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Synthesis, characterization and thermal study of poly (methyl methacrylate)-Co_3O_4 nanocomposite film

A. Kodge[1]
S. Kalyane[2]
A. Lagashetty[3,*]

[1]*Department of Chemistry, Singhania University, Rajastan, India.*
[2]*Department of Physics, Rural Engineering College, Bhalki, Bidar, Karnataka, India.*
[3]*Department of Chemistry, Appa Institute of Engineering & Technology, Gulbarga, Karnataka, India.*

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* Corresponding author:
Arunkumar Lagashetty
Department of Chemistry, Appa Institute of Engineering & Technology, Gulbarga, Karnataka, India.
Tel +91 8472-242688
Fax +91 8472-229835
Email *arun_lagashetty@yahoo.com*

ABSTRACT

Nanosized metal oxides dispersed polymer composites constitute a fascinating class of polymer composite materials. Synthesis of such composite materials through solvent casting enhances the polymer synthetic technology. Solvent casting method was used to prepare Cobalt oxide (Co_3O_4) dispersed Poly (methyl methacrylate) (PMMA) nano composite. X-ray diffraction tool is used to know the structural behavior composite Development of the crystallinity in the composite film is observed. Scanning Electron Micrograph (SEM) tool is used for morpholoical study of the sample. The fine dispersion of the cobalt oxide particles is observed in the composite image. Bonding nature in the pure PMMA and composite materials are studied by FT-IR tool. This study shows the shift in some peaks and disappearance of some peaks reveals the formation of composite between PMMA and cobalt oxide. Thermal behavior of the composite is also well studied.

Keywords: *Polymer composite; Co_3O_4; PMMA; Solvent casting; Crystallinity.*

INTRODUCTION

Particles with diameter less than 100 nm are under nanomaterials and they exhibit special properties. Polymer nanocomposite materials are composed of two or more phases, one of which has a grain size of less than 100 nm. The grain size limit of 100 nm is general, but most phenomena related to grain size are restricted to particles with sizes below 10 nm. Many research groups have contributed to synthesis [1-3] and properties [4-6] of nanomaterials and nanocomposites. In the past few years nanomaterials have become one of the most extensively studied materials all over the world as they have shown to posses several applications such as effective quantum electronic devices, magnetic recording materials, sensors etc [7-9].

Moreover, nanocomposite materials composed of oxides and non-conducting polymers have brought out more fields of applications. Thus nanocomposites formed by combining nonconducting polymers and inorganic nanoparticles viz. metal oxides; possess all the good properties of both the constituents and an enhanced utility thereof. The properties of nanocomposite of such a kind are strongly dependent on concentration of polymer. Poly (methyl methacralyte), one of the nonconducting polymers, has received lot of attention in the preparation of nanocomposites due to its high stability [9]. Nanocomposite materials based on nanosized magnetic metal oxide materials have been of great interest to researchers due to their possible applications [10]. These composites are often prepared by dispersing magnetic materials in a non-magnetic matrix. In the present investigation, nonconducting polymer viz. poly (methyl methacralyte), has been chosen to act as the nonmagnetic matrix and cobalt oxide is magnetic material.

In this paper we report the preparation of cobalt oxide dispersed PMMA composite film by solvent casting method. The structure of prepared nanocomposite film was characterized by XRD, morphology by SEM and bonding nature by FT-IR tool. Thermal behavior of the PMMA-Co_3O_4 well studied.

EXPERIMENTAL

Materials and methods

Poly(methyl methacrylate) was obtained commercially. Acetone was used as a solvent for preparation of cobalt oxide dispersed PMMA composite film. Solvent casting method was adopted for the synthesis of composite film.

Synthesis of Pol(methyl methacrylate)-Cobalt oxide (PMMA-Co_3O_4) nanocomposite film

In our earlier work, we reported the synthesis of cobalt oxide nanoparticles through thermal decomposition of cobalt oxalate precursor by microwave route and also cobalt oxide dispersed polyvinyl alcohol composite by solvent casting method [11,12]. Cobalt oxide dispersed PMMA (PMMA-Co_3O_4) nanocomposite film was prepared by the same solvent casting method. 2g of PMMA was stirred in 50ml of acetone and was kept for 10 hours in a closed container and the solution was stirred under magnetic stirrer for 10 minutes. 0.1g of as prepared cobalt oxide nanoparticles were mixed in to the PMMA gel thoroughly in a rotary evaporator which was constantly maintained at 80-90 ^{0}C in a water bath. The gel solution containing gamma cobalt oxide was transferred into a petridish containing hot water as an immiscible solvent and it was kept 8hours for evaporation of acetone. The fine Co_3O_4 dispersed PMMA film was obtained and were characterized by various characterization techniques.

Characterization

The powder X-ray diffraction pattern of cobalt oxide and PMMA-Co_3O_4 nanocomposite film was obtained from GEOL JDX-8P X-ray diffractometer using CoKα radiation. The morphology of the nanocomposite film was obtained from Leica Cambridge-440 scanning electron microscope. The infrared spectrum of PMMA and PMMA-Co_3O_4 nanocomposite film was recorded on a Perkin-Elmer FTIR spectrometer [model 100] in the range 3000-300cm^{-1}. The thermal traces were obtained from Perkin-Elmer instrument.

RESULTS AND DISCUSSION

X-ray diffraction (XRD)

XRD pattern of pure PMMA sample is shown in Figure 1a. The pattern shows the absence of Braggs reflections indicating the amorphous nature of PMMA. Figure 1b shows the indexed XRD pattern of cobalt oxide dispersed PMMA nanocomposite film. The pattern shows the presence of some cobalt oxide reflections which are indexed in the pattern by the reference of cobalt oxide JCPDS file 42-1467. The cobalt oxide peaks in the composite pattern confirm the formation of cobalt oxide dispersed PMMA composite and development of crystallinity in the polymer matrix. Development of crystallinity in PMMA matrix is due to the complexation of cobalt oxide nanomaterials with PMMA matrix. A comparison of the X-ray diffraction pattern of pure PMMA with its cobalt oxide dispersed PMMA nanocomposites

shows that the diffraction peaks is due to the development of crystallinity in the amorphous PMMA. The broad halos in the region 5 to 20^0 correspond to the organic phase present in the PMMA.

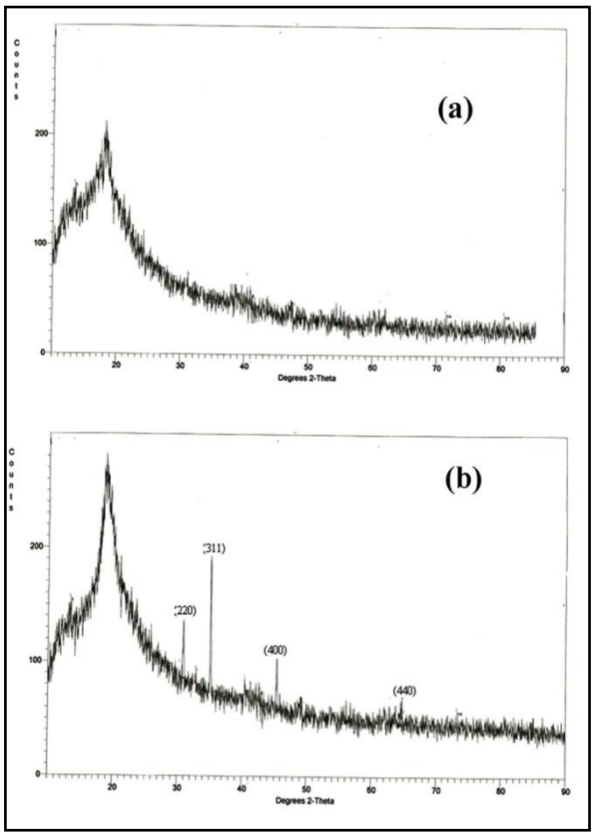

Fig. 1. (a): XRD pattern of pure PMMA sample. (b): Indexed XRD pattern of cobalt oxide dispersed PMMA nanocomposite film.

Scanning Electron Micrograph (SEM)

The morphological image of cobalt oxide dispersed PMMA nanocomposite film is shown in Figure 2. Uniform dispersion of cobalt oxide particles in the PMMA matrix can be observed. A sheet like arrangement of the polymer with cobalt oxide particles can be observed. Irregular shaped Co_3O_4 particles agglomeration can also be seen. A compact structure formed by close packing arrangement of oxide particles with polymer is observed. It is also observed in the image that, some spherical shaped particles dispersed in the PMMA matrix.

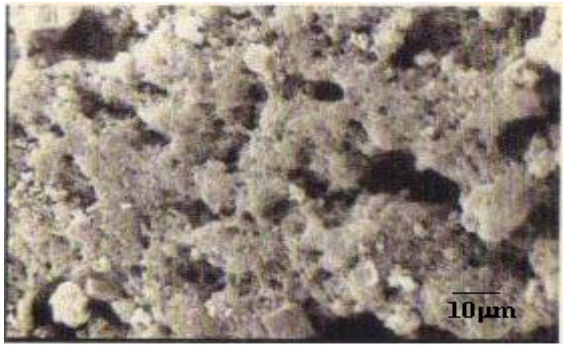

Fig. 2.SEM image of cobalt oxide dispersed PVA nanocomposite film.

Fourier transmission infrared (FTIR) spectroscopy studies

Figures 3a-b shows FTIR spectrum of pure PMMA and PMMA-Co_3O_4 composite film respectively. Some additional peaks and shift in the frequencies in the PMMA composite spectrum in comparison with that of pure PMMA is observed, which confirms the formation of PMMA composite. These additional peaks and shifts of the peaks in composite spectrum are due to the presence of inserted cobalt oxide in the polymer matrix and it may be possible due to H-type interactions between cobalt oxide and polymer matrix. Some characteristic peak of metal-oxygen [13] below 1000 cm^{-1} appeared in the composite spectrum indicating the presence of cobalt oxide particles. The cobalt oxide peaks in the composite spectrum due to masking of oxide particles in the polymer matrix.

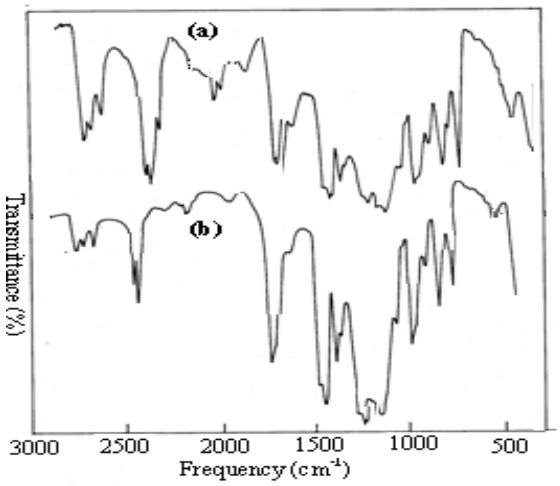

Fig. 3. FTIR spectrum of a) pure PMMA b) PMMA-Co_3O_4 composite sample.

Thermal Study

The thermogravimetric analysis (TGA) and the differential thermal analysis (DTA) of PMMA-Co_3O_4 polymer composites is carried out for its thermal behaviour. The TGA data for PMMA composite shows that it is stable up to 280 °C and the degradation starts at around 300°C with concomitant weight loss as shown in the Figure 4. The endothermic peak in the DTA plot at around the same temperature is further evidences.

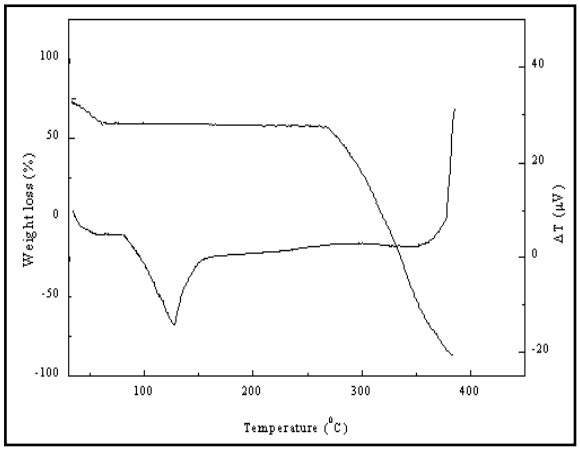

Fig. 4. TGA/DTA trace of PMMA-Co_3O_4

CONCLUSION

Cobalt oxide dispersed poly (methyl methacrylate) composite film was prepared by a simple solvent casting method. The development of crystallinity in PMMA is due to incorporation of cobalt oxides in to PMMA matrix. Presence of cobalt oxide particles in the PMMA matrix changes the morphology of the pure PMMA. Additional reflections and shifted frequencies in the FTIR spectrum confirm the presence the composite formation. Thermal study concludes the increase in thermal behavior of the polymer composite compared to its pure polymer.

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