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# IR and SEM-EDX Studies on Polymer Matrix-Mixed Metal (Fe-Mn-Zn) Oxides Nanocomposite Adsorbent for Removing Zn(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 can cause the serious damage to our central nervous system and can harm kidney, liver, pancreas, and lungs, etc. So, the removal of such type of contamination from water is highly required. Various methods are adopted by researchers to overcome this difficulty. The adsorption technique is one of the most widely used to eliminate the heavy metals from the contaminated water. In the present study we have synthesized nanocomposites if mixed metal oxides of Fe-Mn-Zn in the copolymer matrix of aniline-formaldehyde. The material has been used to adsorb Zn (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 Zn ions with the copolymer backbone. Further, the SEM-EDX study confirmed the formation of nanosized particle of the materials and weight percentage increase of the Zn after adsorption.

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

## INTRODUCTION

The fresh water available for the use of mankind is less than 1% which is also getting contaminated due to day-to-day activities. Contamination of fresh water can occur due to presence of pathogenic agents[1], chemicals[2], heavy metals[3], pesticides[4], water disinfectants[5] and release of various other waste produced by industries. These contaminants are either added directly to water bodies or are indirectly added to the fresh water resources by leaching from the soil, rocks and atmospheric deposition. Heavy metals are elements with a relatively high density (> 5 g $cm^{-3}$) and varying degrees of toxicity even at low concentrations[6]. Lead, cadmium, mercury, arsenic, chromium, silver, copper, iron, zinc and platinum are examples of some heavy metals[7]. When these metals are not metabolized by the body and accumulate in soft tissues, they cause damage to the health of living organisms and even become hazardous[8]. Various remediation techniques have been employed to deal with these contaminants[9]. Adsorption technique has been preferred due to its simplicity, operability and diversity[10]. In the present study we have synthesized magnetic nanocomposites of mixed metal oxides in copolymer matrix of aniline formaldehyde and applied it for remediation of Zn(II) ions from aqueous matrices.

## METHODOLOGY

To synthesize polymeric nanocomposites of mixed oxides, 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, (0.0151 mol) Mn and (0.0039 mol) of Zn of respective salts. 1000 ppm stock solution of Zn(II) ion was prepared by using standard method as reported[18]. The details of synthesis process are reported elsewhere[11]. 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 Zn(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. 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 Zn ions on the surface of the samples[12-.14].

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. | 3287 (B) | 3350 (B) | N-H stretching |
| 2. | 2791 (B) | 2899 (B) | C-H aliphatic stretching |
| 3. | 2117 (S) | 2113 (S) | Aromatic ring bending/ C=N stretching in quinoid ring |
| 4. | 1994 (M) | 1990 (M) | Aromatic overtone or cominational band (1,4 substitution in ring) |
| 5. | 1655 (Sh) | 1655 (Sh) | C=N aromatic stretching |
| 6. | 1599 (ST) | 1587 (ST) | C=C aromatic ring stretching (deformed quinoid) |
| 7. | 1539 (Sh), 1509 (ST) | 1513 (ST) | C=C aromatic ring stretching (deformed benzenoid) |
| 8. | 1446 (ST) | 1446 (Sh), 1401 (W) | C-H bending of $-CH_2$ |
| 9. | 1338 (ST) | 1342 (W) | C-N aromatic stretching ($-CH_2$-N bonding ) |
| 10. | 1177 (ST) | 1163 (ST) | Charge delocalization on polymer backbone (polyaniline salt) |
| 11. | 827 (ST) | 816 (ST) | C-H out of plane bending |

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

The IR spectra of the sample before and after adsorption studies are shown in Figure 1(a) and Figure 1(b). From the IR spectra we observe that the peaks corresponding to N-H stretching and C-H aliphatic stretching shits to higher wavenumber. The shift is small in the region 2117-1509 $cm^{-1}$ toward higher wavenumber. In the region 1446-827 $cm^{-1}$ the variation is of both types increasing as well as decreasing. The intensity and postions of three peaks e.i. at 1446 $cm^{-1}$, 1342 $cm^{-1}$ and 1177 $cm^{-1}$ are highly affected by the asorption of Zn ions. This can be attributed to interaction of Zn 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.[15-21.] The presence of C-H aliphatic stretching is and idicator of participation of formaldehyde in polymerization and formation of cross chain network.

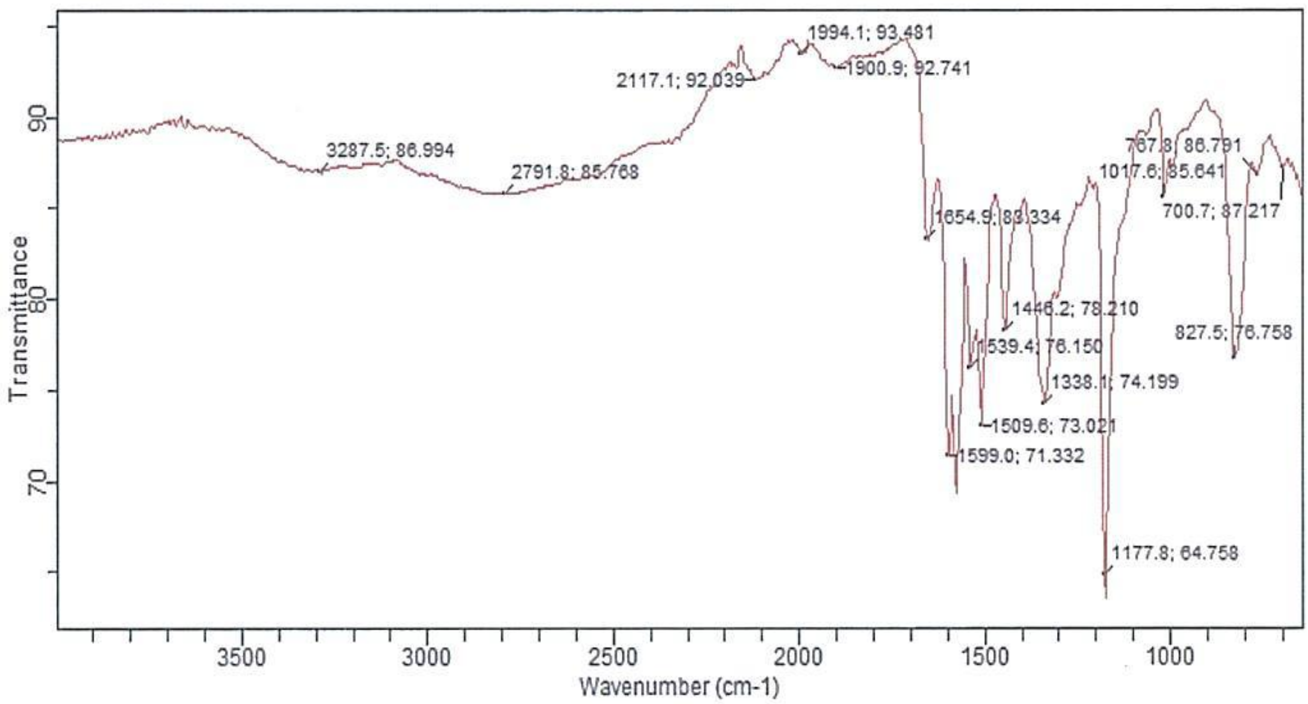


Figure 1(a): IR spectra of sample before adsorption of Zn(II) ions

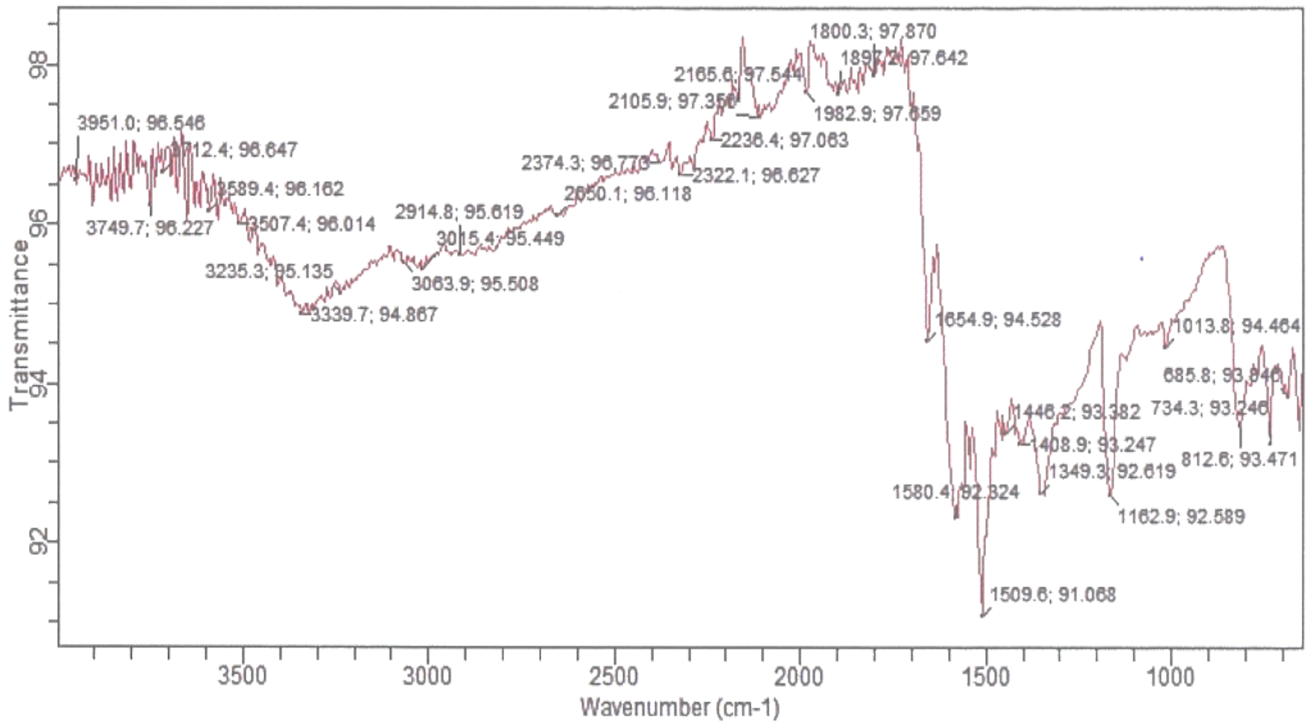


Figure 1(b): IR spectra of sample after adsorption of Zn(II) ions

## SEM and SEM-EDX studies

SEM images (Figure 2) after adsorption of the Zn(II) ions showed space filled particles of the adsorbent. This was due to adsorption of Zn(II) ions over the surface of the adsorbent. From this figure we can estimate the particle size of polymeric nanocomposites in the range of 95-100 nm and they are having almost spherical morphology.

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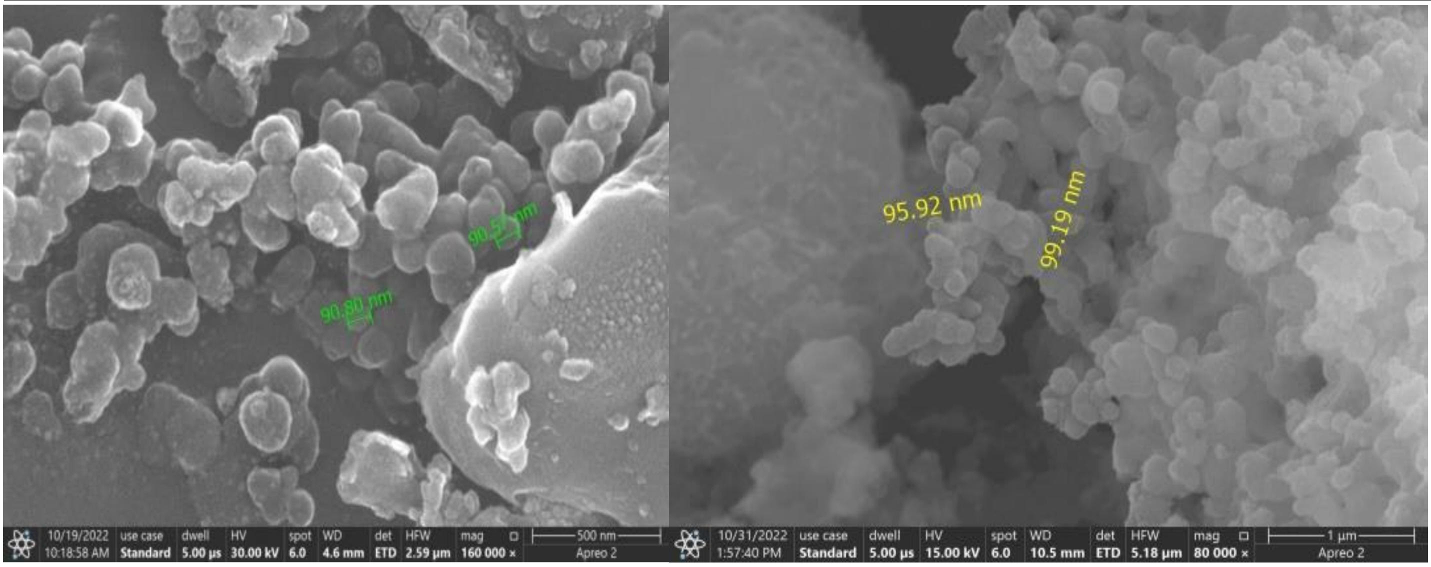


(a) (b)

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

SEM-EDX (Figure 3) provided direct evidence for the adsorption of the Zn(II) ions as there was increase in weight percentage of Zinc over the adsorbent surface from 0.67% to 2.03% along with rise in atomic percentage from 0.13% to 0.42% before and after the adsorption of metal ions.

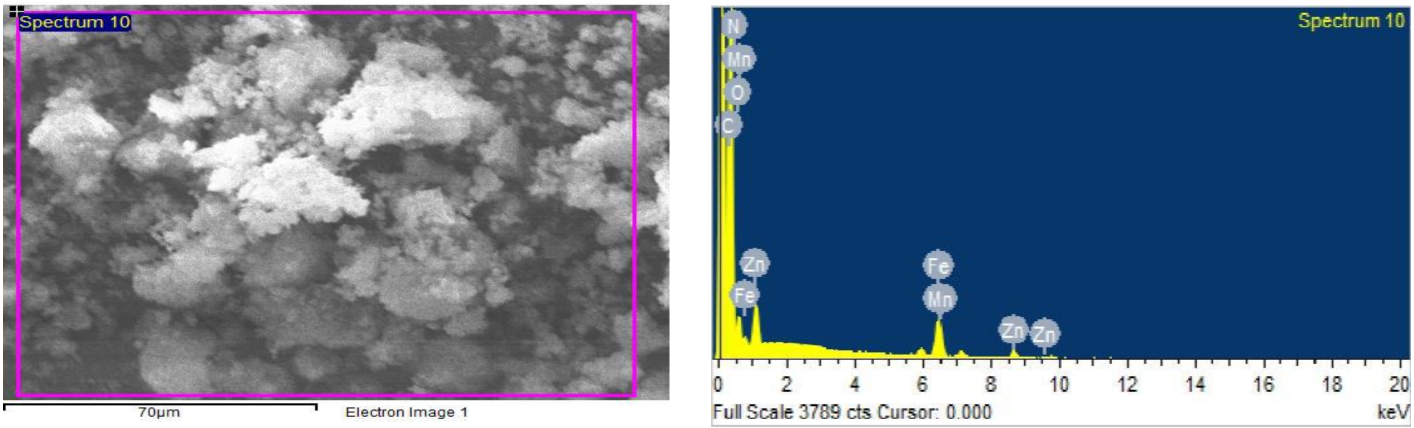


Figure 3 (a): SEM-EDX analysis of sample before adsorption of Zn(II) ions

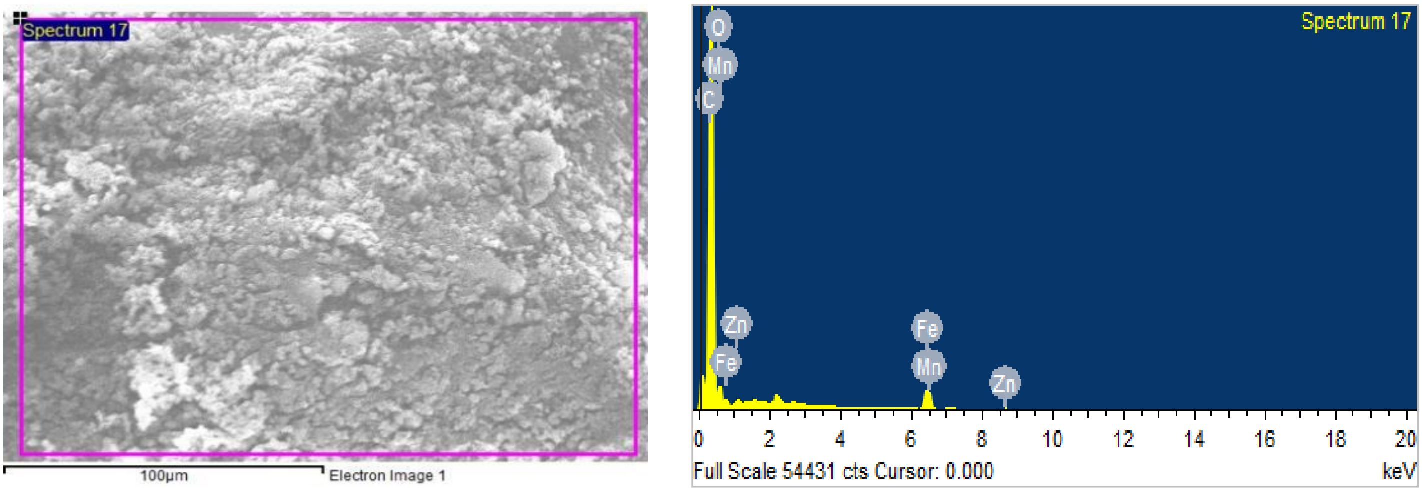


Figure 3 (b): SEM-EDX analysis of sample after adsorption of Zn(II) ions

## CONCLUSIONS

From the IR studies it is established that the formation of mixed oxides nanocomposites in the copolymer matrix of aniline-formaldehyde. When this nanocomposite is used to adsorb Zn(II) ions from water then shifting and intensity variation in the peaks position is an indication of some interaction of Zn ions with polymer backbone. The SEM studies confirm the particle size of nanocomposite to be of the order of 100 nm and particles are nearly spherical. The increase in the weight percentage of Zn after adsorption as observed form SEM-EDX studies is direct indicative of Zn ions removal by adsorption by the use of polymeric mixed oxides nanocomposites.

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