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Research Article

NOVEL THERMAL DECOMPOSITION APPROACH FOR THE FABRICATION OF Mn (II) AND Zr (II) DOPED NANOPARTICLES AND STUDIES THEIR PHYSICAL PROPERTIES

Jyoti Chaudhary, Monika Sharma*

Department of polymer science, Mohanlal Sukhadia University, Udaipur, India
**Corresponding author: sharma.monika.b@gmail.com*

ABSTRACT

The aim of this research was to fabricate the manganese (II) and zirconium (II) nanoparticles by novel thermal decomposition approach. The physical properties such as viscosity and density of sample have been reviewed. The nanoparticles samples were characterized by FTIR (Fourier transform infra red spectroscopy) and SEM (Scanning electron microscopy) technique. Functional group present in the sample was analyzed by FTIR while the surface morphology of nanoparticles was confirmed by scan electron microscopy. The presence of methylene bridge observed at 2929 cm^{-1} and 2940 cm^{-1} which conforms the bonding between formaldehyde and p- bromo aniline while peaks at 512 cm^{-1} and 469 cm^{-1} conforms the zirconium and manganese metals bonding with polymer. Viscosity and density of nanoparticles were observed in our research laboratory by using ubbelohde viscometer and pycnometer respectively.

Keywords: Thermal decomposition, Physical property, Nanoparticles, SEM, FTIR

1. INTRODUCTION

Nanoparticles have many applications in different fields like environmental technology, biological application, chemical manufacturing etc. These metal nanoparticles often exhibit different activity from the corresponding bulk materials due to their different size and shape [1, 2]. Therefore, a variety of fabrication methods such as sol-gel processing methods, thermal decomposition method, gas-phase, microwave, hydrothermal, colloidal-templating has applied for the preparation of metal nanoparticles [3]. The transition metal nanoparticles are prepared by the thermal decomposition method due to it is environmentally friendliness, low cost and a high versatility. The structure of nanoparticles is mainly dependent upon the calcinations time, heating rate and calcinations temperature [4]. A simplest structure of Formaldehyde is often used for the electro-catalytic activity of electrodes [5]. The transition metal zirconium is a strong resistance to corrosion used in alloying agent that are employed as corrosive agents like in vacuum tube getters, surgical appliances, explosive primers, and filaments. It is also used for making zirconium inorganic and organic compounds. Many examples of the synthesis of zirconium complexes are employed for catalytic applications [6, 7]. A good deal of manganese polymer composite developed is extensively used due to its optical and electrical properties [8, 9]. Manganese is important transition metal used in many applications such as molecular adsorption, catalysis, high-density magnetic storage media, electrodes, electronics, ion exchangers and sensors [10-12].

In this research article, we have fabricated and characterize the Mn (II) and Zr (II) nanoparticles of p-bromo aniline formaldehyde resin by using novel thermal decomposition method and further studied their physical properties.

2. EXPERIMENTAL DETAILS

In the thermal decomposition method, metal salt are mixed with polymeric solution and then subjected to thermal decomposition at a high temperature. In the present study metal salt of manganese sulphate and zirconium sulphate have been used along with p- bromo aniline formaldehyde resin as polymeric solution.

2.1. Material and Methods

Analytical grade chemicals were used in this research like p- bromo aniline, formaldehyde are obtained from central drug house pvt. Ltd. Sulphuric acid (H_2SO_4), sodium hydroxide (NaOH), metal salts like manganese sulphate, zirconium sulphate was obtained from fisher scientific. Distilled water used for the preparation and purification of nanoparticles. FTIR Perkin Elmer spectrometer was used for the detection of IR characteristics bands and X- ray pattern was analyzed by using X- ray diffractometer with Cu-Kα radiation source. The ubbelohde viscometer and pycnometer were used in laboratory for the determination of viscosity and density, respectively.

2.2. Sample fabrication Method

Two nanoparticles samples were prepared by changing the metal salt mixed with p- bromo aniline formaldehyde solution. The reaction scheme for the synthesis of Mn (II) and Zr (II) nanoparticles are described in below Fig. 1 and following thermal decomposition method has been adopted.

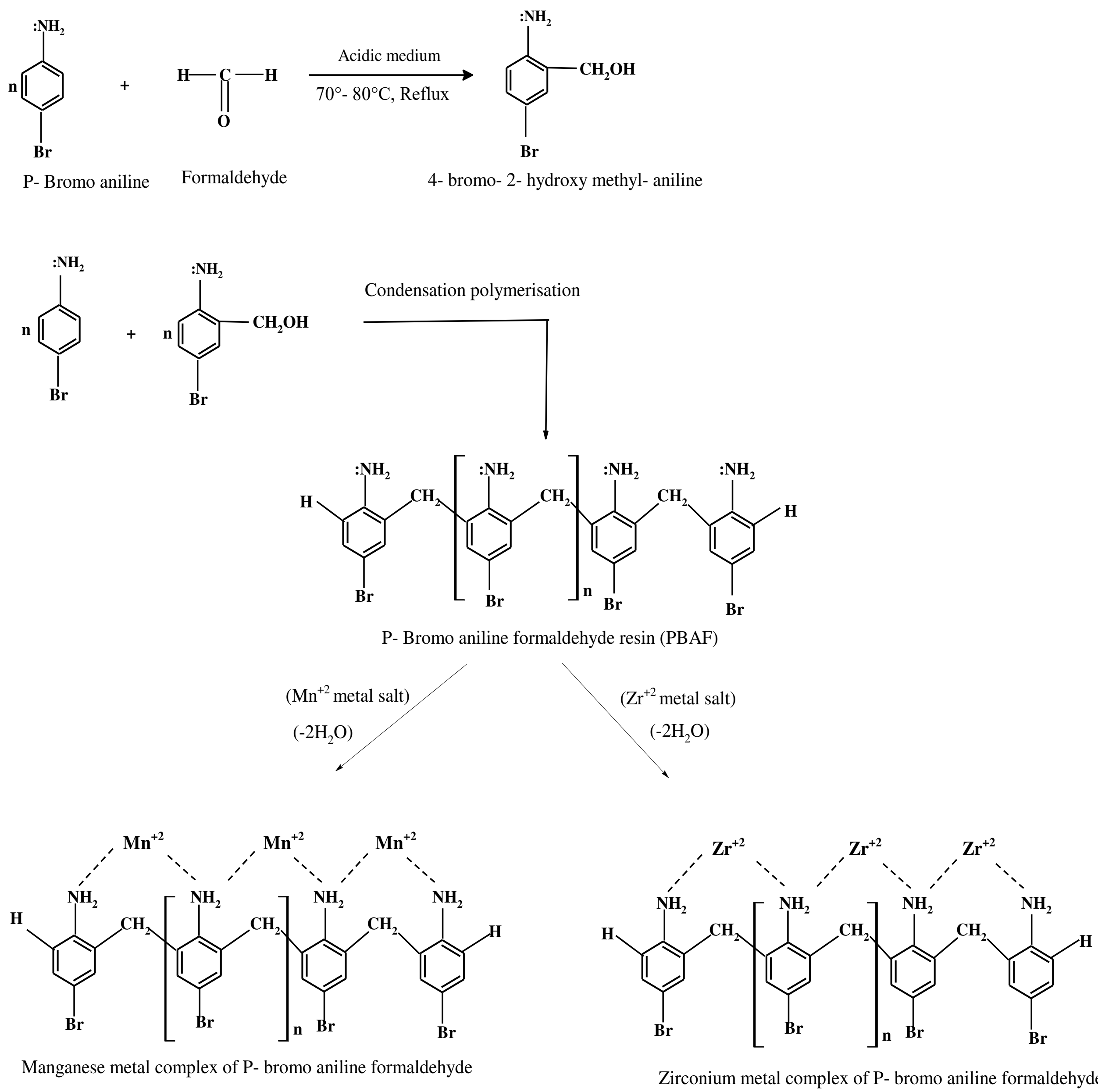

Fig. 1: Reaction scheme

In this method 10gm of p- bromo aniline taken in 500 ml beaker then 20 ml of formaldehyde solution was mixed with continuous stirring for 15 minute. After that few drops of sulphuric acid was added in the solution followed by mixing of sodium hydroxide to obtain creamy color precipitate of polymeric solution. Later on 1N metallic solution of manganese sulphate and zirconium sulphate was prepared in beaker and added into polymeric solution separately. This solution was heated with magnetic stirrer at 60-70ºC for 1 hour.

The obtained precipitation was washed with distilled water and dry in desiccators overnight. Now the powder of metal complexes of p- bromo aniline formaldehyde resin was calcined in muffle furnace at 700°C for 2 hours. The grayish black color of nanoparticles was obtained after thermal decomposition of polymeric metal complexes.

3. RESULTS AND DISCUSSION

3.1. FTIR (Fourier transform infra red) Analysis

The infra red spectra of manganese and zirconium metal complexes of p- bromo aniline formaldehyde resin are shown in figure 2 and 3 respectively and characteristics bands are shown in table 1.

Table 1: Characteristic IR bands of zirconium and manganese metal complex with p- bromo aniline formaldehyde (PBAF) resin

Type of vibration	Bands of zirconium metal complex (cm^{-1})	Bands of manganese metal complex (cm^{-1})
N-H stretching of aniline	3441	3417
-C-H stretching of aromatic CH=CH	3068	3035
$-CH_2-$ stretching of methylene group	2929	2940
N-H bending of aniline	1657	1665
C-C stretching of CH=CH	1587	1578
C-N stretching of amine moiety	1223, 1301	1258, 1310
-Br group in aromatic ring	608	694
Ortho substituted aromatic ring	764	773
Para substituted aromatic ring	807	807
Zirconium metal bond with N (Zr-N)	512	-
Manganese metal bond with N (Mn-N)	-	469

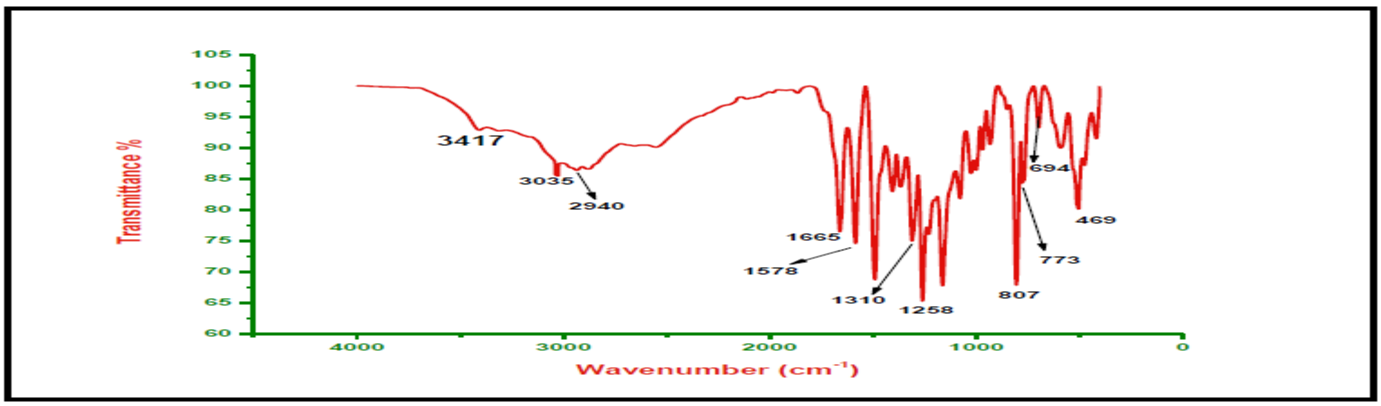

Fig. 2: FTIR spectra of manganese metal complex with p- bromo aniline formaldehyde resin

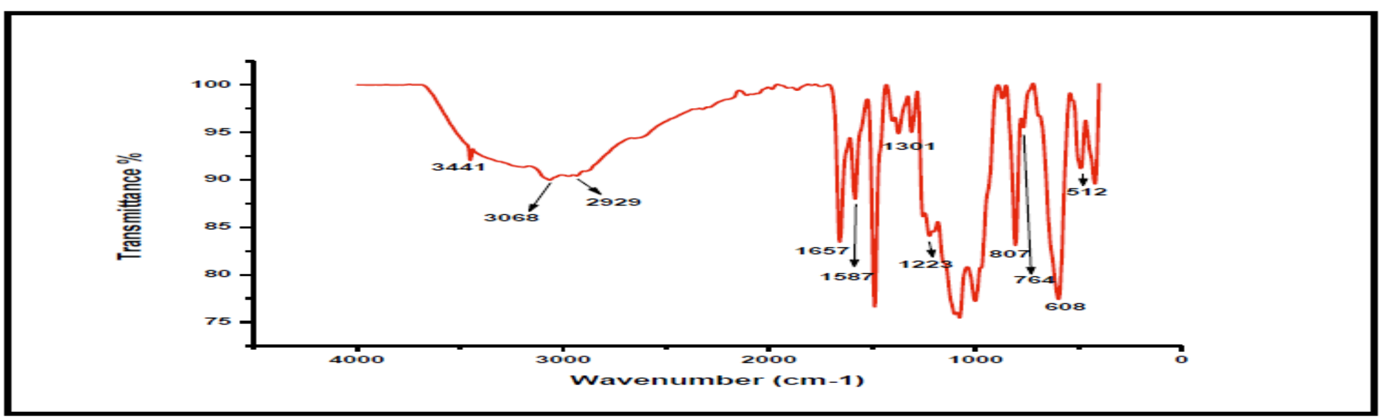

Fig. 3: FTIR spectra of zirconium metal complex with p- bromo aniline formaldehyde resin

3.2. SEM (Scanning electron microscopy) analysis

The surface morphology of zirconium and manganese nanoparticles of p- bromo aniline formaldehyde resin is illustrated by below figures (4, 5) at a different magnification. We analyzed by these images that surface of nanoparticles are homogeneous in nature. It shows spherical shape dot in the surface of nanoparticles which concluded that zirconium and manganese metal incorporated into p- bromo aniline formaldehyde resin. The SEM micro graphs of zirconium nanoparticles are brighter than manganese nanoparticles of p- bromo aniline formaldehyde resin due to high molecular weight.

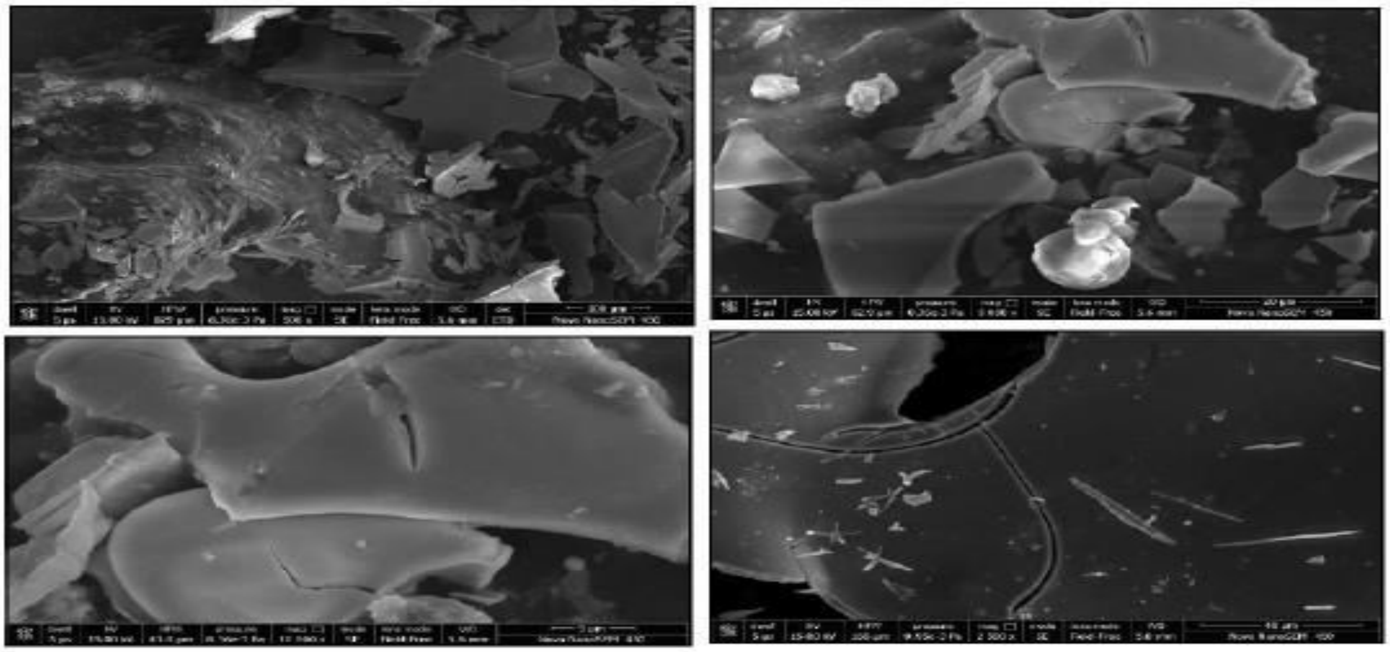

Fig. 4: SEM Images of zirconium doped in p- bromo aniline formaldehyde resin at a different magnification

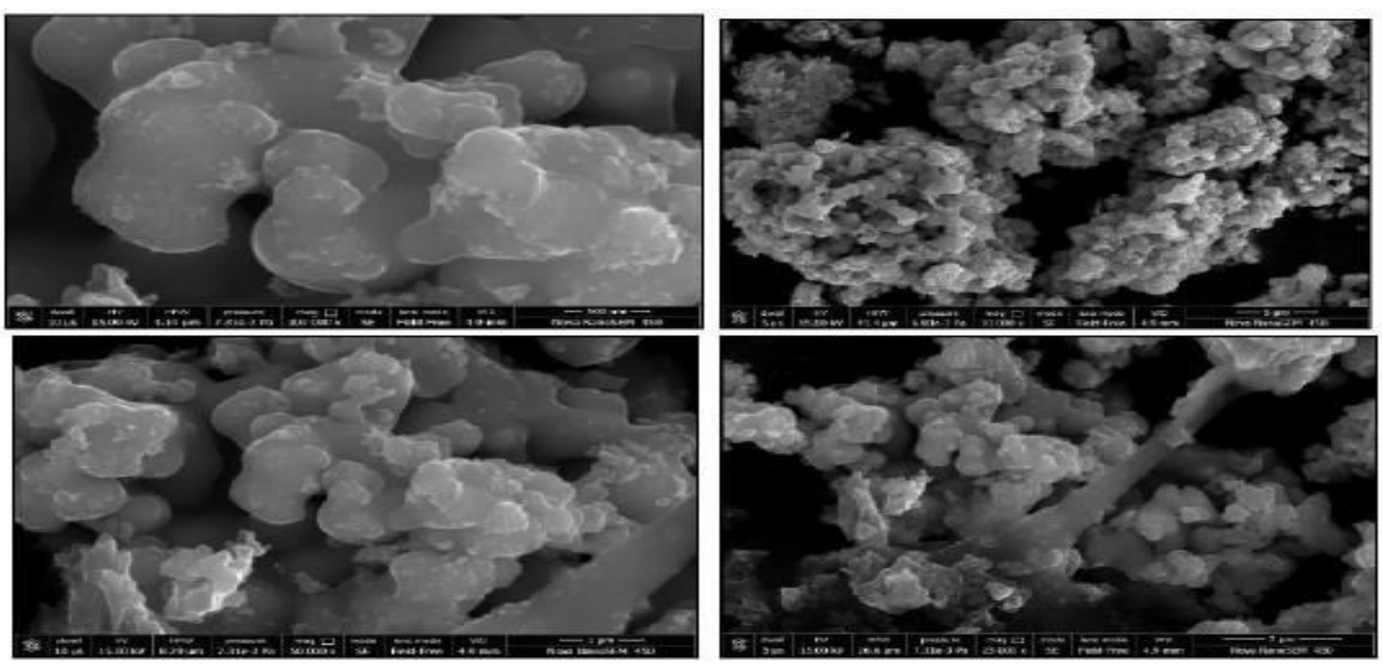

Fig. 5: SEM Images of manganese doped in p- bromo aniline formaldehyde resin at a different magnification

3.3. Viscosity measurement

Viscosity is a physical property of fluids which describes as "fluid thickness". The concentration of solute, temperature, pressure, molecular weight and inter molecular force affect the viscosity. As the molecular weight of nanoparticles is increases, viscosity is also increases. We determined the viscosity by using ubbelohde viscometer in our laboratory and calculate the relative viscosity (η_{rel}), specific viscosity (η_{sp}) and reduced viscosity ($\eta_{red.}$). We plot the graph between concentration of solution and reduced viscosity and observed the value of intrinsic viscosity by intercepting of this plot at zero concentration. We find out by the viscosity measurement that zirconium nanoparticles have more viscosity than manganese nanoparticles due to large molecular weighty. The viscosity of Mn (II) and Zr (II) nanoparticles of p- bromo aniline formaldehyde resin are demonstrated in table 2 and fig. 6.

Table 2: Viscosity of Mn (II) and Zr (II) nanoparticles of p- bromo aniline formaldehyde resin in DMF at 30^{0}C

Conc. (g/dl)	$\eta_{rel.}$	η_{sp}	η_{red} or (η_{sp}/C)	[η] of Mn (II) (dl/g)	Conc. (g/dl)	$\eta_{rel.}$	η_{sp}	η_{red} or (η_{sp}/C)	[η] of Zr (II) (dl/g)
0.1	1.050	0.050	0.500		0.1	1.072	0.072	0.720	
0.2	1.106	0.106	0.530		0.2	1.149	0.149	0.745	
0.3	1.162	0.162	0.540		0.3	1.224	0.224	0.747	
0.4	1.218	0.218	0.545		0.4	1.296	0.296	0.740	
0.5	1.274	0.274	0.548	0.510	0.5	1.373	0.373	0.747	0.725
0.6	1.330	0.330	0.550		0.6	1.458	0.458	0.763	
0.7	1.389	0.389	0.556		0.7	1.536	0.536	0.766	
0.8	1.448	0.448	0.560		0.8	1.613	0.613	0.767	
0.9	1.506	0.506	0.562		0.9	1.693	0.693	0.770	
1.0	1.578	0.578	0.578		1.0	1.779	0.779	0.779	

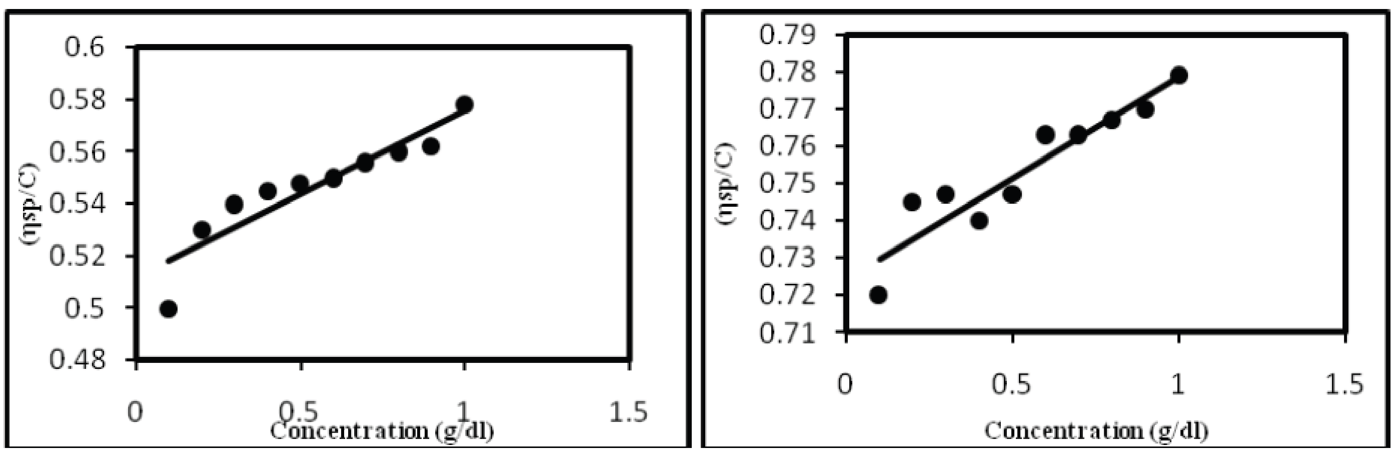

Fig. 6: Specific viscosity v/s concentration for manganese and zirconium nanoparticles of p- bromo aniline formaldehyde resin

3.4. Density measurement:

Density of polymer metal nanoparticles is measured by pycnometer (specific gravity bottle) in our laboratory by using water displacement method. Density varies with change in temperature and pressure because by increasing the temperature of substance, volume of substance increases therefore density decreases. Here we calculate the specific density of manganese and zirconium nanoparticles of p- bromo aniline formaldehyde resin by given mathematical formula. We found that the specific density of Mn (II) and Zr (II) nanoparticles of p- bromo aniline formaldehyde resin are 2.740 gm/cm^3 and 5.840 gm/cm^3 respectively.

$$SG = \frac{M_2 - M_1}{(M_4 - M_1) - (M_3 - M_2)}$$

SG = Specific gravity
M_1 = Weight of empty density bottle.
M_2 = Weight of density bottle and polymer sample.
M_3 = Weight of density bottle, polymer sample and water.
M_4 = Weight of density bottle packed with water.

4. CONCLUSION

In this scenario we have surveyed the fabrication, characterization and physical properties like viscosity and density studies of polymeric metal nanoparticles synthesized by novel thermal decomposition method. The synthesized nanoparticles were characterized by using FTIR and SEM analysis techniques to prove their formation. FTIR technique gives the detailed information of functional group present in the sample and bonding of metal with polymer while surface morphology of nanoparticles is examined by SEM analysis. By measuring viscosity and density of nanoparticles, we have found out that zirconium nanoparticles have more viscosity and density than manganese nanoparticles due to their high molecular weight.

5. ACKNOWLEDGMENT

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