

Dielectric and Electromagnetic Shielding Investigations of Activated charcoal Loaded Acrylic composite Films

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ABSTRACT

Activated carbons (AC) between 1 wt% to 30 wt% have been mixed to synthetic acrylic (AR) resin by solution casting method. The prepared composites have been characterized by various analytical techniques to draw a detailed correlation between the filler and the structural, dielectrical, morphological and electromagnetic properties. The XRD analysis confirms the incorporation of AC particles between lamellar structures of AR. The SEM images describe that incorporation of AC particles leads the systemic change in the morphology of composites. The EMI shielding attributes to the highest microwave conductivity means high attenuation levels that means blocking of incident radiation demonstrate the potential of these materials for making future microwave shields.

Keywords: Acrylic resin, Activated carbon, Antistatic & static dissipative composites, electrical conductivity, electromagnetic interference (EMI) shielding.

I. INTRODUCTION

EMI shielding is an offshoot of explosive growth of electronics and telecommunication. The EMI among electronic instruments /appliances may lead to degradation of device performance and may even adversely affect human health. Therefore, coherent strategies and suitable counter measures are essential to mitigate the harmful effects of EMI to ensure uninterrupted performance of gadgets(1-6). So polymer based composites have drawn attention due to their efficient properties and potential applications in many fields as aerospace, automotive, power, medicine, construction and electronic areas [7-11]. The inorganic filler loaded polymer composites are widely studied due to their excellent structural, electrical, barrier and good chemical resistance properties[7,8,12-15]. More importantly, carbonaceous filler[e.g. carbon fibres, carbon black, graphite, carbonnanotubes, activated carbon, fullerenes and more recently graphene] based composites are becoming very popular as they display additional properties such as good dielectric and moderate electrical properties, low density, excellent erosion resistance, improved flexibility and superior mechanical properties [7,8,11,16-24]. The above cited properties particularly the dielectric properties, further pushed the range of applications of such composites to encompass such areas as energy generation/storage, environmental pollution monitoring/control, biosensing, electroactive parts, antistatic, electronics/optoelectronics and electromagnetic

interference (EMI) shielding materials [9,11,19,21,23-33]. 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 [34-36].

The aim of our work is to develop/synthesize electrically conducting AC/AR composites by solution casting method. 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, spectral, electrical and morphological attributes using techniques such as XRD, Raman spectroscopy, Dielectric and EMI shielding respectively. These compositions with low density, easy processing, excellent corrosion resistance, good electrical properties and porous morphology are expected to guard the electronic articles from static charges or EMI effects.

II. EXPERIMENTAL

All chemicals used in the work were analytical grade (AR). Acrylic resin (50% solid content) was provided by Pidilite industries limited, India. Xylene (Rankem, India) and activated carbon (Merck) were received without further purification.

In the synthesis 14g of AR was dissolved in 20ml of xylene and a varied amount of AC powder was added into it in different wt % of 0%, 1%, 5%, 10%, 20% and 30% of AC. Then each and every solution was magnetically stirred for 1hr. The prepared solutions were put onto a petridish and were dried for 60hr. to form coatings (~ 300 μm thickness).

III. MEASUREMENT AND CHARACTERIZATION

X-ray diffraction patterns in the 2θ range of $10-70^\circ$ were obtained using Bruker-D8 advanced diffract meter using $\text{CuK}\alpha$ line ($\lambda = 1.540598\text{\AA}$) radiation source. The surface morphologies were recorded from scanning electron microscope (SEM, Leo-440, UK). Lab RAM HR 800 (Horiba JY) micro Raman spectrometer at 632.8 nm laser wavelength was used for recording Raman spectra of these samples in $3500 - 400\text{ cm}^{-1}$ region. The laser beam was focused on the sample using confocals microscope with LWD x 50 objective. Shielding effectiveness (SE) and electromagnetic attributes (complex permittivity and permeability) were measured with the help of network analyzer (VNA E8263B Agilent technologies) by keeping the samples in between the sample holder placed between flanges of waveguides and keeping the input power level at -5dB.

IV. RESULTS & DISCUSSION

Pure AR display a broad peak located at $2\theta = 15.95^\circ$ as well a weak peak at $2\theta = 30.92^\circ$. 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 characteristic peak of AC at $2\theta = 43^\circ$. Particularly, the $2\theta = 15.95^\circ$ peak of AR shifts towards lower angle,

which shows the uniform incorporation of AC particles between AR lamellae and confirms the formation of composites.

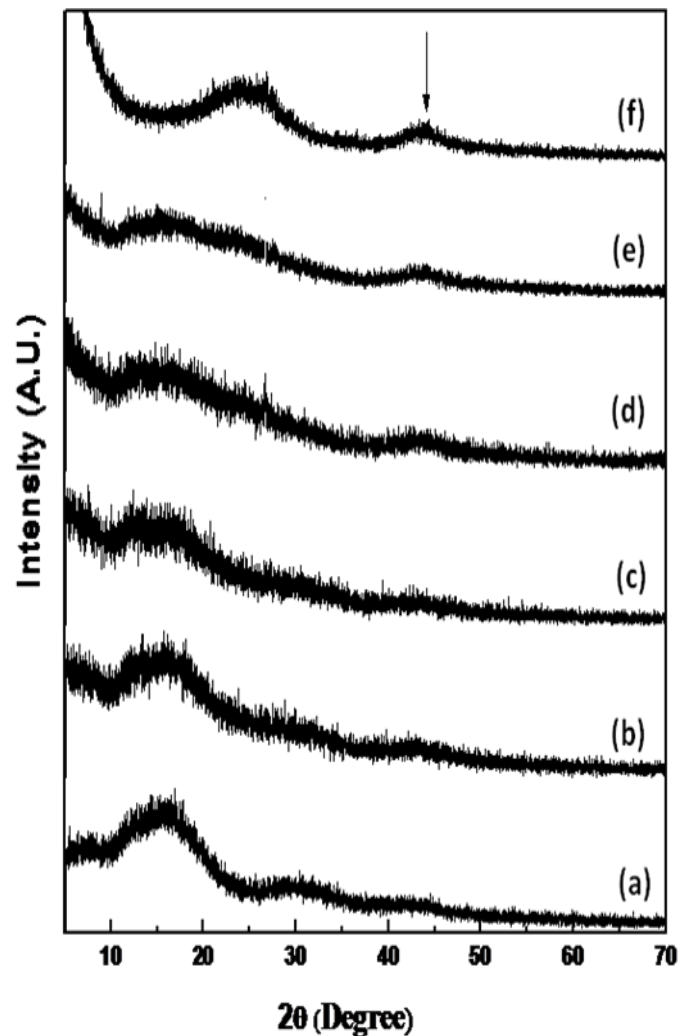


Fig 1. X-ray diffraction patterns of (a) Pure AR, (b) 5 wt%, (c) 10 wt %, (d) 20 wt %, (e) 30% and (f) Pure AC

V. MORPHOLOGICAL DETAILS

Fig. 2 shows the SEM micrographs of pure AR and its AC based composites. It can be seen that pure AR film shows (Fig. 2(a)) smooth morphology with homogenous patterns throughout the scanned region which confirms the semicrystalline nature of pure acrylic.

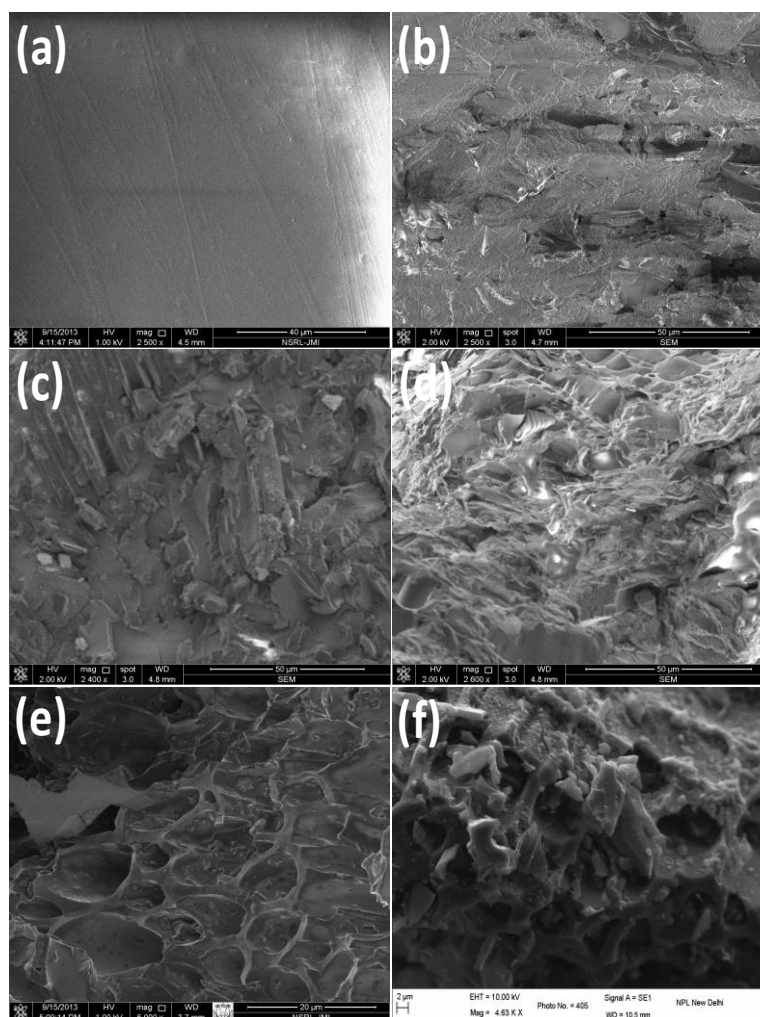


Fig2. SEM images of (a) pure acrylic resin,(b)1 wt%, (c) 5 wt %, (d) 10 wt % (e) 20 wt% and (f) 30 wt% of AC

The incorporation of AC into AR matrix affects the crystallization of matrix and the systematic change in morphology (Fig. 2(b) to 2(f)) 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, and 26]. The further studies to assess the actual EMI shielding effectiveness value and the shielding mechanism are under investigation.

VI.RAMAN SPECTROSCOPY

Raman spectra of carbon and its conjugated analogues give strong bands dominated by sp^2 and sp^3 hybridized carbons as G and D peaks at ~ 1560 and 1360 cm^{-1} respectively. The in-phase vibration of the synthetic acrylic and activated carbon composites G peak as shown in Fig. 3, arises due to the bond stretching of all pairs of sp^2

atoms in both rings and chains at 1582 cm^{-1} which shifts to 1598 cm^{-1} on increasing activated carbon percentage in composites. While the weak disorder band caused by the activated carbon edges D peak is the breathing modes of sp^2 carbon atoms in rings at 1304 cm^{-1} which shifts to 1320 cm^{-1} in 30% activated carbon containing composite [1-3]. The changes in G and D bands on increasing the activated carbon concentration in composites is attributed to the increase in electron-phonon interactions which blue shifts and sharpens the G peak and broadens D band.

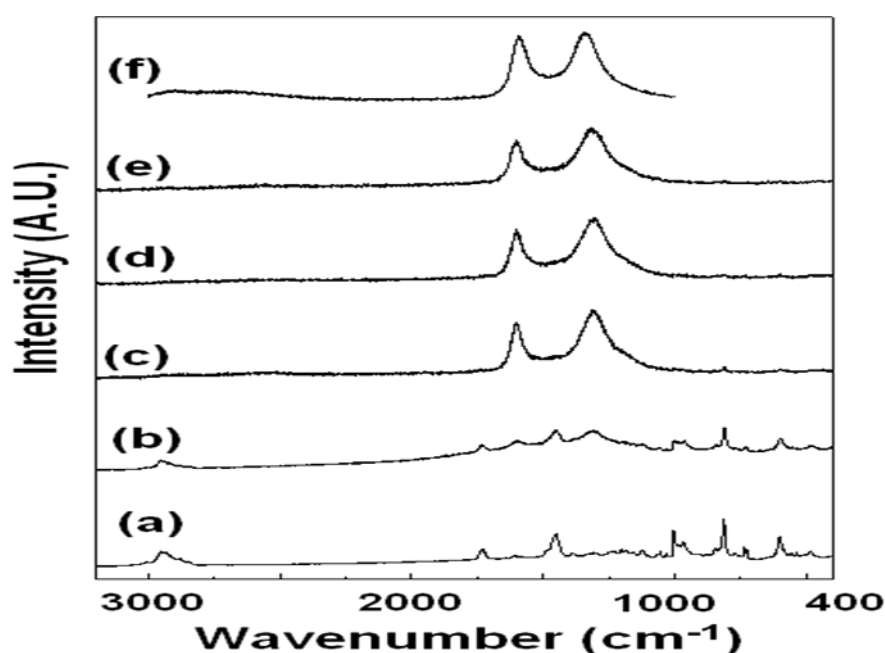


Fig.3. Raman spectra of (a) pure acrylic resin, (b) 1 wt%, (c) 5 wt %, (d) 10 wt %, (e) 20 wt% and 30 wt% of AC

VII.DIELECTRIC MEASUREMENT

The study of dielectric constant as a function of temperature and frequency is one of the most convenient and sensitive methods of investigating the polymer structure of a polymeric films.

The polarization of a dielectric is controlled by the electronic, ionic, and dipolar polarization, Electronic polarization occurs during a very short interval of time (of the order of 10^{-15} sec) and the process of ionic polarization occurs in the range of (10^{-13} - 10^{-12}) sec, while the dipole polarization requires relatively longer time. In the case of polar polymers, the dielectric constant begins to drop after a certain critical frequency. The dipole molecules cannot orient themselves in the lower temperature region. However, as the temperature increases orientation of dipoles is facilitated and this increase the dielectric constant. So in this paper dielectric constant of AR doped activated carbon at room temperatures and frequencies have been carried out to understand the role of different filler concentrations on polymer matrix. AR films filled with AC were prepared in order to investigate the effect of AC on the electrical properties of AR host. The electrical measurements (dielectric constant of

AR/AC composites at different filler contents of dielectric constant were obtained at a frequency range from $(1 \times 10^5 \text{ to } 5 \times 10^5)$ Hz at a room temperature. Upon increasing the filler content an increases the magnitude of dielectric constant.

The variation of dielectric parameters with frequency can be explained in terms of the relaxation time; at low frequencies, the electric dipoles have sufficient time to align with the field before the field changes its direction; consequently the dielectric constant is high. At high frequencies, the dielectric constant value decreases due the shorter time available for dipoles. Electrical parameters dielectric constants were calculated from conductance and capacitance. The dielectric constants of samples were calculated using the following relations:

$$\epsilon_1 = C_s d / \epsilon_0 A$$

where d is thickness, A is the effective cross-sectional area of the sample, ϵ_0 is permittivity of free space, and is the conductivity of the sample that arises from the motion of charge carriers through the polymer.

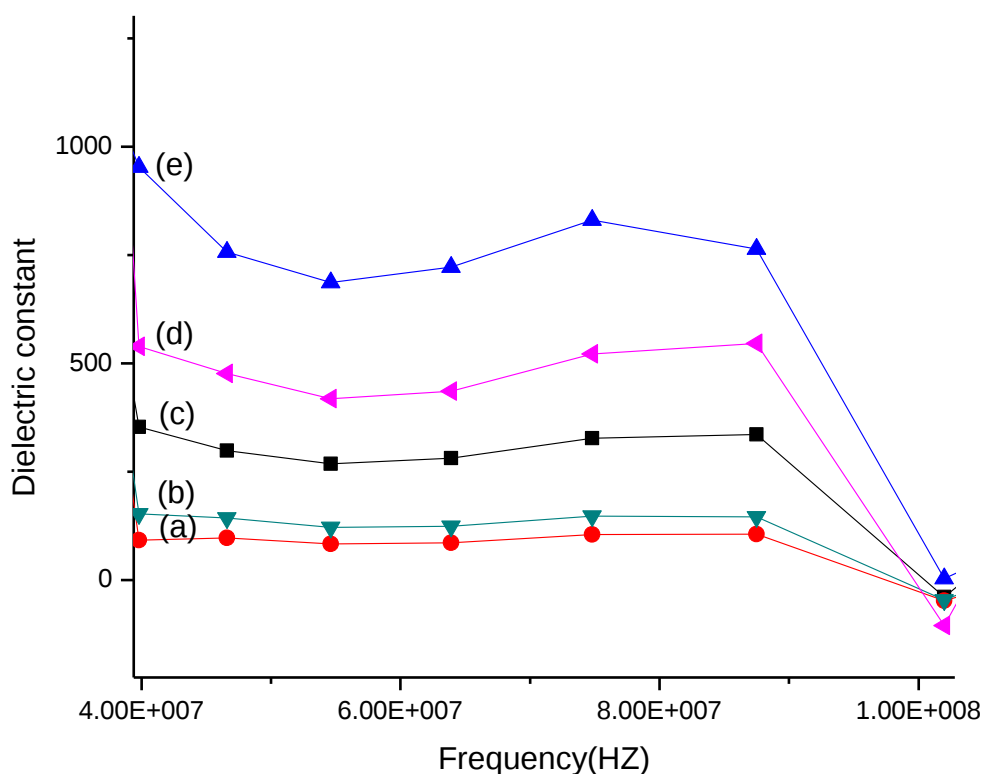


Fig.4.1: Dielectric measurement (a) pure acrylic resin, (b) 1 wt% (c) 5 wt %, (d) 10 wt %, (e) 20 wt% and 30 wt% of AC

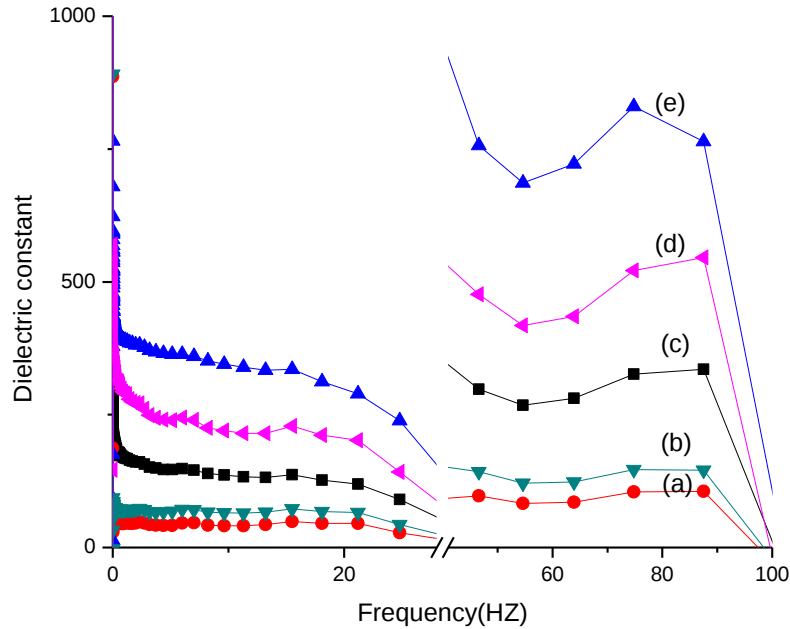


Fig. 4.2: (a) pure acrylic resin and its composites containing (b) 1 wt% (c) 5 wt % and (d) 10 wt % (e) 20 wt% and 30 wt% of AC

VIII. MICROWAVE SHIELDING

The term shielding is defined as attenuation of the propagating electromagnetic (EM) wave produced by the proposed shield and can be expressed as:

$$SE_T \text{ (dB)} = 10 \log_{10} \left(\frac{P_T}{P_I} \right) = 20 \log_{10} \left(\frac{E_T}{E_I} \right) = 20 \log_{10} \left(\frac{H_T}{H_I} \right) \quad (1)$$

Where P_I (E_I or H_I) and P_T (E_T or H_T) are the power (electric or magnetic field intensity) of incident and transmitted EM waves, respectively. As the ratio P_T/P_I (E_T/E_I or H_T/H_I) is always less than unity, therefore, SE_T (total shielding effectiveness) is a negative quantity such that a shift towards a more negative value means an increase in the magnitude of SE_T . In actual practice, three different phenomena named reflection, absorption and multiple reflections contribute towards SE_T , therefore, $SE_T = SE_R + SE_A + SE_M$. Interestingly, when SE_A is greater than -10 dB, SE_M can be safely ignored.

Equations (SE_R) and SE_A can be expressed as:

$SE_T = SE_R + SE_A$ such that:

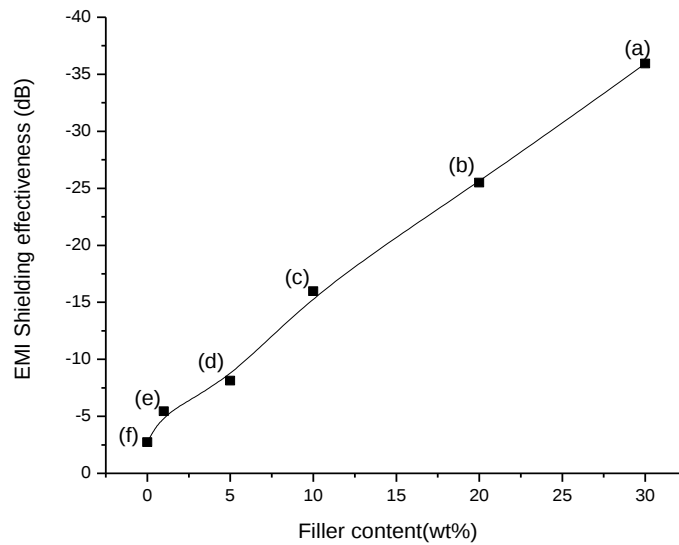
$$SE_R \text{ (dB)} = -10 \log_{10} \left(\frac{\sigma_T}{16 \omega \epsilon_0 \mu_r} \right) \quad (2)$$

$$SE_A \text{ (dB)} = -20 \frac{t}{\delta} \log_{10} e = -8.68 \left(\frac{t}{\delta} \right) = -8.68 t \left(\frac{\sigma_T \omega \mu_r}{2} \right)^{\frac{1}{2}} \quad (3)$$

where the parameters σ_T (*i.e.* $\sigma_{ac} + \sigma_{dc}$), ω , μ_r , δ and t represent total conductivity (*i.e.* ac and dc components), angular frequency, relative permeability, skin depth and shield thickness, respectively. The σ_T and δ can be

further related to imaginary permittivity (ϵ'') and real permeability (μ') as $\sigma_T = \omega \epsilon_0 \epsilon''$ and $\delta = \left(\frac{2}{\sigma_T \omega \mu'} \right)^{\frac{1}{2}}$.

These expressions depict the correlation between theoretical shielding and electromagnetic attributes (permeability and permittivity) and revealed that the combination of electrical, dielectric and magnetic properties are important to achieve desired attenuation levels.



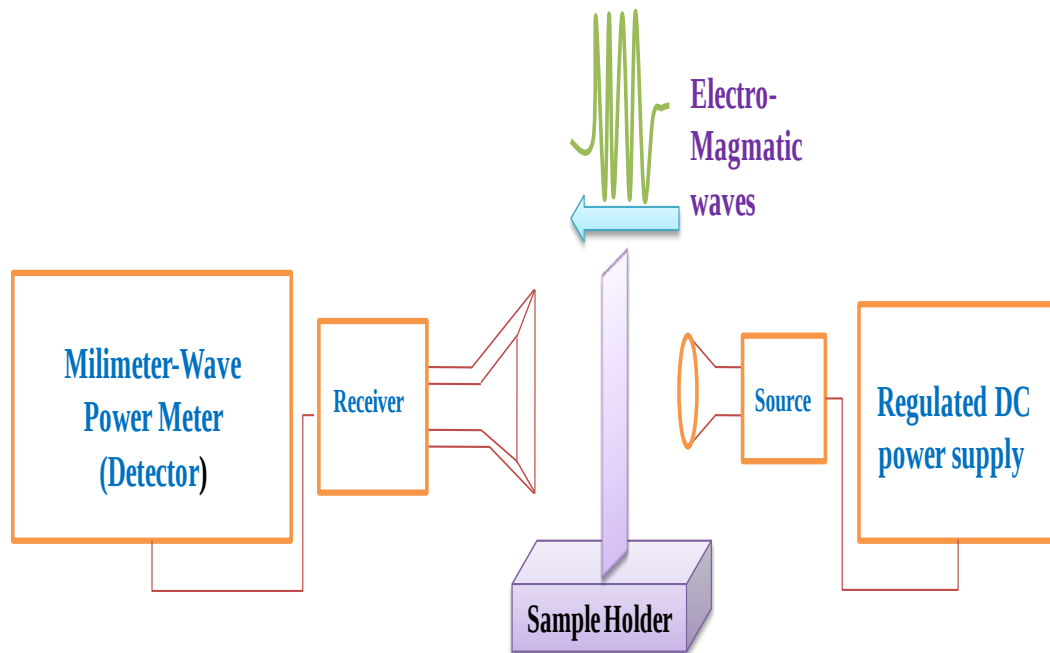


Fig.5.1. (a) pure acrylic resin and its composites containing (b) 1 wt% (c) 5 wt % and (d) 10 wt % (e) 20 wt% and 30 wt% of AC(a) pure acrylic resin and its composites containing (b) 1 wt% (c) 5 wt % and (d) 10 wt % (e) 20 wt% and 30 wt% of AC

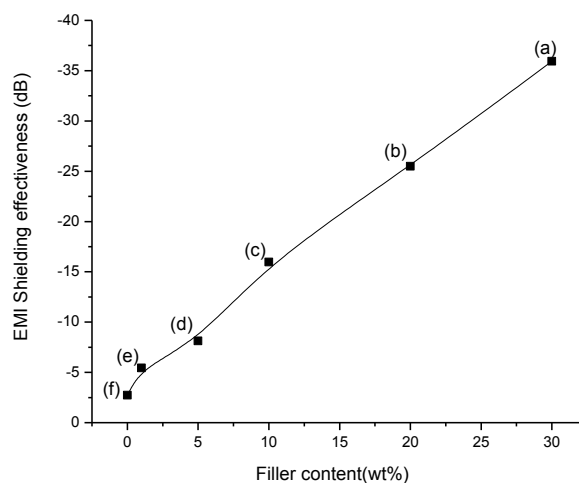


Fig. 5.2 (a) pure acrylic resin and its composites containing (b) 1 wt% (c) 5 wt % and (d) 10 wt % (e) 20 wt% and 30 wt% of AC

IX. CONCLUSION

AC/AR composites have been prepared by dispersing 1 to 30 wt % of AC in AR matrix using a simple solution processing route. XRD patterns give the superimposed peaks of AC and AR which confirm the presence of dispersed AC particles in AR matrix and that were complemented by Raman spectroscopy results. 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 dielectric measurements revealed that pure AR displays low relative permittivity value and increases with the incorporation of AC and becomes maximum at 30 wt.% which displays EMI shielding effectiveness value. This shielding value corresponding to blockage of nearly 99.9% of incident electromagnetic radiation and which suggest that AC/AR composites are promising material for making EMI shields.

REFERENCES

- [1] P Saini & M Arora, Gomes, A.D. (ed.). *New Polymers for Special Applications Intech:Croatia*,(2012) 71 <http://www.intechopen.com/download/pdf/38964>
- [2] Wu J & Chung DDL. *Carbon* 40- 445, 2002.
- [3] Saini P, Choudhary V, Singh BP, Mathur RB & Dhawan SK. *Mater Chem Phys*, 113-919 , 2000 .
- [4] Saini P, Arora M, Gupta G, Gupta BK, Singh VN & Choudhary V. *Nanoscale* 5- 4330 2013.
- [5] Chubb JN.J. *Electrostatics* 65- 607, 2000.
- [6] Saini P, Choudhary V, Sood KN & Dhawan SK. *J Appl Polym Sci*, 113- 3146, 2009.
- [7] Hussain F, Hojjati M, Okamoto M & Gorga RE. *J Composite Mater*, 40-1511, 2006.
- [8] Saini P, Choudhary V, Sood KN & Dhawan SK .*J Appl Polym Sci*, 113-3146, 2009.
- [9] Okada A, Kawasumi M, Usuki A, Kojima Y, Kurauchi T & Kamigaito O. *MRS Symp Proc. Pittsburgh PA: Materials Research Society*; 199045-171.
- [10] Saini P & Arora M, Gomes, A.D. (ed.). *New Polymers for Special Applications Intech: Croatia*, (2012) 71 <http://www.intechopen.com/download/pdf/38964>.
- [11] Godovsky DY. *Adv Polym Sci*, 153-163, 2000.
- [12] Choudhary V, Dhawan SK & Saini P. *Research Signpost/Transworld Research Network*, ISBN: 978-81-308-0499-6, pp. 67, 2012.
- [13] Alexandre M, Dubois P. *Mater Sci Eng: R28* -11, 2000.
- [14] Ray SS & Okamoto M. *Prog Polym Sci* 2-2003, 1539.
- [15] Gao F. *Materials Today*, 7- 50, 2004.
- [16] Yasmin A, Luo J & Daniel IM. *Composites Sci Tech*, 66-1182, 2006.
- [17] Chen GH, Wu DJ & Weng WG. *J Appl Polym Sci*, 82- 2506, 2001.
- [18] Bower C, Rosen R, Jin L, Han J & Zhou O. *Appl Phys Lett*, 74-3317, 1999.
- [19] Cooper CA, Ravich D, Lips D, Mayer J & Wagner HD. *Composites Sci Tech*, 62-1105, 2002.
- [20] Lau KT & Hui D. *Composites Part B*, 33-263, 2002.

- [21] Gojny FH, Wichmann MHG, Kopke U, Fiedler B & Schulte K. *Composites Sci Tech*, 64- 2363, 2004.
- [22] Allaoui A, Bai S, Cheng HM & Bai JB. *Composites Sci Tech*, 61-1993, 2002.
- [23] Stankovich S, Dikin DA, Dommett GHB, Kohlhaas KM, Zimney EJ, Stach EA, Piner .
- [24] Yang Y, Gupta MC, Dudley KL & Lawrence RW. *Nanolett* 5- 2131,2005.
- [25] Y, Du F, He X, Lin X, Gao H, Ma Y, Li F, Chen Y & Eklund PC. *Nano Lett*, 61-1141, 2006.
- [26] Colaneri NF & Shacklette LW. *IEEE Trans. Instrum. Meas.* 41-291,1992.
- [27] Saini P & Choudhary V. *J Nanoparticle Res*, 15 (2013) 1415. Yang Y, Gupta MC, Dudley KL & Lawrence RW. *Nanolett* 2005, 5– 2131.
- [28] Li N, Huang Y, Du F, He X, Lin X, Gao H, Ma Y, Li F, Chen Y & Eklund PC. *Nano Lett*, 6–1141, 2006.
- [29] Saini P, Choudhary V, Singh BP, Mathur RB & Dhawan SK. *SynthMet*161 2011.
- [30] Baldacchini T, LaFratta CN, Fairer RA, Eugene F, Teich MC, Bahaa EA, Michael S, Naughton J & Fourkas JT. *J ApplPhys*, 95- 6072,2004.
- [31] Vallittu PK. *The Journal of Prosthetic Dentistry*, 81-318, 1999.
- [32] 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*. doi: 10.1002/app.1592,, 81-1614,2001.
- [33] Xu JL, Zhong ZC, Huang ZX, Luo JM. *J Alloys and Compounds*, 570-28, 2013.
- [34] Saini P & Choudhary V. *Ind J Pure Appl Phys*, 51-112 (2013).
- [35] Ono H, Ohsawa A & Tabata Y. *J Electrostat*, 57-355 (2003).
- [36] Chubb JN. *J. Electrostatics* 65-607(2007).