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IR and SEM-EDX studies on Polymer based mixed Metal (Fe-Mn-Zn) Oxides Nanocomposite Adsorbent for Removing Pb (II) ions from contaminated water

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ABSTRACT

The contamination of fresh water due to heavy metal is a serious problem of the present. The presence of heavy metals in fresh water bodies is continuously increasing due to industrialization and unsafe discharge of waste from these industries. The heavy metals are inorganic pollutants and are non-biodegradable. So, the removal of such type of contamination from fresh water is highly required. Various methods are adopted by researchers to overcome this difficulty. The adsorption is one of the most widely used technique to eliminate the heavy metals from the contaminated water. Among the adsorption methods, the method making use of polymeric magnetic nanocomposites is gaining interest in the researcher's community. The advantage of polymeric nanocomposites lies in their easy separation from the aqueous system by applying external magnetic field. In the present study we have synthesized nanocomposites of mixed metal oxides of (Fe-Mn-Zn) in the copolymer matrix of aniline-formaldehyde. The material has been used to adsorb Pb (II) ions from the aqueous medium. IR and SEM-EDX studies have been used to study the adsorption characteristics of the material. From the IR studies the shift in IR peak positions and variation in the intensities of peaks after adsorption has been observed. This variation suggests the interaction of Pb ions with the copolymer backbone. Further, the SEM study confirmed the formation of nanosized particle of the materials and SEM-EDX study directly confirmed the Pb ions adsorption by these nanocomposites.

Keywords: Heavy metals, polymeric nanocomposites, metal-oxides, IR studies, SEM-EDX studies

INTRODUCTION

In today's world the contamination of freshwater bodies and shortage of clean water are critical issues[1]. The rapid population growth and rapid industrialization to fulfil the needs of the growing population have led to an enormous increase in freshwater consumption as well as water pollution[2]. Both industrial and domestic uses lead to the release of a wide range of toxic organic/ inorganic compounds, including carcinogenic heavy metals into water bodies. Inorganic pollutants in particular heavy metal ions in ground and freshwater bodies, impose a threat to human health and the environment as they are biologically nondegradable[3]. Lead (Pb) is a toxic heavy metal that can cause a range of health problems, including issues with cardiovascular disorders, reproductive problems, kidney damage, high blood pressure, and even an increased risk of high mortality rates as reported by the World Health Organization[4]. As a result, it is critical to effectively remove Pb(II) ions from Pb-contaminated water/wastewater which is generated by industries such as lead smelting, paint manufacturing, battery manufacturing, mining, and municipal waste incineration [5-7]. Different treatment methods such as chemical precipitation, ion exchange, electrolysis, and adsorption that have been explored for Pb(II) removal, adsorption is a highly preferred due to its higher efficiency, lower cost and simpler operationality.[8,9] In the present study we report the synthesis of magnetic nanocomposites of mixed oxides of (Fe-Mn-Zn) in the polymer matrix of

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aniline-formaldehyde. The synthesized material is used to treat the contaminated water and explained with IR and SEM-EDX studies.

METHODOLOGY

To synthesize polymeric nanocomposites of mixed oxides in copolymer matrix we used the chemicals of high grade. The chemicals used are as 10.0 mL (100 mmol) of aniline (Sigma Aldrich), 10mL (0.10 mol) formaldehyde (Merck), 12.0 mL (120 mmol) of concentrated HCl (Thermo Fischer), $FeCl_3.6H_2O$ (Otto Kemi), $MnCl_2.4H_2O$ (Otto Kemi), $ZnCl_2$ (Merk) and NaOH (Otto Kemi) were used. For the adsorption studies we have synthesized the polymeric mixed oxides nanocomposites with aniline (0.10 mol), concentrated hydrochloric acid (0.12 mol), 10 mL formaldehyde (0.10 mol), 50 mL of doubled distilled water along with metal ions concentrations as (0.04 mol) Fe, (0.013 mol) Mn and (0.0079 mol) of Zn of respective salts. The details of the synthesis method are reported elsewhere[10]. 1000 ppm stock solution of Pb(II) ions was prepared by dissolving 1.598 g of Pb(II) nitrate in deionized water and the same was diluted to 1000 mL. Intermediate 10 ppm solution was prepared by diluting 1 mL of stock solution to 100 mL. Test solution of different concentrations were prepared. The synthesized material is characterized using FTIR, and SEM-EDX techniques. The material synthesized will be referred as sample in the whole text of this study.

RESULTS AND DISCUSSIONS

IR studies

Before and after adsorption of Pb(II) ions on the sample, the IR spectra showed marked variation in the IR peaks. This variation is in term of slight shifting and in intensity of peaks.

Table 1: Prominent IR peak positions before and after adsorption

S. N	Peak position before adsorption (cm^{-1})	Peak position after adsorption (cm^{-1})	Probable assignment of peak
1.	3332 (B), 3019 (B)	3358 (B), 3019 (B)	N-H stretching and C-H stretching vibrations[11,12,13]
2.	2918 (S)	2847 (S)	C-H aliphatic stretching of -CH2-group[12,13]
4.	2105 (W)	2098 (W)	Aromatic overtones or carbonyl C=O group of formaldehyde[14]
5.	1871 (W)	1886 (W)	Aromatic overtone or cominational band (1,4 substitution in ring)[14]
6.	1655 (Sh)	1655 (Sh)	C=N stretching vibrations of quinoid ring[11]
7.	1589 (ST)	1591 (ST)	C=C stretching vibrations of quinoid ring [11,12]
8.	1513 (ST)	1513 (ST)	C=C stretching vibrations of benzoid ring[11,12,13]
9.	1446 (ST)	1446 (W)	C-H Bending vibrations of -CH2-[13]
10.	1338 (ST)	1312 (M), 1268 (M)	C-N aromatic stretching ($-CH_2-N$ bonding)[12]
11.	1177 (ST)	1162 (ST), 1121 (W)	In-plane bending vibrations of aromatic C–H or Charge delocalization on polymer backbone (polyaniline salt)[12]

12.	1073	1073	Quinoid ring –NH^+– benzoid ring stretching vibrations[12]
13.	1013	1013	In-plane bending vibrations of aromatic C–H[12]
14	820 (ST)	816 (ST)	Para substitutions, 1,4 disubstitution in benzene ring (C-H out of plane bending)[11,12,13]

B: Broad peak, S: Small peak, M: Medium peak, Sh: Shoulder type, ST: Strong peak

The shift in some of the prominent IR peaks are reported in Table 1. The shift in the peaks positions is an indication of adsorption of Pb ions on the surface of the samples[15-.17]. The IR spectra of the sample before and after adsorption studies are shown in (Figure 1(a) & (b)) From the IR spectra we observe that the peaks in the region between 3332 cm^{-1} and 1886 cm^{-1}, which are due N-H stretching and C-H aliphatic stretching or due to 2,4 substitution of ring or due to comination bands shits to higher or lower wavenumber. This kind of shift is an indicative of adsorsorption of Pb(II) ions on these sites and sugest the formation of cordination type of bond. The peak in the region 1655-1315 cm^{-1} remain uneffected which are due to quinoid and benzenoid ring. The three peaks e.i. at 1446 cm^{-1}, 1338 cm^{-1} and 1177 cm^{-1} are highly affected by the asorption of Pb ions. There is intensity variation as well as slight shifting in position of these peaks, This can be attributed to interaction of Pb ions ions with the polymer bakbone via formation of hydrogen bonding and coordinate bonding. Similar behaviour of shifting of IR peaks to higher wavenumber has been reprted by many researchers.[17-23.] The presence of peak at 820 $cm^{-1}$is attributed to C-H aliphatic stretching which is an idicator of participation of formaldehyde in polymerization and formation of cross chain network. This peak shifts to low wave number on adsorption.

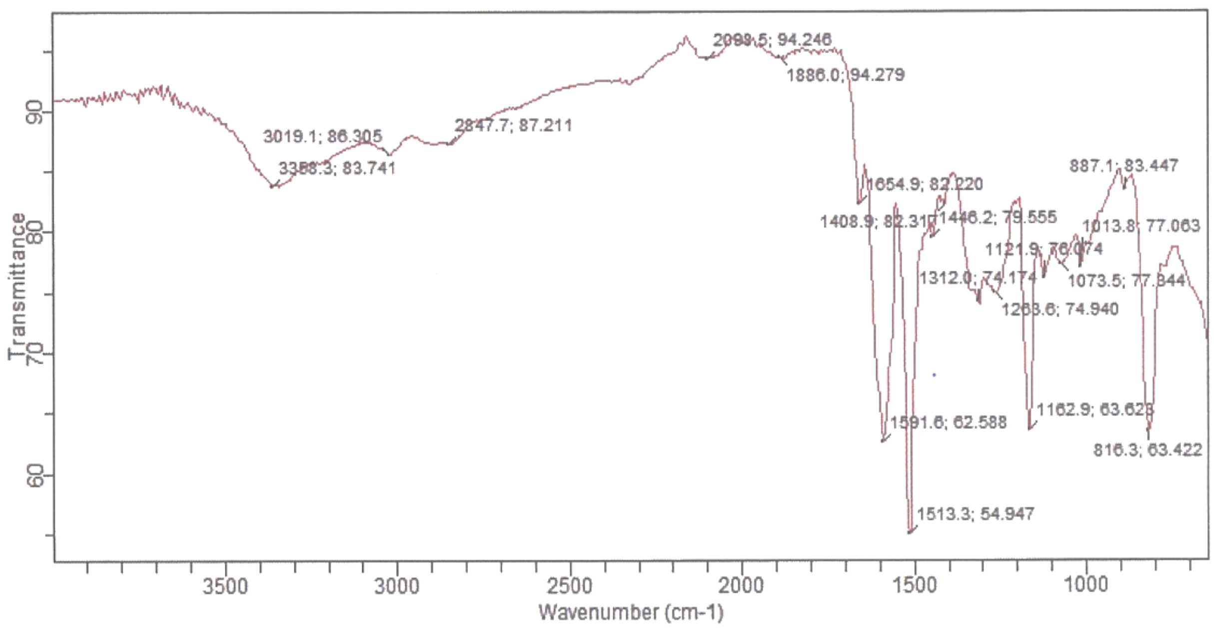

Figure 1(b): IR spectra of sample after adsorption of Pb (II) ions

SEM studies

SEM images of the sample before and after adsorption are given in figure 2(a &b). The average particle size calculated from this study is of the order of 82 nm. The morphology of the particles as viewed from the figures is nearly spherical. It is clear that the adsorbent has a considerable number of heterogeneous pores where there is a good possibility of heavy metal ions to be adsorbed. From the SEM micrographs of sample after adsorption of lead it is evident that the surface texture of sample is partially changed after the adsorption of lead ions. The surface of the metal-loaded adsorbent clearly shows that the surface of the adsorbent dosage was covered with

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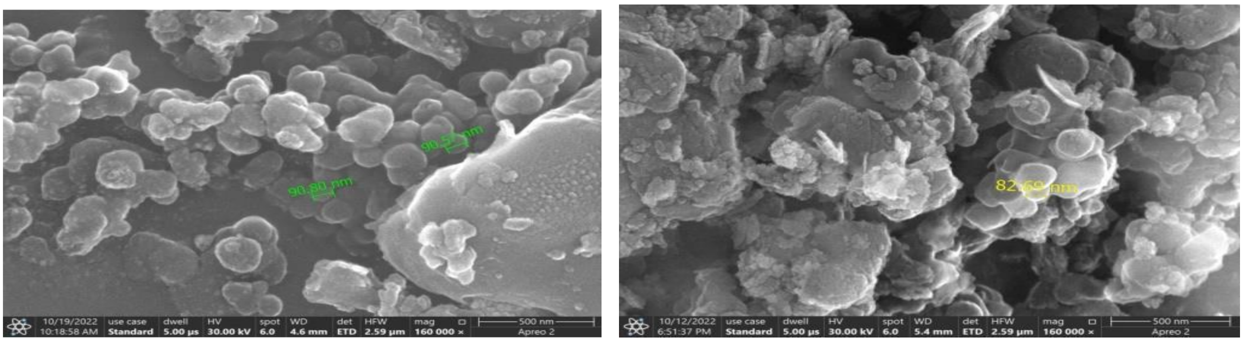
metal ions. The pores are getting filled with the metal ions after the adsorption of lead. This observation indicates that the lead ions is adsorbed to the functional groups present inside the pores of the adsorbent. Similar kind of behavior is reported in some studies of adsorption of Pb and Cd on activated carbon.[25]



(a) (b)

Figure 2(a & b): SEM Image of sample before and after Pb(II) adsorption

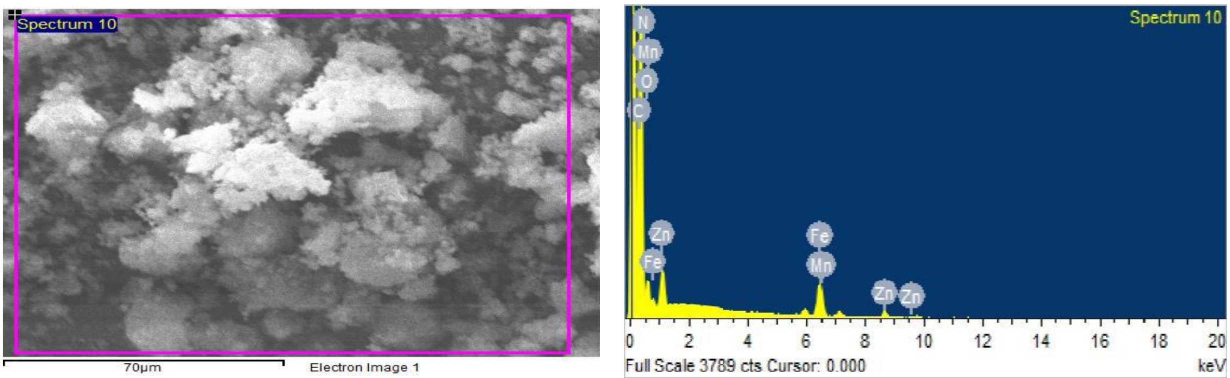


Figure 3 (a): SEM-EDX analysis of sample before adsorption of Pb(II) ions

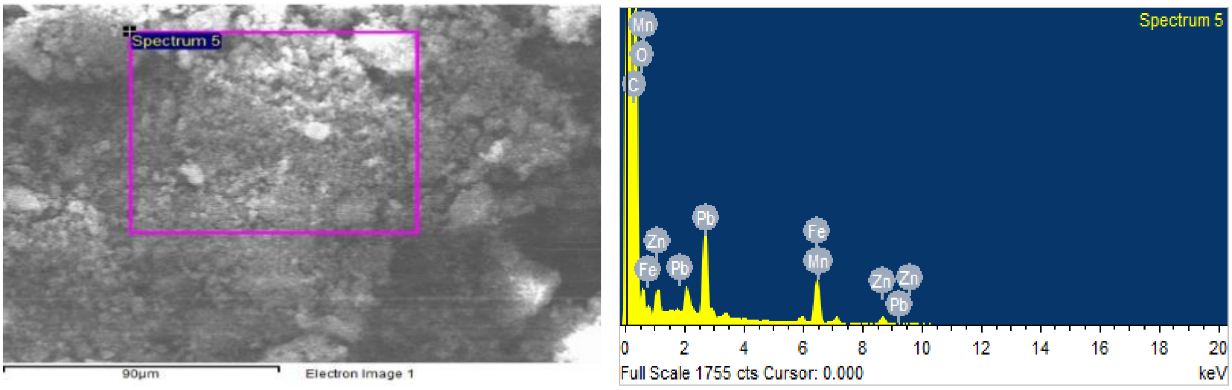


Figure 3 (b): SEM-EDX analysis of sample after adsorption of Pb(II) ions

SEM-EDX study (Figure 3 (a) & (b)) provided direct evidence for the adsorption of the Pb(II) ions as there is a peak of Pb after adsorption on the sample as compared to before adsorption.

CONCLUSIONS

From the IR studies it is established that the formation of mixed oxides of (Fe-Mn-Zn) in polymer matrix of aniline-formaldehyde. When this nanocomposite is used to adsorb Pb(II) ions from water then shifting and intensity variation in the peaks position take place which is an indication of some interaction of Pb ions with polymer backbone. The SEM studies confirm the particle size of nanocomposite to be of the order of 82 nm and particles are nearly spherical. The existence lead peak in SEX-EDX image is the direct indicative of Pb ions removal by adsorption by polymeric mixed oxides nanocomposites.

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