

Temperature Dependence Electrical Conductivity of Synthesized Poly O-Anisidine Fly Ash Composites.

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Manuscript Details

Available online on <http://www.irjse.in>

ISSN: 2322-0015

Cite this article as:

Dahegaonkar AD, Kondawar SB and Tabhane VA. Temperature Dependence Electrical Conductivity of Synthesized Poly O-Anisidine Fly Ash Composites., *Int. Res. Journal of Science & Engineering*, February 2020, Special Issue A7 : 227-233.

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ABSTRACT

In recent years composites reinforced with fly ash particulates have attracted considerable interest due to their inherent good mechanical properties and low cost. A new composite (POAS/FA) system using low-cost materials poly(o-anisidine) an substitute derivative of polyaniline and fly ash was prepared by chemical oxidation method in presence of ammonium persulphate (APS) with various composition of 10, 20, 30, 40 and 50 wt % of fly ash in conducting polymer. The structural characterizations of the composites were carried out by using XRD, SEM and UV-Vis spectroscopy techniques. A homogenous semi crystalline structure of the composites has been observed. Scanning electron microscopy (SEM) shows a good homogeneous mixture of the composite materials. In UV-Vis technique, characteristic bands in POAS have been shifted to longer wavelength due to incorporation of FA. Electrical conductivity studies were carried out by four in-line probe DC electrical conductivity measuring instrument. The electrical conductivity was found within the range of semiconductor region. An expected decrease in conductivity was observed with addition of FA into poly(o-anisidine). Electrical properties of FA can be used to control the conductivity of POAS to make it suitable for various applications.

Keywords: Conducting polymers, Poly(o-anisidine), Fly ash, Composites, Electrical conductivity

INTRODUCTION

Among the conducting polymers polyaniline and its derivatives have attracted much attention due to its ease of synthesis by chemical or electrochemical polymerization, ammonium persulphate is generally used as the oxidizing agent for the preparation. Although the chemical method offers mass production at a reasonable cost; the electrochemical method involves the direct formation of conducting polymer thin film with better control of thickness and morphology, which are suitable for application in electronic devices [1-6]. Polyaniline and its derivatives are mainly used as coating to improve the corrosion resistance of oxidizing metals. These coating have also been considered for several applications such as the electronic industry; biosensor application [7-13]. Polyaniline has shown variety of application such as rechargeable batteries, electro catalysis, electrochromic display; gas separation and biosensors. However the role of structural and mechanical behavior of polyaniline and its derivatives for their applications to biosensor has not yet been explored [14]. The polymers of substituted derivatives of aniline exhibit slightly lower conductivity than that of unsubstituted PANI [15].

Fly ash is being considered as a waste material produced from thermal power plants is creating severe environmental pollution. It is creating severe environmental pollution; so much research is being conducted for more than two decades for its proper utilization as well as to control environmental pollution in the surrounding areas of power plants. Its utilization in industry or in market sector may bring economic and ecological benefits and impact technological development [16-25]. A composite material is a combination of two or more materials (in certain proportions) whose performance characteristics exceed that of its individual components. Now a day polymer composites are of interest; these can provide great strength and stiffness along with resistance to corrosion [26-27]. Hence development of low cost composite materials using conducting polymer industrial waste materials from local resources is an active field of research [28-29]. Presently much research attention is being given to use such materials for capacitor dielectric, insulation,

encapsulation, multilayer or ceramic chip, printed circuit boards etc. Fly ash based composites exhibit low dielectric constant and stabilized dielectric loss at high frequencies of applied field and hence are suitable for electronic application [30-32]. Electrical conductivity is a very useful method in solid state electronic system. This work reports on the synthesis of POASFA composite by chemical oxidative polymerization method. The study shows the synthesis and characterization of POASFA composite by SEM, XRD, UV-VIS and Electrical conductivity techniques.

METHODOLOGY

The GR grade o-anisidine was purified by distillation under reduced pressure. A fine fresh clean and pure FA powder was collected from the Thermal Power Station, Chandrappur, India. FA contains elements like Cu, Pb, Cd, Ag, Mn, Ti, Na, Mo, S, P, Zn and Cl in different concentrations [33]. 0.4 M of distilled o-anisidine was added to the solution of 0.4 M of ammonium persulfate $[(\text{NH}_4)_2\text{S}_2\text{O}_8]$ and this reaction mixture was stirred continuously at room temperature to obtain poly(o-anisidine). To this reaction mixture, varied weight per cent of fly ash powder (10, 20, 30, 40 and 50) was added to form poly(o-anisidine)-fly ash composites. The obtained product was filtered and washed thoroughly with methanol (CH_3OH) and the sample was dried under vacuum for more than 24 h at room temperature. The obtained composites were pressed in the form of circular pellets of 1 cm diameter and 1 mm thickness in hydraulic press at five metric ton pressure. The conductivity measurements were carried out by a four-probe method. The SEM images of poly(o-anisidine)-fly ash composites were investigated using Field Emission Gun Scanning Electron Microscope. The X-ray diffraction patterns of the samples in this present case were recorded on Philips PW-1700 X-ray diffractometer using $\text{CuK}\alpha$ radiation of wavelength 1.544 \AA . Continuous scan of 2° minimum with accuracy of 0.01. The characterization of poly(o-anisidine) and its composites by spectroscopic methods is important, as it gives information not only about various molecular-level interactions but also on the type of charge carriers. The variation of conductivity with temperature has been studied for all samples. Fly ash is a finely divided amorphous powder with the

particle size ranging from 150 nm–120 μ m. It is abrasive and refractory in nature. In the present study the fly ash employed is of cenosphere type, collected from Thermal Power Station Chandrapur, India.

RESULTS AND DISCUSSION

A. UV-Vis Properties:

Figure 1. Shows the UV –Vis absorption spectra of self-doped POAS with different weight percentage of fly ash in DMF .POAS showed two characteristics band at 290 nm in UV correspond to Π - Π^* transition along the backbone of the POAS chain and 550 nm in visible corresponds to inter ring charge transfer ration of benzenoid to quinoidmoities [34].The characteristics bands in POAS have also been shifted to longer wavelengths due to incorporation of fly ash indicating the interaction between quinoid ring and fly ash observed in case of POAS[35].

B. Scanning Electron Microscopy (SEM):

SEM of POAS shows porous, non-uniform structure. POASFA composites shows the formation of dine base form of POAS significantly changes the aggregate state of polymeric molecular chain. The incorporation of metal oxides into the polymeric network induces uniform porosity and is expected to be advantage for gas and biosensing application. A very high magnification revels the homogeneous distribution of fly ash (cenospere) particle. It is

seen from the micrograph that clusters and granular structure of POAS is maintained even after the addition of fly ash in POAS. Hence a network of fly ash and granular POAS has been formed in case of composites [36]. The presence of -OCH₃ group in each benzene ring of the poly (o-anisidine) push apart the dopant and it make more disordered system as compared to that of polyaniline, as seen from its SEM which shows highly porous. It has been revealed that after binding of poly (o-anisidine) with fly ash the morphology has been changed. The change in the particle size was noticed due to incorporation of the different weight percentage of fly ash in poly (o-anisidine) matrix. The original fly ash is mainly constituted by compact or hollowed sphere but with a regular smooth texture. Also some quartz particles residue of un-burned coal or some vitreous unshaped fragments could be seen.

C. X-ray Diffraction Spectroscopy (XRD):

Figure 3 shows the XRD of pure POAS and POAS composites. It has been suggested that XRD study that POAS undergoes interfacial interaction with FA crystallites and losses its own morphology by its mixing with fly ash .POAS fly ash composites show peaks of fly ash as well as POAS indicating that fly ash crystallites have been uniformly mixed within the polymer chain. Carefully of X-ray diffraction of POASFA composite suggested that it exhibits semi-crystalline behavior. No structural change has been observed in fly ash dispersion in polymerization of reaction of POAS [37].

Table 1: Conductivity of POAS, FA and POASFA composite with temperature

Sr. No	Polymer	Conductivity (S/cm) at Room Temp.	Conductivity (S/cm) at 100°C	Conductivity (S/cm) at 150°C
1	POAS	4.68×10^{-4}	1.21×10^{-3}	1.52×10^{-3}
2	POASFA-10	1.31×10^{-5}	2.99×10^{-5}	5.29×10^{-5}
3	POASFA-20	3.81×10^{-6}	1.811×10^{-5}	3.73×10^{-5}
4	POASFA-30	8.53×10^{-7}	3.11×10^{-6}	5.37×10^{-6}
5	POASFA-40	8.91×10^{-8}	3.92×10^{-7}	7.16×10^{-7}
6	POASFA-50	9.42×10^{-9}	1.19×10^{-8}	1.56×10^{-8}
7	FA	1.01×10^{-11}	3.62×10^{-11}	9.50×10^{-11}

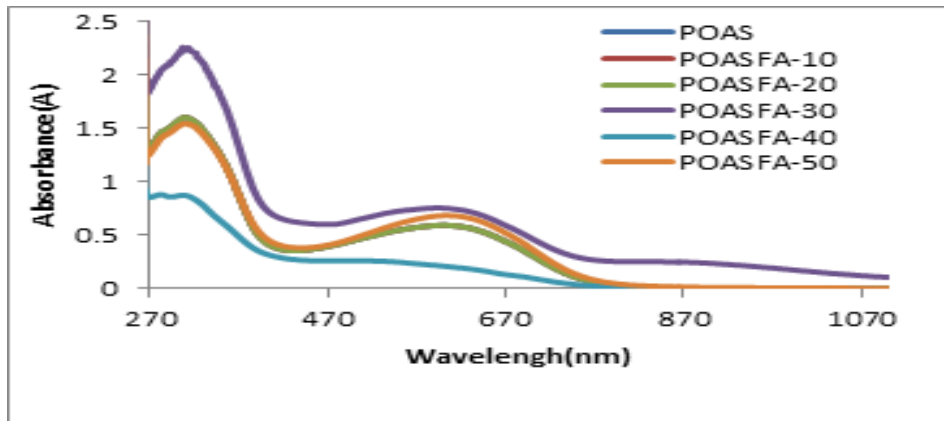


Figure 1. UV-Vis spectra of POAS and POASFA composites

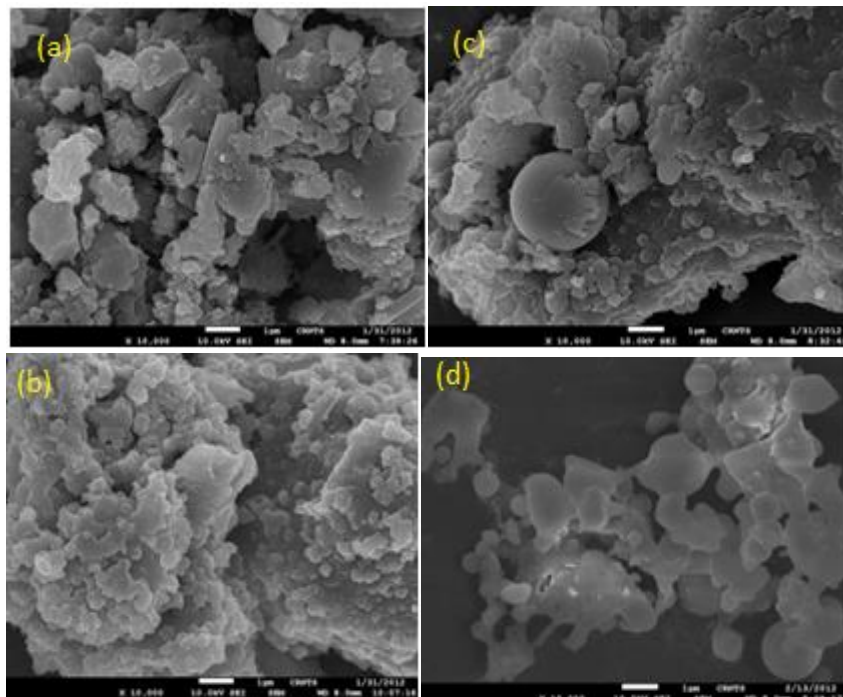


Figure 2 Scanning electron micrograph of a) POAS b) POASFA-10 c) POASFA-40 d) FA

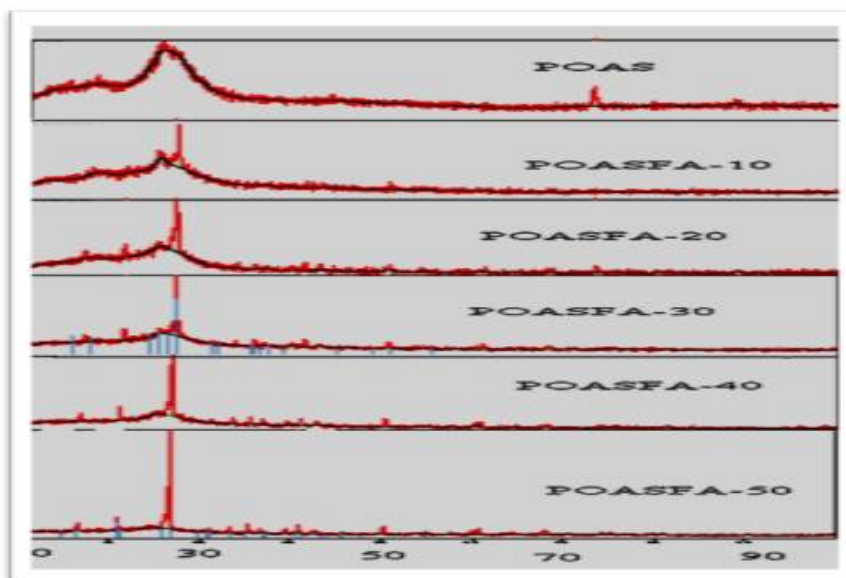


Figure 3 XRD spectra of chemically synthesized of POAS and POASFA composites

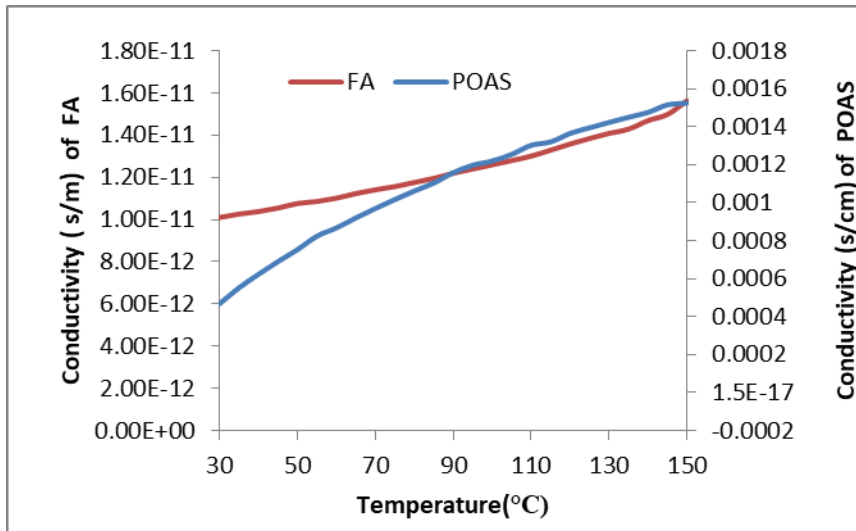


Figure 4 Electrical Conductivity POAS and FA with temperature

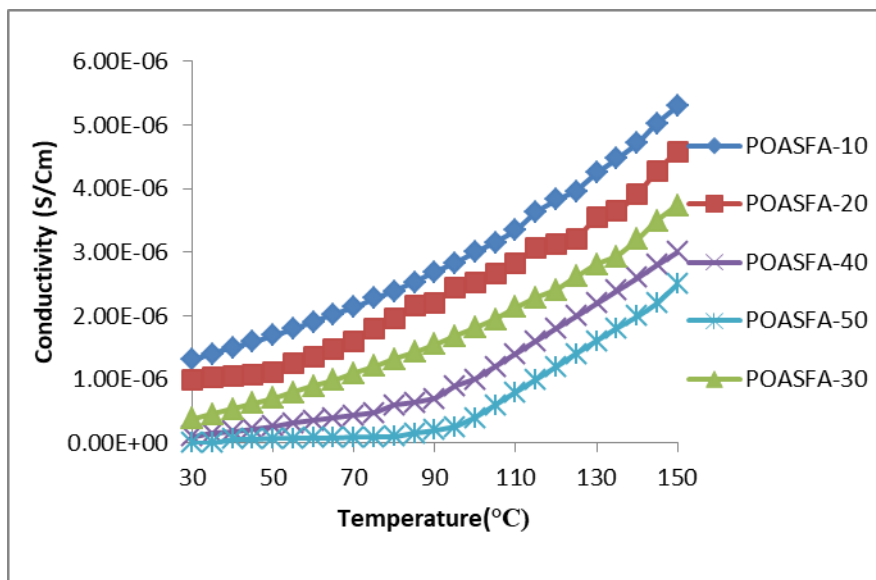


Figure 5 Electrical conductivity POASFA composites with temperature

D. Conductivity measurements:

Figure 4 shows the dc conductivity for pure POAS and FA with temperature. The electrical measurements declare that at room temperature poly o-anisidine shows semiconductor behavior (4.68×10^{-4} S/cm) and Fly ash act as an insulator (1.01×10^{-11} S/cm). Also the conductivity was increased by increasing the temperature. Figure 5 shows the variation of dc conductivity with the temperature for POASFA composites. The dc conductivity of POAS decreases by the addition of fly ash. In polymers conduction takes place by the mobility of charge carriers from an occupied site to an unoccupied site. The defects generally increase with temperature and are independent of

temperature at lower temperatures. At higher temperatures additional defects are created. At lower temperatures, they are frozen. Hence, the conductivity variation with temperature is divided into two regions: (I) pertaining to higher temperature and (II) pertaining to lower temperature. It may be easily noted that the conductivity decreases with the increasing concentrations of FA. The room temperature resistivity increases (conductivity decreases) with the addition of FA in POAS. From the study of dc conductivity as a function of temperature, it was inferred that both the POASFA composites as well as the pure POAS were semiconducting in nature. Moreover, the conductivity was expectedly found

to decrease with the addition of FA in POAS [38]. The lower conductivity relative to polyaniline may be explained by an increase of the interchain distance and diluting effect of the charge carriers caused by the presence of bulky methoxy group in the polymer. The substituent present at the ortho position of the benzene ring induces additional deformation along the polymer backbone. This in turn results in a decrease of the degree of conjugation and hence a decrease in conductivity [39]. Table 1 show the effect of temperature on the electrical conductivity. This means that by adding fly ash poly o-anisidine converted from semiconductor behavior to insulator behaviour which can uses in electronic industry [40].

CONCLUSION

With more than 100 million tones of fly ash produced in India, use of fly ash for the preparation of poly(o-anisidine)-fly ash composites will in no way help in its bulk utilization. Still the authors have made an effort towards the better utility of fly ash by synthesizing poly(o-anisidine)-fly ash composites. The detailed characterization of these composites was successfully carried out through XRD; SEM and UV-Vis. Electrical property is an important property of material. The results of electrical show a strong dependence on the weight percent of fly ash in poly(o-anisidine) and fly ash can be used to control the conductivity of poly(o-anisidine) which can be best suitable for various electronics application.

Conflicts of interest: The authors stated that no conflicts of interest.

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