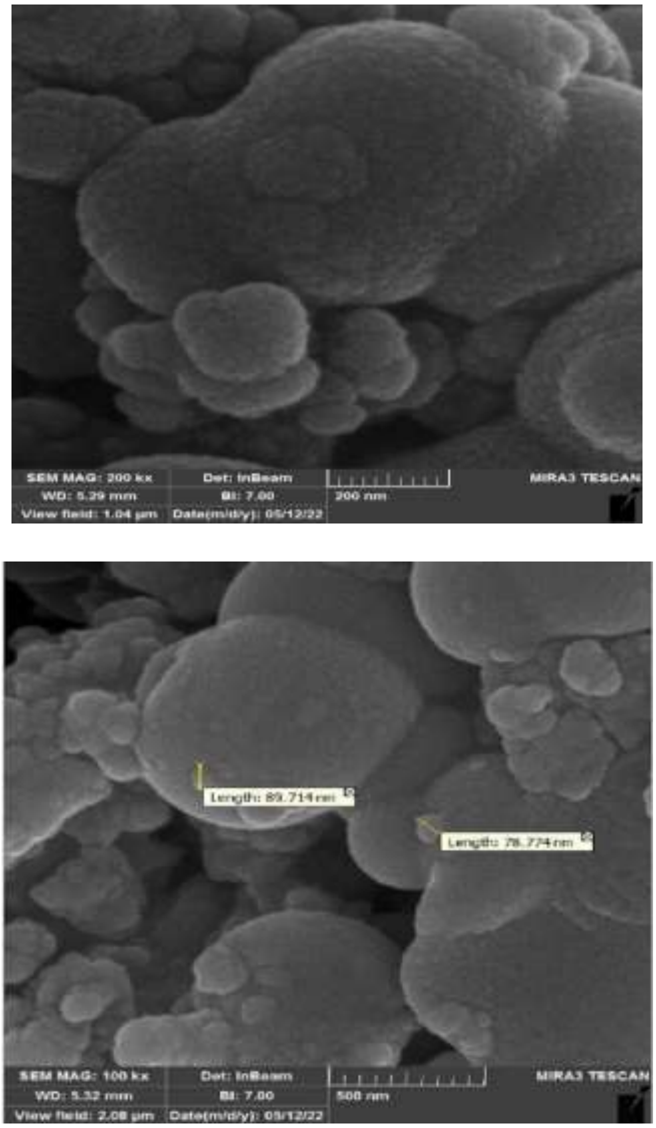


Figure 2: SEM image of $CuFe_2O_4$@SiO_2-PA-TFA MNPs

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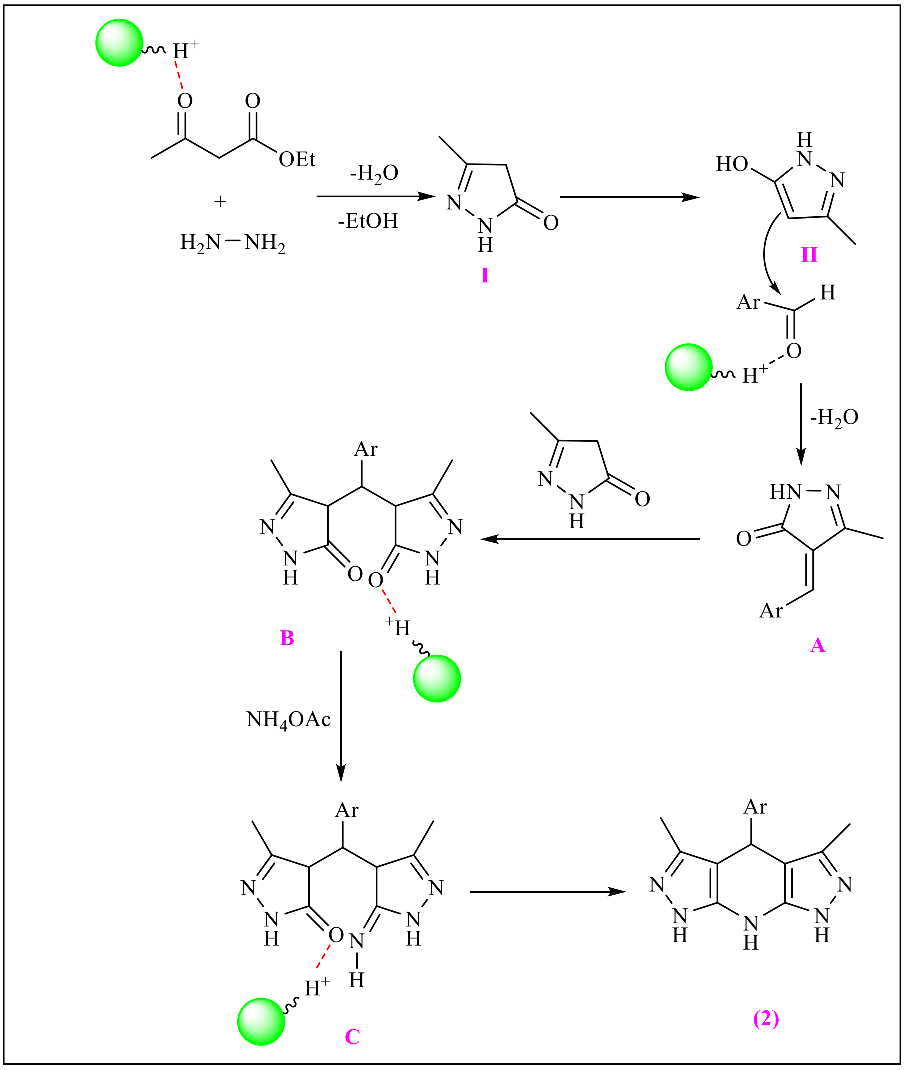
3.1. EDX analysis of $CuFe_2O_4@SiO_2$-PA-TFA MNPs

EDX analysis is one of the appropriate techniques for the description of the elements in the structure of nanocatalysts. According to the EDX analysis of $CuFe_2O_4@SiO_2$-PA-TFA (Figure 3) and EDS mapping (Figure 4), Cu, Fe, Si, O, C, N and F elements are observed in the structure of $CuFe_2O_4@SiO_2$-PA-TFA MNPs that approved the successful preparation of the nanocatalyst.



Scheme 2.mechanism in order to synthesis of tetrahydropyrazolopyridines using $CuFe_2O_4@SiO_2$-PA-TFA MNPs

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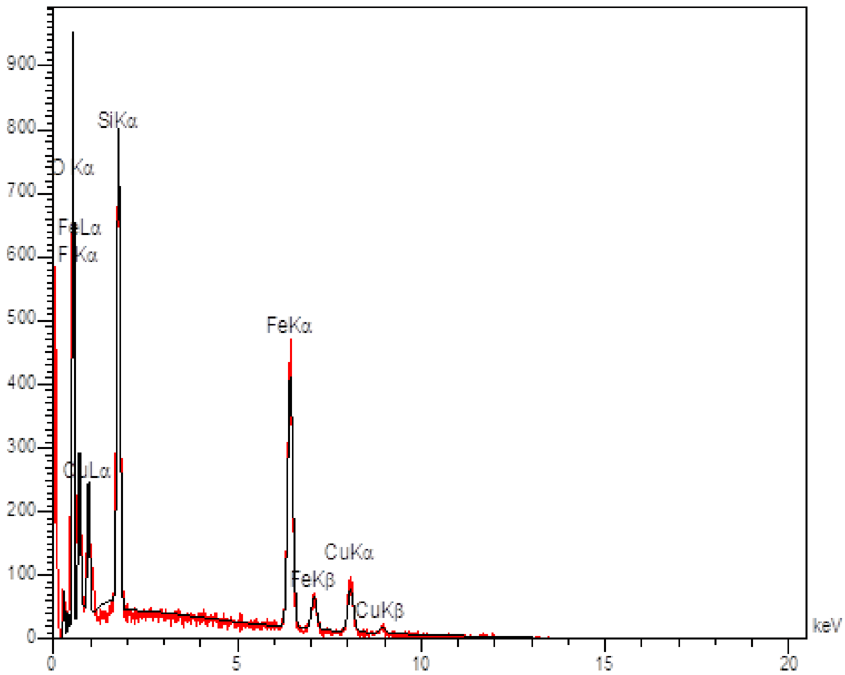
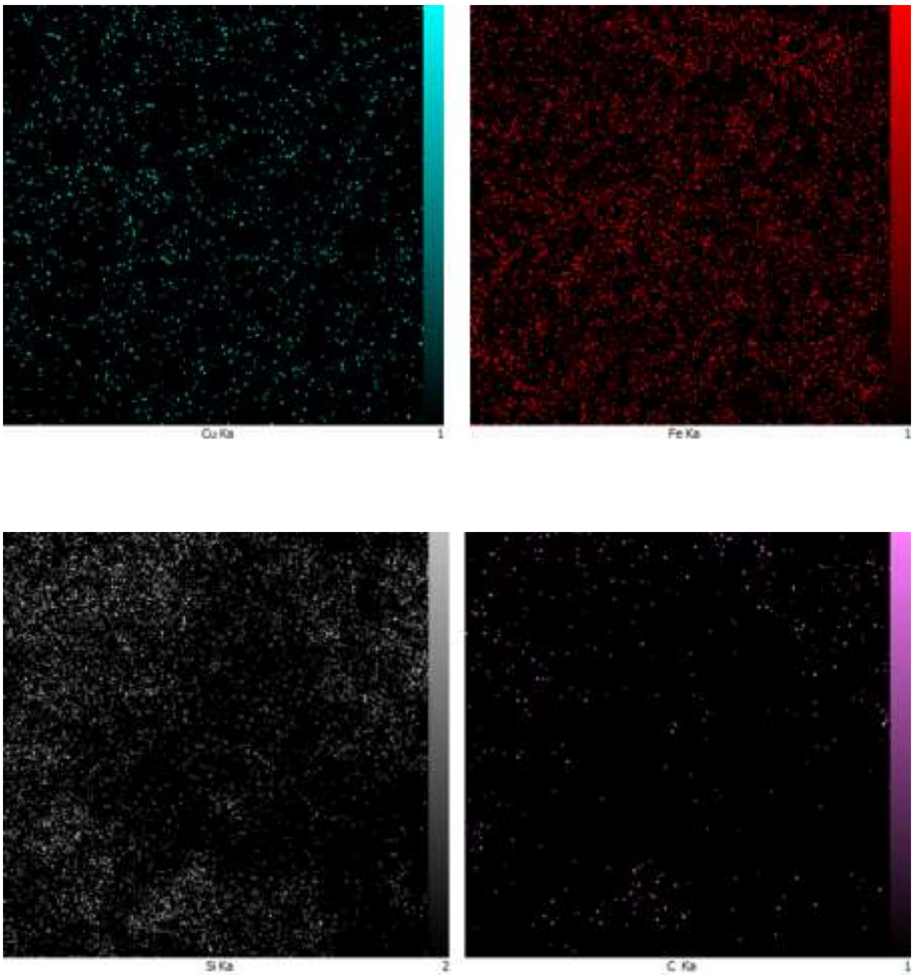


Figure 3: EDX image of $CuFe_2O_4@SiO_2$-PA-TFA MNPs



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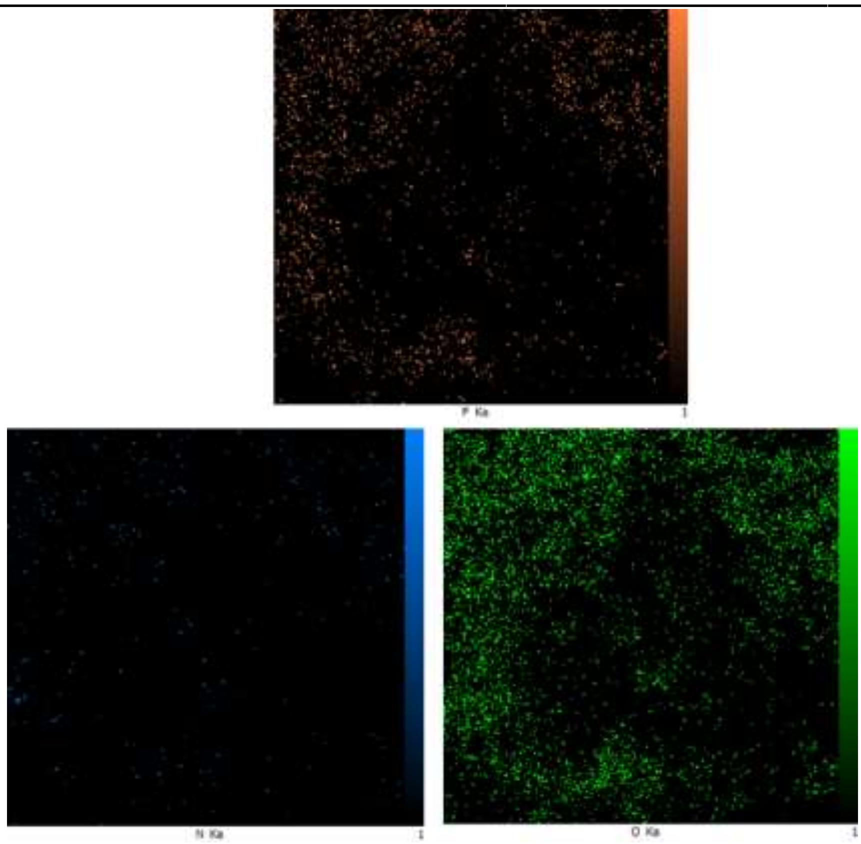

Figure 4. Elemental mapping of $CuFe_2O_4@SiO_2$-PA-TFA MNPs

3.2 XRD analysis of $CuFe_2O_4@SiO_2$-PA-TFA MNPs

XRD analysis was utilized to study the crystalline structure of Nano catalyst. The X-ray diffraction patterns of $CuFe_2O_4@SiO_2$-PA-TFA is shown in Figure 5. This curve shows eight peaks at 2θ = 18.390, 30.040, 35.390, 38.890, 42.990, 53.340, 56.740 and 62.340 that in perfect match with standard pattern and certain the shell of compound $CuFe_2O_4$ MNPs.

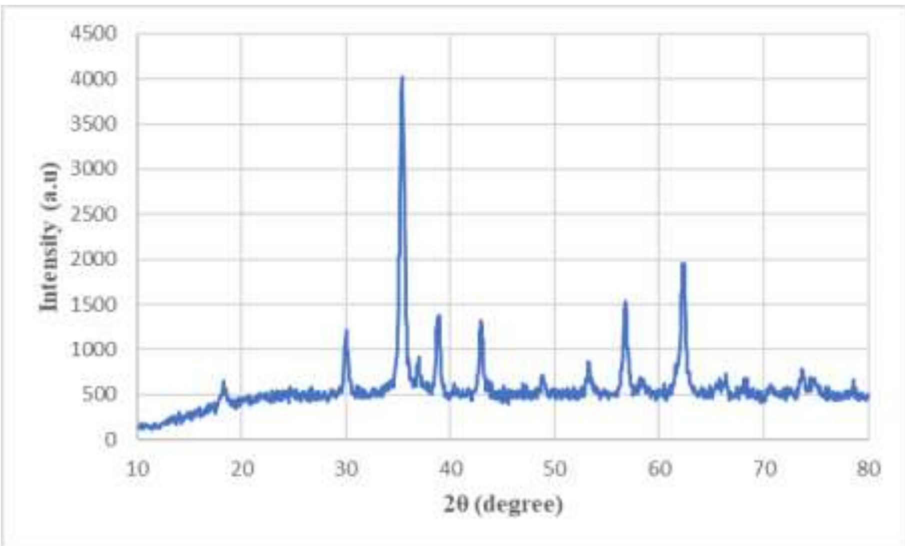

Figure 5: XRD of $CuFe_2O_4@SiO_2$- PA-TFA MNPs

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3.3. TGA analysis of $CuFe_2O_4@SiO_2$-PA-TFA MNPs

The thermal stability of $CuFe_2O_4@SiO_2$-PA-TFA MNPs was studied by TGA analysis. The TGA curve of the $CuFe_2O_4@SiO_2$-PA-TFA MNPs is illustrated in (Figure 6). According to this curve, thermal decomposition happened in (2 steps). The initial weight loss that was observed in the below 200.0 °C related to the removal physical adsorbed solvent and on the surface of the catalyst for OH groups and the next weight loss appeared between (250.0-800.0) °C that related to the decomposition of organic compounds on the surface of the catalyst and confirmed that $CuFe_2O_4$ was functionalized successfully with organic groups.

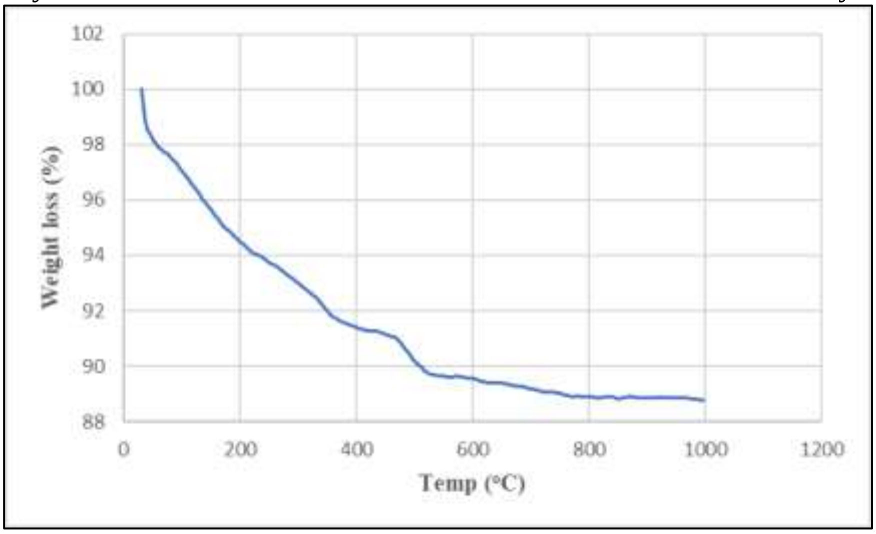

Figure 6: TGA diagram of $CuFe_2O_4@SiO_2$-PA-TFA MNPs

3.4. Magnetic properties of $CuFe_2O_4@SiO_2$-PA-TFA MNPs

VSM analysis was used to study the magnetic characteristics of CuFe2O4@SiO2-PA-TFA. Figure 7 shows the curves VSM for CuFe2O4@SiO2-PA-TFA, and CuFe2O4 which exhibit values of 24 $emug^{-1}$and 33 $emug^{-1}$ for saturation magnetization ,respectively, indicating that saturation magnetization decreases owing to $CuFe_2O_4$ surface functionalization.

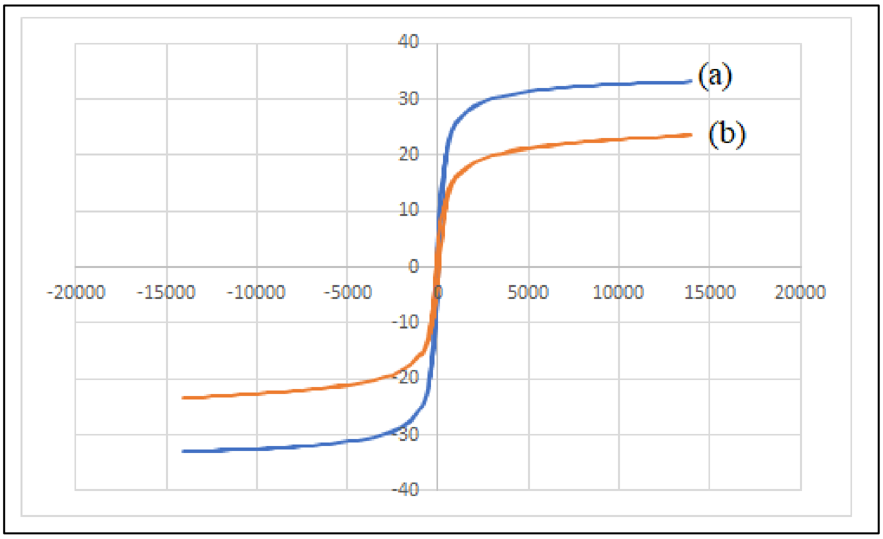

Figure 7: VSM diagram of $CuFe_2O_4$ (a) and $CoFe_2O_4@SiO_2$-PA-TFA (b)

4- Conclusion

Tetrahydropyrazolopyridines were synthesized *via* one-pot four-component reaction of hydrazine hydrate, ethylacetoacetate, ammonium acetate and benzaldehyde derivatives CuFe2O4@SiO2-PA-TFA MNPs were synthesized and evaluated utilizing FT-IR, EDX, XRD, SEM,TGA, and VSM analysis as an unique and efficient nanocatalyst in this work. This nanocatalyst was used in the production of tetrahydropyrazolopyridine derivatives, which yielded good to outstanding yields in a short reaction time.

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CuFe2O4@SiO2-PA-TFA MNPs were also recovered and reused numerous times while preserving their catalytic activity.

Exp.	Catalyst	conditions	Time (min)	Yield (%)
1	$CuFe_2O_4@SiO_2$-PA-TFA	H_2O, r.t.	5	98

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