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Spectroscopic Investigation of Manganese (II) Bakelite Composite

Reetuka Raj[1], B.Tiwari[2]

[1] Department of Chemistry, Krishna Engineering College, Ghaziabad, (UPTU, Lucknow), Uttar Pradesh, India
[2] D.S. Institute of Technology and Management, Ghaziabad, (UPTU, Lucknow), Uttar Pradesh, India

ABSTRACT: Manganese (II) Bakelite composite were prepared by incorporating polymer reactants of Bakelite in prepared homogeneous solution of $MnCl_2.4H_2O$ salt in distilled water. It was characterised by FTIR spectroscopy and [1H]NMR spectroscopy. The results reported the feasibility of composite formation.

Keywords: FTIR, [1H]NMR, Bakelite, Metal matrix composite, Manganese salt.

1. INTRODUCTION

The amazing properties of metal matrix composite materials has led to their extensive use in variety of engineering industries. These material have replaced metallic materials in the market due to their cost effectiveness and light weight [1]. These materials play major role in automotive, aerospace application as they fosters superior mechanical, thermal, electrical, cryogenic and dynamic properties in the material. The developmental efforts for finding newer composites for existing and novel applications assume top priority A tremendous efforts in raising new blends and composites is seen recently, with which has emerged a galaxy of book reviews and conferences proceedings covering different aspects of the preparation, phase behavior and applications of different types of blends and composites[2].

Bakelite has been the hardest formulation developed in 1905.Its tensile strength, insulation fire retardancy has always attracted it in industrial and foundry application. Bakelite composite and blends with various metals such as B, Fe, Bronze, Ag, and Cu etc. have been reported to improve its properties and are used in industrial applications [3, 4, 5, 6, 7]. Manganese salts have been reported to influence electrical capacitance, Ac conductivity and magnetic nature of polymers [8, 9] .XRD of Mn Bakelite has reported to influence crystalline nature of composite to amorphous state [10]. Present work attempts to study novel Mn (II) Bakelite composite structural composition by spectroscopic analysis which can benefit further in processing this composite for industrial application, which require thermal resistance or electrical conductivity in an insulator.

2. EXPERIMENTAL PROCEDURE
2.1 MATERIALS

Supply of Phenol was done by Qualigens Fine Chemicals (India).Formaldehyde and Hydrochloric Acid was supplied by Fisher Scientific, Qualigens (India). Glacial acetic Acid was bought from Central Drug House (P) Ltd (India). Manganese (II) Chloride tetrahydrate ≥98% (MnCl2.4H2O) was purchased from Sigma-Aldrich ACS reagent (India). Chemically pure grade distilled water was used to prepare composites. Metal solutions were prepared by dissolving appropriate amount of its chloride salt in distilled water.

2.2 SAMPLE PREPARATION

2gm of MnCl2.4H2O Manganese dichloride tetra hydrate was weighed separately and was mixed in 5ml of Distilled water in a beaker. 2gm of phenol and 4 ml of formaldehyde solution was added in it. Then 5ml of glacial acetic acid was added to it The preparation of the Mn (II) Bakelite composite was according to a method reported in the literature [10].It includes condensation polymerization to form homogenous mixture of Bakelite and manganese dichloride tetrahydrate which on solidification form reddish hard shiny composite. The reaction is exothermic in nature and thus can be explosive. The composite was kept in desiccators and then grounded into fine powder for undergoing various spectral analysis.

2.3 CHARACTERISATION

Spectroscopic techniques are important procedures to characterize various composites. These techniques are powerful tools for characterization of composites quantitatively and qualitatively. FTIR spectra of Mn (II) Bakelite powdered samples were obtained at 400-4000cm[-1] on a Perkin–Elmer (Model 843) Infra-red Spectrophotometer, using KBr pellet.1HNMR was conducted on a 300 MHz Brüker DPX spectrometer using methyl-d sulfoxide as solvent. The proton chemical shifts are recorded in ppm and calibrated on the solvents as internal standard

3. RESULT AND DISCUSSION

3.1 [1H]NMR

The standard values for proton [1H]NMR for Bakelite resin lies in the range 7-6.4 ppm, 3.7- 3.33 ppm, 2.5

ppm -1.24 ppm [11] for various functional groups. The 1H-NMR spectrum of the composite is shown in the Fig. 1. The wide resonance line (multiplets) at the region of 6.646, 6.785, 6.885, 6.967 ppm refer to the aromatic protons (Ar-H) and are comparable to commercial novalac resin but has decrease intensity. The resonances at the region of 3.395 ppm is assigned to the methylene groups – (----CH2---)--. There is decrease in chemical shift in this region from 3.77 ppm to 3.395 ppm as expected. It is due to presence of manganese (II) in the interstitial spaces of the composite. The free electrons of d orbital of manganese increase shielding and lead to resonance at higher field or lower chemical shift value. The peaks at the region of 2.49 ppm are assigned to the DMSO solvent. The peaks at 1.988-1.893ppm appear as allylic signal due to presence of double bonds either due to alkene or aromatic functional group.
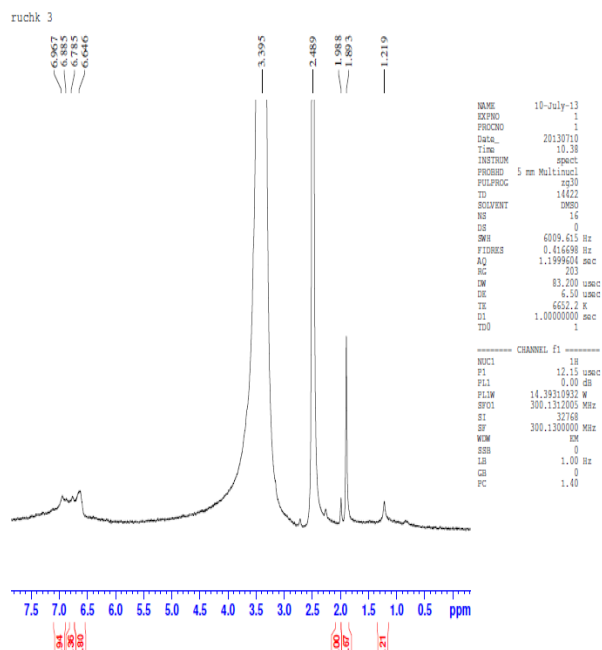

Fig.1 [1H]NMR of Manganese (II) Bakelite composite

3.2 FTIR

Infrared spectra of Bakelite has been reported in band region of 3000-3620 cm[-1] ;1315-1400 cm[-1]; 1500 cm[-1] ; 820 cm[-1]; 1200 cm[-1] for various functional groups(OH stretch, methylol hydroxyl phenolic ring, phenolic ring substituted at the ortho- parapositions , aromatic ethylene bonds (C=C) of phenolic ring, stretching vibration of phenol-O group)[12]

FTIR spectra of composite is shown in Fig. 2 which lies in band region 3501.85 - 3363.13 cm[-1]which can be attributed to OH stretching, 1633.70 cm[-1] due to C=C stretch, 1506.57-1478.70 cm[-1] phenolic ring substituted at the ortho-parapositions, 1435.8-1328.5 cm[-1]due to presence of hydroxyl groups (methylol hydroxyls) in the molecules, 1254.98 cm[-1] stretching

vibration of phenol-O group, 1098.84 cm[-1] due to CH_2OH stretch.

FTIR of Mn (II) Bakelite spectra indicate comparable result to standard Bakelite spectra but there was transition of band range to higher frequency which has occurred due to metal incorporation in polymer backbone.
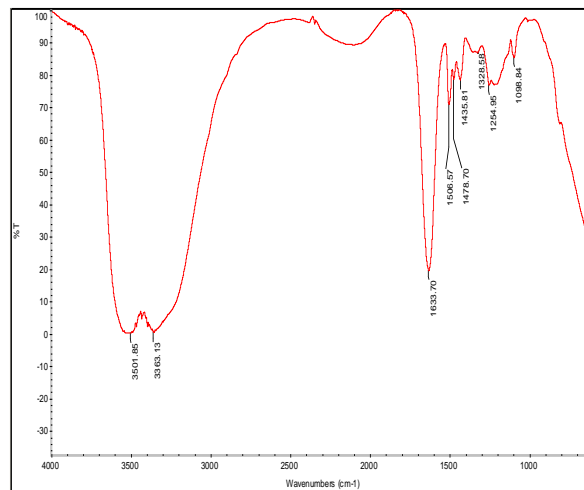

Fig.2 FTIR of Mn (II) Bakelite composites

4. CONCLUSION

The incorporation of metal in polymer to form metal matrix composite has been reported by [1H]NMR and FTIR studies. The FTIR of composite indicated transition of band region to higher frequency due to influence of metal and [1H]NMR revealed influence of metal d electrons leading to lower chemical shift value in the spectra.

5. ACKNOWLEDGEMENT

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