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CHARACTERIZATION OF PANI-PVAc COMPOSITE THIN FILMS USING XRD ANALYSIS

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ABSTRACT

Polyaniline (PANI) is the one of the most promising conducting polymer due to a good combination of properties, stability, price and ease of synthesis by different routes. In this study, PANI and poly (vinyl acetate) (PVAc) composite thin films were synthesized by chemical oxidative polymerization method with the solution of ammonium per sulphate as an oxidant in methanol. The films were prepared with 10-50 wt. % of PVAc. The thickness was in the range of 1-4 μm for all films. The films were characterized by XRD technique. This method can be used to show the perfection of PVAc network structure in the composite. XRD technique has been used to determine the possible crystallinity in the composite films. The XRD of all samples was carried in the 2θ range from 10 to 100 degree.

Key words: Polyaniline, Composites, Thin films, XRD.

INTRODUCTION

Polyaniline (PANI) is one of the most attractive conducting polymers, which has potential applications in electrochemical sensors and biosensors[1,2]. Owing to its good biocompatibility and inherent electroactivity, PANI can act as a suitable matrix for immobilization of biomolecules and mediator for redox and enzymatic reactions[3,4], which exhibits impressive signal amplification and antifouling properties[1].

Polyaniline, Polypyrrole, Polythiophene and other conducting polymers prepared from heterocyclic monomers exhibit high conductivity and stability. However, their mechanical properties e.g. brittleness and a low level of processability are inferior compared to conventional polymers. Several attempts have been made to improve the mechanical properties of the conducting polymers by forming blends or composites with other polymers. A combination of conventional polymers or copolymers[5] with conductive polymers allows the creation of new polymeric materials with interesting electrical properties. The system containing two different conducting polymers can be prepared by various methods. An electrochemical technique is one of the most widely used methods for their preparation .The electrolysis of two monomers gives generally a copolymer as in the case of pyrrole and bithiophene[6]. The same study reports that the bithiophene/pyrrole ratio in the copolymer could easily be adjusted by controlling the polymerization potential. Funt et al.' stated that the electrical conductivity of the copolymer obtained by the joint electrolysis of pyrrole and 2,2'-bithiophene was quite high.

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In the present work, PANI-PVAc composite thin films were synthesized by chemical oxidative polymerization method with the solution of ammonium per sulphate as an oxidant in methanol. The wt. % of PVAc was varies from 10-50 wt. %. The films were characterized by XRD and SEM techniques.

EXPERIMENTAL

Poly(vinylacetate) were obtained from M/S Lobo Chemie (INDIA). Aniline monomer was received from M/S E. Merck (India). The oxiding agent ammonium per sulphate was obtained from M/S SD. Fine chem. (India).

Polyaniline was synthesized at room temperature by mixing aniline with ammonium per sulphate of the ratio 2 : 1 M and PVAc in 5 mL methanol. When ammonium per sulphate was added to the solution of aniline and PVAc, a dark brownish homogeneous solution was obtained which was then poured on chemically cleaned and mercury leveled glass substrate to prepare the films of composite. Initially the mixture of ammonium per sulphate and PANI-PVAc solution was homogeneous but phase separation took place as the polymerization proceeds. The aniline polymerization progresses because the evaporation of solvent increases the oxidation potential of cast solution. Phase separation leads to formation of conducting PANI network throughout the film. After evaporation of solvent the thin films were formed. The thicknesses of all the films was measured by using Digimatic micrometer (Mitutoyo make, Japan) with least count 1 μm and are in the range of 1-4 μm.

The films of composite were characterized by using X-ray (XRD) and scanning electron microscopic (SEM) techniques. X-ray diffraction pattern of films was recorded on a Phillips-1730 (PANalytical) X-ray diffractometer using Cu K α radiation (λ = 1.54 Å). The diffractogram was in terms of 2θ in the range 10^o–100^o. The morphology of films was investigated by using JEOL-JSM (Model-5200) SEM instrument.

RESULTS AND DISCUSSION

X-Ray Diffraction (XRD)

The X-Ray Diffraction technique has been used to determine the possible crystallinity in the composite films. The X-ray diffraction of all samples was carried in the 2θ range from 10 to 100 degree and shown in Fig. 1.

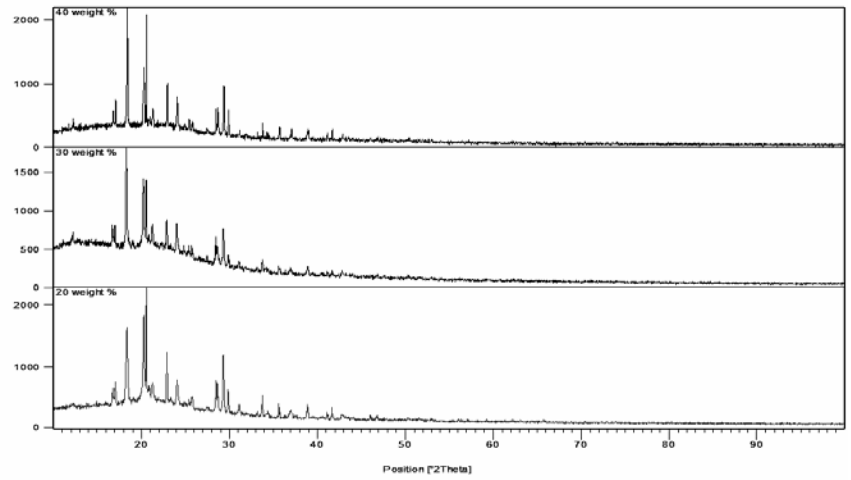

Fig. 1: The XRD spectra of the PANI-PVAc composite films with 20, 30 and 40 wt. % of PVAc

The absence of peak in the intensity versus 2θ curve represents complete amorphous state of the sample. Indication of peak to peak in curve suggests the formation of phase or phases in the composite during polymerization process. All the spectra for 20, 30 and 40 wt. % PVAc wt. % reveal an amorphous halo in the low 2θ region. It is due to short-range order and indicating that these PANI-PVAc composite films are amorphous. XRD pattern shows semi-crystalline nature of composites. The average crystallite size from a sharp peak is estimated by using the Scherer's formula[7]. The average crystallite size was found to be 220 nm for 20 wt. %, 171 nm for 30 wt. % and 215 nm for 40 wt. %.

Conductive polymer films are intrinsically conductive of both ions and electrons. The electronic conductivity of the polymer films is governed by the size of the counter ion in corporated into the polymer matrix during the polymerization process and by the oxidation state of the polymer. With increasing size of the anion the conductivity decreases dramatically. The exchange of incorporated counter anion into the polymer matrix by the others present in the solution does not provide any change in the conductivity of the originally synthesized polymer. That was observed independently on the size or kind of anion. The transport of the electrons that occurs by the site-site doping or by the electron self exchange mechanism between adjacent redox sites is described by the charge transfer diffusion constant[6].

CONCLUSION

The films of PANI-PVAc were synthesized by chemical oxidative polymerization method. The thickness was in the range of 1-4 μm for all films. The XRD study reveals that the films of 20, 30 and 40 wt. % PVAc wt. % showed an amorphous halo in the low 2θ region and semi-crystalline nature. The partly crystalline behavior was seen from SEM as in XRD. The phases so obtained are due to ammonium per sulphate.

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