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Synthesis and characterization of acrylic resin/activated carbon composites

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Activated Charcoal (AC) filled acrylic resin (AR) based composites have been prepared by solution processing technique. Different compositions were formulated by variation of AC content between 1 to 30 wt % and the formed composites have been characterized by various techniques to establish a correlation between the filler concentration and the structural, thermal, electrical and morphological attributes. The FTIR spectra show the presence of AC in AR matrix whereas XRD patterns confirm the incorporation of AC particles between lamellar structures of AR. The TGA investigation revealed that composites are thermally stable up to 200°C after which they display multistep weight loss due to removal of functional groups and decomposition/carbonization of AC as well as polymeric backbone of AR. The SEM images show that incorporation of AC particles leads to systematic change in the morphology of composites. The electrical measurements show that 30 wt % AC loading composite displays much lower resistivity (10^4 Ω/Sq) as compared to pure AR (10^{11} Ω/Sq) and suggest its suitability as static dissipative material. Further, the porous structure and electrically conducting nature of the composites suggest their suitability for making electromagnetic interference shielding coatings.

Keywords: Acrylic resin, Activated carbon, Antistatic and static dissipative composites, Electrical conductivity, Electromagnetic interference shielding

1 Introduction

The polymer based composites have drawn enormous attention due to their unique properties and potential applications in the diverse areas such as power, automotive, aerospace, medicine, construction and electronic sectors[1-5]. Particularly, the inorganic filler loaded polymer composites are widely studied due to excellent structural, thermal and barrier properties as well as good chemical resistance[1,2,6-9]. However, the carbonaceous filler [carbon black (CB), graphite, activated carbon (AC), fullerenes, carbon nanofibers (CNFs), carbon nanotubes (CNTs) and recently graphene] based composites are becoming increasingly popular as they display additional properties such as good electrical/thermal conductivity, low density, excellent corrosion resistance, improved flexibility and superior mechanical properties[1,3,5,10-18]. These properties, particularly the electrical conductivity, further pushed the range of applications of such composites to encompass areas such as energy generation/storage, environmental pollution monitoring/ control, electronics/optoelectronics, biosensing, electroactive parts, antistatics and electromagnetic interference (EMI) shielding materials[3,5,13,15,17-27]. It is important to note that the properties of such composites are closely related to properties of constituents (dispersed filler as well as polymer matrix), their relative abundance and degree of intermixing as well as extent of interfacial bonding between them. Among various matrix polymers, acrylics are extremely popular due to low cost, facile processing via solution/thermal route, ability to accommodate high filler concentration, ability of acrylic resins (AR) to form scratch/abrasion resistant, mechanically strong and tough coatings with good barrier properties[28-31].

The synthesis of electrically conducting AC/AR composites by solution processing technique has been studied in the present paper. Different compositions were formed by variation of weight ratio of phases and the formed AC/AR composites were characterized for their spectral, structural, thermal, electrical and morphological attributes using techniques such as FTIR, XRD, TGA, resistivity measurement and SEM, respectively. These composites with easy processing, low density, excellent corrosion resistance, good electrical properties and porous morphology are expected to guard the electronic articles from static charges or EMI effects.

2 Experimental Details

2.1 Materials

Acrylic resin (AR, 50% solid content) was purchased from Pidilite industries limited, India. Activated carbon (Merck) and xylene (Rankem, India) were of analytical grades and used as received without further purification.

2.2 Synthesis of AC/AR composites

The AC/AR composites were prepared by solution processing technique as shown schematically in Fig. 1. In a typical reaction, about 14 g of AR was dissolved in 20 ml of xylene and a known amount of AC powder was added. The contents were magnetically stirred for 1 h, casted in a glass petridish and dried at 40°C for 24 h to form coatings (~300 micron thickness). Weight ratio of AC:AR was varied to form composites containing 0%, 1%, 5%, 10%, 20% and 30% of AC.

2.3 Measurements and structural characterization

Infrared transmission spectra of these activated carbon synthetic acrylic composites were recorded on Agilent Cary 630 FTIR spectrophotometer in 4000-400 cm^{-1} region at ambient temperature by using ATR accessory. X-ray diffraction patterns in the 2θ range of 10°-70° were obtained using Bruker-D8 advanced diffractometer using Cu Kα line (λ= 1.540598 Å) radiation source. Morphologies were recorded from scanning electron microscope (SEM, Leo-440, UK). The thermal stability measurements were performed using TGA (Mettler Toledo TGA/SDTA 851e, Switzerland) system in the temperature range 25°-600°C at heating rate of 10°C/min under inert atmosphere (nitrogen gas).

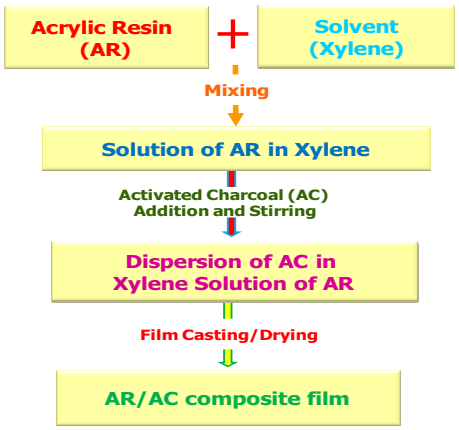

Fig. 1 — Schematic representation of formation of AC/AR composites by solution processing route

3 Results and Discussion

3.1 FTIR measurements

Figure 2 shows the IR transmission spectra of pure synthetic acrylic and different activated carbon concentration dispersed composites film. These spectra are dominated by the acrylic peaks[32] and addition of activated carbon results in the shift in peak position of some band. In these spectra, we have observed water and methylene/methyl group stretching mode in 3500-2900 cm^{-1} region at 3425 cm^{-1} and 2934 cm^{-1}, respectively. The symmetric trans-conformation of C=O stretching mode of ester group appeared at 1730 cm^{-1} as a strong band. The medium intensity peak at 1242 cm^{-1} confirms the symmetric stretching mode of trans-conformation of monodentate C(O)–O ester functional group. The band at 1144 cm^{-1} with weak shoulder at 1075 cm^{-1} is assigned as C–O stretching coupled with in-plane bending of O-H of poly acrylic and aliphatic C-H/C=O stretching mode, respectively. While the weak bands at 696 and 467 cm^{-1} are assigned as CCC bending in s-*trans* form and CH_2 wagging mode and CCH rocking mode in s-*cis* conformer and CH_2 twisting mode.

3.2 XRD patterns

Figure 3 shows the pure AR displays a broad peak located at 2θ = 15.95° as well a weak peak at 2θ = 30.92°. As AC is introduced into the AR matrix, a systematic shift in the peak position and intensity was observed along with the evolution of a

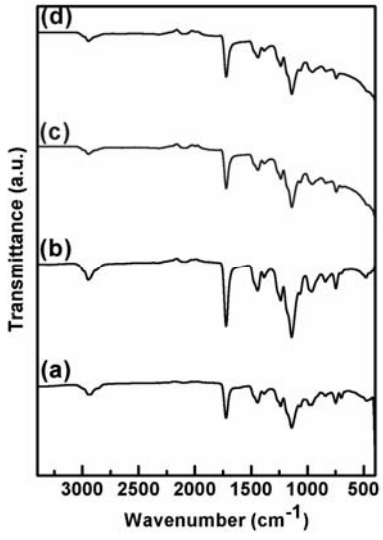

Fig. 2 — IR transmission spectrum of (a) AR and its AC based composites containing, (b) 1 wt% (c) 10 wt % and (d) 30 wt % of AC

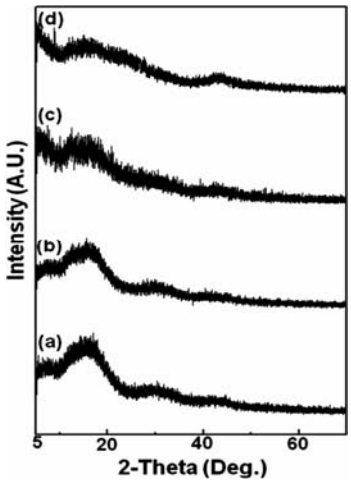

Fig. 3 — X-ray diffraction patterns of (a) AR and its AC based composites containing, (b) 1 wt% (c) 10 wt % and (d) 30 wt % of AC

characteristic peak of AC at 2θ = 43°. Particularly, 2θ = 15.95° peak of AR shifts towards lower angle, which shows the incorporation of AC particles between AR lamellae and confirms the formation of composites.

3.3 TGA and thermal stability

Thermogravimetric (TG) traces of AC/AR composites (Fig. 4) show a multi-step thermal decomposition with no sharp transition between steps.

It can be seen that the composites are thermally stable up to 200°C with no appreciable weight loss. However, they show weight loss of ~ 4-5% in the first step (i.e. between 200°C to 230°C) followed by weight loss of 7-12% in the second step (~230-275°C), that may be correlated with the loss of functional groups and fragmentation of polymeric chains. Similarly, the weight loss of 12-13% in the third loss step (~275-315°C) and major weight loss of 50-60% in the steep forth step (~315-450°C) can be ascribed to the thermal decomposition of the polymeric backbone and carbonization of fragments as well as AC particles to form char residue. It can be seen that as the AC content of the composites increases, the char residue content increases which confirms the increase in amount of incorporated AC.

3.4 Resistivity measurements

The pure AR is a poor electrical conductor and the coatings exhibit very high resistivity value ~10^{11} Ω/Sq. However, the incorporation of electrically conducting AC particles lead to a decrease in resistivity due to the formation of conducting links

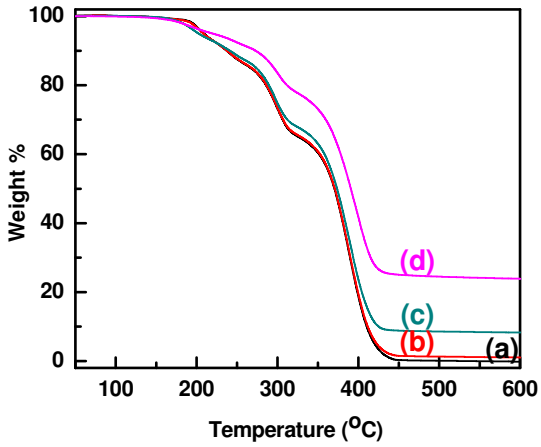

Fig. 4 — TGA curves of (a) pure acrylic resin and its composites containing (b) 1 wt%, (c) 10 wt % and (d) 30 wt % of AC

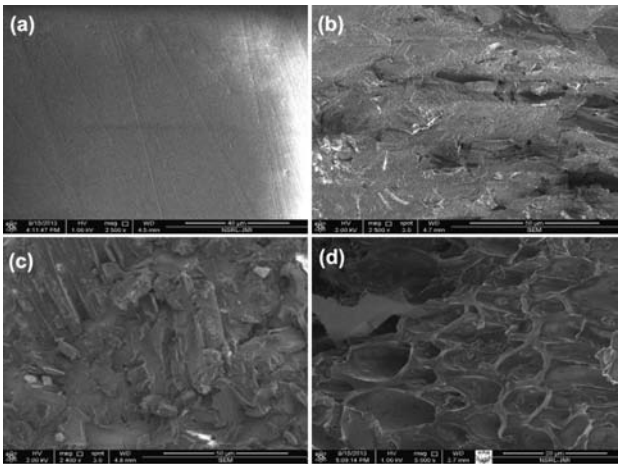

Fig. 5 — SEM images of (a) pure acrylic resin and its composites containing (b) 1 wt%, (c) 10 wt % and (d) 30 wt % of AC

within AR matrix. Therefore, composite with 30 wt % loading of AC display very low resistivity (~10^4 Ω/Sq) which is about 7 orders lower than that of neat AR. The composites with >10 wt % AC also satisfy the static dissipative criteria[24,26,33-36] of surface resistivity less than 10^9 Ω/Sq and can be used for making static charge free coatings or encasing materials.

3.5 Morphological details

Figure 5 shows the SEM micrographs of AR and its AC based composites. It can be seen that pure AR film shows (Fig. 5a) smooth morphology with homogenous patterns throughout the scanned region which confirms the semi-crystalline nature of pure acrylic.

The incorporation of AC into AR matrix affects the crystallization of matrix and the systematic change in

morphology (Fig. 5b to 5d) was observed with the increase in AC content. Particularly, the composite with 30 wt % loading of AC shows that presence of porous and channels made up of electrically conducting AC infiltrated with non-conducting AR matrix. It is important to point out that the porous composites with electrically conducting filler are known to display good EM radiation blocking efficiency due to high surface area and possibility of multiple reflections[3,5,19,21,26]. Further studies to assess the actual EMI shielding effectiveness value and the shielding mechanism are under investigation.

4 Conclusions

AC/AR composites have been prepared by dispersing 1 to 30 wt % of AC in AR matrix using a simple solution processing route. FTIR spectra display slight shifting in the peak position of AR whereas XRD patterns give the superimposed peaks of AC and AR which confirm the presence of dispersed AC particles in AR matrix. These composites show good thermal stability up to 200°C and decompose afterwards leaving behind char residue which increases with increase in AC content. SEM images revealed that AC particles affect the morphology of composites and for 30% loading sample, porous structure of AC coated with AR phase can be clearly seen. The pure AR shows very high resistivity (10^{11} Ω/Sq) which decreases to 10^4 Ω/Sq for composite with 30 wt % loading of AC. These composites satisfy the static dissipative criteria and may be useful for making static charging free encasing materials. The combination of porous architecture and very low surface resistivity are expected to provide enhanced absorption of EM radiations and can be envisaged as a potential candidate for making anti-radiation coatings/paints.

References

1 Hussain F, Hojjati M, Okamoto M & Gorga RE, *J Composite Mater*, 40 (2006) 1511.
2 Okada A, Kawasumi M, Usuki A, Kojima Y, Kurauchi T & Kamigaito O. *MRS Symp Proc.* Pittsburgh PA: Materials Research Society; 171 (1990) 45.
3 Saini P, Arora M, in *New Polymers for Special Applications* (ed. Gomes AD), Intech: Croatia, (2012) pp 71 http://www.intechopen.com/download/pdf/38964.
4 Godovsky DY. *Adv Polym Sci*, 153 (2000) 163.
5 Saini P, *Electrical Properties and Electromagnetic Interference Shielding Response of Electrically Conducting Thermosetting Nanocomposites*, in *Thermoset Nanocomposites* (ed V Mittal), Wiley-VCH Verlag GmbH & Co. KGaA, Weinheim, Germany, doi: 10.1002/ 9783527659647. ch10, 2013.
6 Alexandre M, Dubois P. *Mater Sci Eng:R,* 28 (2000) 1.
7 Ray SS & Okamoto M. *Prog Polym Sci,* 28 (2003) 1539.
8 Gao F. *Materials Today*, 7 (2004) 50.
9 Cox H, *4th World Congress in Nanocomposites*, EMC, San Francisco, 1–3 September 2004.
10 Yasmin A, Luo J & Daniel IM. *Composites Sci Tech*, 66 (2006) 1182.
11 Chen GH, Wu DJ & Weng WG. *J Appl Polym Sci*, 82 (2001) 2506.
12 Bower C, Rosen R, Jin L, Han J & Zhou O. *Appl Phys Lett*, 74 (1999) 3317.
13 Cooper CA, Ravich D, Lips D, Mayer J & Wagner HD. *Composites Sci Tech*, 62 (2002) 1105.
14 Lau K T & Hui D, *Composites Part B*, 33 (2002) 263.
15 Gojny FH, Wichmann MHG, Kopke U, Fiedler B & Schulte K. *Composites Sci Tech*, 64 (2004) 2363.
16 Allaoui A, Bai S, Cheng H M & Bai J B, *Composites Sci Tech*, 62 (2002) 1993.
17 Stankovich S, Dikin D A, Dommett G H B, Kohlhaas K M, Zimney E J, Stach E A, Piner R D, Nguyen S T & Ruoff R S, *Nature,* 442 (2006) 282.
18 Wu J & Chung DDL, *Carbon*,40 (2002) 445.
19 Yang Y, Gupta MC, Dudley KL & Lawrence RW, *Nanolett*,*5* (2005) 2131.
20 Saini P, Choudhary V, Singh BP, Mathur RB & Dhawan SK. *Mater Chem Phys,* 113 (2009) 919.
21 Li N, Huang Y, Du F, He X, Lin X, Gao H, Ma Y, Li F, Chen Y & Eklund P C. *Nano Lett,* 6 (2006) 1141.
22 Saini P, Choudhary V, Sood KN & Dhawan SK. *J Appl Polym Sci*, 113 (2009) 3146.
23 Saini P, Arora M, Gupta G, Gupta BK, Singh V N & Choudhary V. *Nanoscale*, 5 (2013) 4330.
24 Saini P, Choudhary V, Singh BP, Mathur RB & Dhawan SK. *Synth Met*,161 (2011) 1522,
25 Colaneri NF & Shacklette LW. *IEEE Trans Instrum Meas,* 41 (1992) 291.
26 Saini P & Choudhary V. *J Nanoparticle Res,* 15 (2013) 1415.
27 Tripathi SN, Saini P, Gupta D & Choudhary V, *J Mater Sci*, 48 (2013) 6223.
28 Baldacchini T, LaFratta CN, Farrer RA, Eugene F, Teich MC, Bahaa EA, Michael S, Naughton J & Fourkas JT, *J Appl Phys*, 95 (2004) 6072.
29 Vallittu P K. *The Journal of Prosthetic Dentistry*, 81 (1999) 318.
30 Park H-S, Yang I-M, Wu J-P, Kim M-S, Hahm H-S, Kim S-K & Rhee H-W. *J Appl Polym Sci*, 81 (2001) 1614. doi: 10.1002/app.1592.
31 Xu J L, Zhong Z C, Huang Z X & Luo J M, *J Alloys and Compounds*, 570 (2013) 28.
32 Belaidi O, Bouchaour T & Maschke U, *Org Chem Int*, 348379 (2013) 14.
33 Saini P, Choudhary V, Vijayan N & Kotnala R K, *J Phys Chem C*, 116 (2012) 13403.
34 Saini P & Choudhary V, *Indian J Pure & Appl Phys*, 51 (2013) 112.
35 Ono H, Ohsawa A & Tabata Y. *J Electrostat,* 57 (2003) 355.
36 Chubb J N, *J Electrostatics*, 65 (2007) 607.