



PMME 2016

Nano sized hybrid electromagnetic wave absorbing free standing thin film of IPANI/PVA/Ag-FA [★]

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Abstract

In this study, electromagnetic wave absorbing nano sized hybrid materials were prepared by using nano silver particle with fly ash and interfacial polyaniline as filler and polyvinyl alcohol as binder. Interfacial polyaniline powder (IPANI) was synthesized by in-situ chemical polymerization method. Nano sized hybrid free standing film material were prepared by mixing varying % amount of fly ash (10.20.30.40 and 50% wt) in silver/interfacial polyaniline as nano filler with polyvinyl alcohol as binder matrix and their microwave absorption studies were compared. The microwave absorption studies were performed by using Transmission/Reflection principle via the waveguide method (S_{11}) in the frequency range of 8–12 GHz. The results were compared in terms of electromagnetic wave absorption performance and characteristics. Prepared nano sized hybrid free standing films were characterized by using several physical methods viz., FTIR, XRD and SEM. It was observed that nanosized silver/fly ash/ interfacial polyaniline/PVA free standing films have good electromagnetic absorbing properties. However, the nanosized silver/fly ash (50% wt of FA) IPANI-filled sample with good conductivity showed superior electromagnetic absorbing performance.

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Selection and Peer-review under responsibility of International Conference on Processing of Materials, Minerals and Energy (July 29th – 30th) 2016, Ongole, Andhra Pradesh, India.

Keywords: Electromagnetic wave absorbing free standing film; Nanosized interfacial polyaniline powder (PANI) filler; Nano silver filler; Microwave absorption measurements;

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1. Introduction

The increase of electronics devices in commercial, industrial, healthcare products and defence areas has led to a novel method of pollution as called as electromagnetic interference [1]. This is a most serious concern caused by the interference effects of current induced by electric and magnetic fields, emanating from neighbouring extensive range of electrical circuit [2]. The interference between business machines, process devices, consumer electronic products and other devices may lead to disturbance of usual performance or even complete malfunction. The disturbances across communication channels, automation and process control may lead to loss of valuable time, energy, resources, money or even precious human life. Therefore some kind of electromagnetic interference shielding mechanism must be afforded to guard the alarmed article from spurious electromagnetic noises or pollution. Commonly metals are using to shield the electromagnetic radiation in the electronic devices [3, 4]. But metals are having high density and less susceptibility to corrosion, complex and economically not viable for processing. Further, metals primarily reflect or scatter the radiation and cannot be used in applications where absorption is major requisite, e.g. in stealth technology [5, 6].

In the four decades ago, conducting polymer and their composites have gained the importance of wealth and technological applications [7–15]. The electromagnetic interference shielding and microwave absorption studies of conducting polymers can be explained in terms of electrical conductivity of polyaniline in presence of localized charges (i.e. polarons/bipolarons) this is due to the polarization and relaxation effects [16, 17]. Recently PANI has drags the attention of researchers is due to its non-redox doping, environmental stability and economic feasibility. The properties could be further tuned by controlled polymerization reaction conditions and using different substituted anilines, precise co-monomers, dopants and other fillers [18–22]. Polyaniline has low inherent specific strength and it requires dispersion to increase the strength of polyaniline by using some binding matrix to form proper composites for any commercially useful material. Nevertheless, higher the percolation threshold in polyaniline is due to low compatibilities, phase separated morphology and low aspect ratio of the conducting polyaniline polymer particles. So high concentration of conducting polymers is required in matrix for adequate electrical properties which often affect the mechanical properties of obtained conducting polymer composites.

In view of the above facts, we report the potential of IPANI coated with silver and fly ash nano particles in poly vinyl alcohol matrix to obtain free standing conducting film for possible microwave absorber. The nano particles of silver and fly ash have incorporated in IPANI/PVA matrix may also acts as functional handle providing good dispersibility and processability of the host matrix.

Experimental details

1.1. Materials

Aniline (Loba Chemie, India) was freshly distilled before use. Silver nano particles have been incorporated in the process and fly ash is sintered at 250-300⁰C. Nitric acid (HNO₃, Merck, India) Silver nitrate (AgNO₃, Merck, India), chloroform (CHCl₃, Merck, India) and ammonium persulfate (APS, Merck, India) were used as received. Aqueous solutions were prepared from the double distilled water having specific resistivity of 10⁶ Ohm-cm.

1.2. Synthesis of Silver-IPANI/FA nanocomposites

IPANI-Silver blends are prepared with the concentration of 0.1 mole of silver nitrate, 0.5 g of aniline monomer is mixed with 1 mole of nitric acid then it is added to the organic phase which contains 10 ml of chloroform. 0.1 mole (NH₄)₂S₂O₈ is mixed with 1.0 mole of nitric acid and gradually added to above mixture of organic and aqueous phase. After five minutes, dark green precipitate is formed gradually at the interface and diffuses into the aqueous phase. After 24- hours, the complete aqueous phase is uniformly distributed with the dark-green precipitate and organic layer observed showing orange colour because aniline oligomers are formed. The aqueous phase having the precipitate is collected and cleaned with (CH₃)₂CO and H₂O for the removal of unreacted aniline monomers. The residue of interfacial polyaniline and silver nanoparticles in the product are obtained and purified. It is dried using vacuum oven at about 40.8 °C for 36 hours. In this study, water-soluble polyvinyl alcohol (PVA) is selected to mix with the electrically conducting interfacial polyaniline-Fly ash (IPANI/Ag-Fly ash) composite to form a free-

standing conducting film and investigate the electromagnetic shielding interference.

1.3. Measurements

The infrared spectra of free standing films were taken at resolution of 4.0cm^{-1} in $4000\text{--}250\text{ cm}^{-1}$ range, with the help of model 783 of Perkin Elmer FTIR spectrophotometer and diamond ATR accessory. The XRD spectra of free standing conducting films composites were taken in $10\text{--}90^\circ$ range at a scan rate of $0.1\text{ degree min}^{-1}$ by using D8 Advance Bruker AXS X-ray diffractometer. Morphology of free standing films were observed using SEM (Leo 440, UK) and TEM (Phillips, CM-12). The EMI shielding measurements were taken on pressed rectangular pellets (2mm thick) placed inside the homemade sample holder. The holder matches the internal dimensions of X-band ($8\text{--}12.0\text{ GHz}$) waveguide placed between the two ports of Vector Network Analyser (VNA E8263B Agilent Technologies). Preliminary studies were measured by using Power Meter (Tektronix Make) model number PSM3S10 and ZVL-KI spectrum analysis function or spectrum analyser (Tektronix Make) model number RSA306 real time spectrum analyser.

2. Results and discussions

2.1. Mechanism of free standing conducting polymer composite formation

Based on the fundamental concepts of in situ polymerization of interfacial polyaniline and it is confirmed by SEM/TEM analysis of our samples, we have proposed the probable formation mechanism of the IPANI/PVA/Ag-FA free standing conducting polymer film composites depicted schematically in Fig. 1. The ammonium persulphate acts as an oxidizing agent and aniline being electron donor develops a kind of weak charge transfer complex and simultaneous reduction takes place with silver ion is reduced to silver nanoparticle [18]. The formation of intermediate complex is enabled by the lone pair of electrons on the amine N atom of aniline monomer. HNO_3 acts as dopant and it also helps to form complexes with aniline enabling its solubilization and dispersion. Hence, the reaction system is heterogeneous because of aqueous and organic phase in presence of required amount aniline on the surface of fly ash/Ag nano particle and the remaining aniline monomer in the solution phase. On addition of ammonium persulphate, polymerization reaction proceeds both in solution (in-situ polymerization) and on the surface of fly ash/Ag nano particles. Similarly other larger surface area nano phase materials of Ag and FA also act as catalyst during polymerization. This helps in increase in the formation of cation radicals on the surface of these nano particle which brings about the surface polymerization. After end of in-situ polymerization reaction filtered and washed with acetone, the wet product of silver doped interfacial polyaniline is stirred with 5% of polyvinyl alcohol, pour on to the glass slides uniformly and kept it on the anhydrous CaCl_2 in desiccator about 48 hours to obtain free standing film as depicted in fig.1.

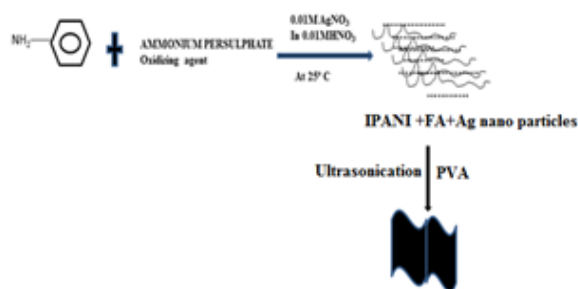


Fig.1. The schematic diagram of the formation of IPANI/PVA/Ag-FA nanocomposites

2.2. IR spectra

Fig.2. reveals the IR spectra of a) IPANI/PVA/Ag b) IPANI/PVA/Ag-FA (50%) nanocomposites. The band around 825 cm^{-1} is because of out of plane C–H bending mode vibrations. The absorption bands at 1571 and 1496 cm^{-1} are characteristic stretching frequency of nitrogen quinoid (N= Q= N) and benzenoid (N–B–N). This kind of variations are due to the conducting path is formed on the polymer chain. The 1296 and 1244 cm^{-1} stretching frequencies are assigned to the bending mode of N–H vibrations and asymmetric C–N characteristic stretching frequency of polaron structure of IPANI/PVA, respectively. The prominent absorption band is observed at 1133 cm^{-1} (C–N stretching) is because of the charge delocalization over the polymeric chain. As increase in the concentration of fly ash content in the IPANI/PVA/Ag matrix, a small shift was observed in the FTIR spectra, the main important characteristic peaks of doped IPANI, which reveals the interaction of fly ash with IPANI/PVA/Ag matrix. The increase in the relative intensity of bands at 1496 and 1294 cm^{-1} may be due to the IPANI/PVA/Ag gets deposited to the fly ash nano particles. The 1117 cm^{-1} peak of IPANI becomes broader and it is shifted to 1134 cm^{-1} in IPANI/PVA/Ag-FA (50%). This will be attributed to the charge transfer interactions with the fly ash particles and quinoid moiety of IPANI/PVA/Ag.

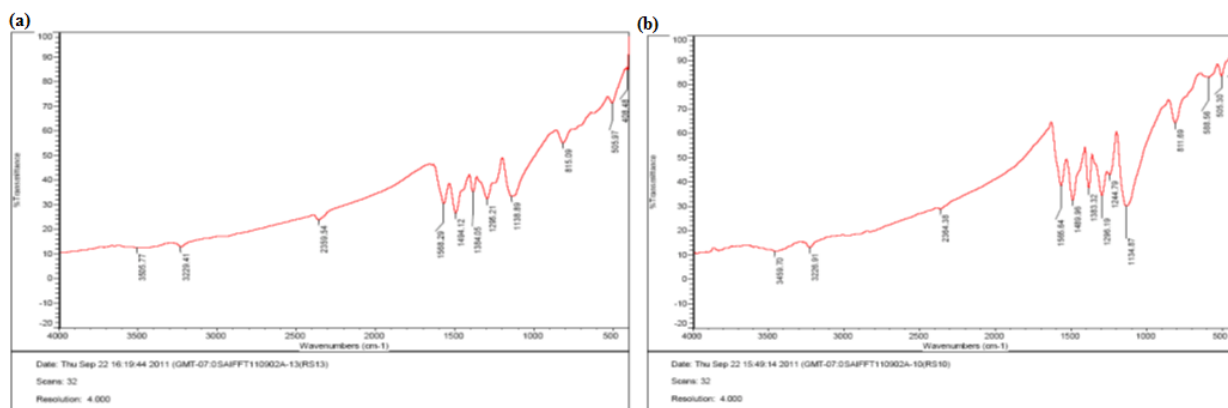


Fig.2. a) IPANI/PVA/Ag; b) IPANI/PVA/Ag-FA (50%) nanocomposites

2.3. XRD studies

Fig. 3 shows the XRD patterns of (a) pure polyaniline (b) IPANI/PVA (c) FA (d) Bulk IPANI/Ag (e) IPANI/PVA/Ag and (f) IPANI/PVA/Ag-FA free standing film of conducting polymer nano composites. Fig. 3(a) the X-ray diffraction pattern of polyaniline indicates the semi-crystalline behaviour with 10 - 30% crystallinity with characteristic broad peak is observed at the diffracting angle, $2\theta \sim 27^\circ$. In the Fig.3(b) broad peak is observed at 27° due to IPANI/ PVA matrix increases the amorphous in the free standing conducting film it represents IPANI is distributed homogeneously. The sharp peaks of the X-ray diffraction pattern show that the prepared nano composite are crystalline and ensures the formation of single crystalline silver nano-particles in polyaniline/silver. According to Bragg's law, reflections at diffracting angle are 38.2° , 44.3° , 64.5° , and 77.4° for 111, 200, 220 and 311 corresponding lattice planes, for fcc structure of Ag nano-particles incorporated in polyaniline. The presence of crystallinity spread throughout, which is due to homogeneous distribution of silver nanoparticles observed in Fig.3 (d). The crystalline component of the diffractogram consists of one of the prominent peaks corresponding to $2\theta = 38.2^\circ$, corresponds to 111 lattice planes, for the fcc structure of Ag nanoparticles incorporated in interfacial polyaniline/PVA. Because of semi crystallinity, the resulting sample shows higher values of conductivity when compared to other IPANI/PVA systems as depicted in Fig. 3 (e). Furthermore, the diffraction pattern of the bare PANI reveals the amorphous nature and the peaks of PANI can hardly be observed in the PANI/PVA and PANI/PVA/Ag/FA-50% composite blends. According to the previous reports [23-24], the d -spacing is the important characteristic space between the planes of C_6H_6 rings in vicinal planes. It is the inter chain space or the close contact space between two vicinal chains. Again, it is just a general observation that the arrangement of polymer chain and

inter chain space are changed through the shape and size of the in between dopants resulting in a change in electron delocalisation length and conductivity on higher d -space. The spectra show the presence of broad peak at 2θ value 15° , 25° and sharp intense peak observed at 38.2° indicates the presence of fly ash particles and silver nanoparticles in the free standing conducting film of PANI/PVA matrix. Hence there is no structural change in fly ash and silver nanoparticles due to its dispersion in polymerization reaction mixture.

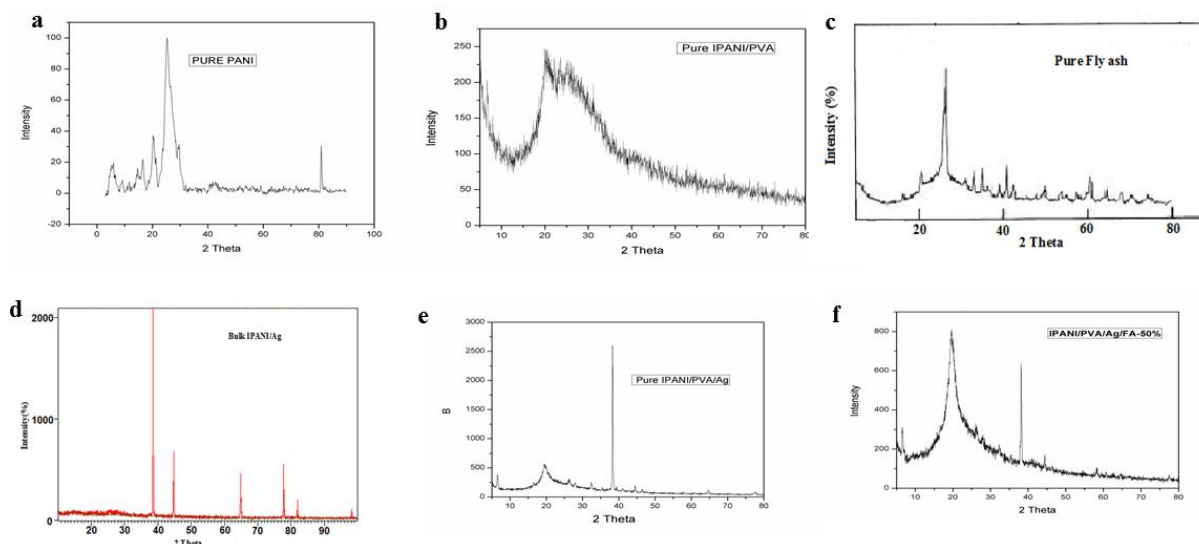


Fig.3. (a) XRD spectra of pure PANI; (b) XRD pattern of IPANI / PVA; (c) XRD spectra of pure Fly ash; (d) XRD spectra of Bulk IPANI/Ag; (e) XRD spectra of IPANI/PVA/Ag; (f) XRD spectra of IPANI/PVA/Ag-FA (50%) composites.

2.4. Morphological details

Fig. 4 shows the SEM photographs of (a) pure PANI (b) IPANI/PVA (c) FA (d) IPANI/PVA/Ag-FA free standing film of conducting polymer nano composites. These micrographs show that pure PANI (Fig. 4a) exhibits highly agglomerated globular structures whereas as Fig. 4(b) represents the nano sized IPANI/PVA free standing conducting film and by using SEM micrograph, we observe that in some places, there are spherical particles which are agglomerated homogeneously distributed having diameter of less than 10nm. Some of the authors have reported that IPANI/PVA obtained from various synthetic methods possesses entirely irregular morphological structure with high heterogeneity in chain orientations. A uniform structure of morphology and chemical homogeneity seen in the very present case gives an edge for the interfacial polymerization over other synthetic methods. A very high magnification of SEM micrograph indicates the existence of fly ash cenosphere particles, which are homogeneously distributed throughout the free standing conducting polymer film samples. From the SEM micrograph, it is clearly seen that the fly ash cenosphere in the sample used for the preparation of the composites conducting free standing film has geometry of spherical or bubble like structure shown in (Fig.4c). IPANI/PVA/Ag-FA (Fig.4d) shows the micrograph of the particles. nano composite is highly temperature sensitive due to the interaction of electrons and the conducting polymer sample. SEM images were obtained from much diluted solution of the nano composite particle. The white spots are indicated in SEM images the presence of silver nano rods in the free standing conducting film composite. From SEM micrograph, it is clearly seen that the cenosphere silver nanoparticles are distributed homogeneously, and the average particle size from the SEM is less than 50 nm. While recording the SEM images, it has been seen that the amount of porosity induced in the composite samples rises with an increase in the concentration of fly ash in the IPANI/PVA/Ag free standing thin film. They are marked on the SEM micrograph, which is also confirmed by XRD studies.

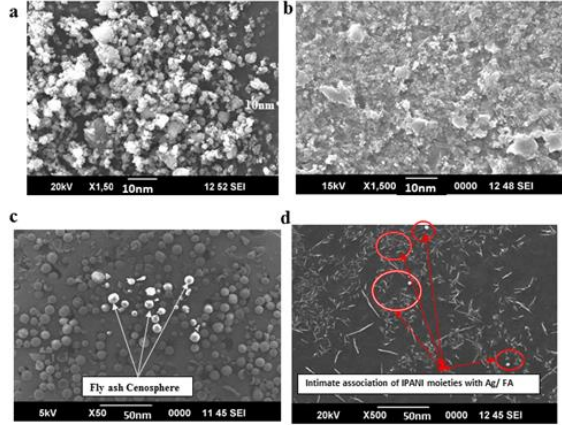


Fig.4. (a) SEM photograph of Bulk PANI; Fig (b) SEM photograph of IPANI/PVA;
Fig (c) SEM photograph of FA; Fig (d) SEM photograph of IPANI/PVA/Ag-FA (50%)

2.5. Shielding effectiveness

EMI shielding is defined as the attenuation of the propagating electromagnetic waves produced by the shielding material. The shielding is a direct consequence of reflection, absorption and multiple internal reflection losses at the existing interfaces, suffered by incident electromagnetic (EM) waves. EMISE can be expressed as [25-28]:

$$EMISE_T = 10 \log \frac{P_I}{P_T} = 20 \log \left| \frac{E_I}{E_T} \right| = 20 \log \left| \frac{H_I}{H_T} \right| (dB) \quad (1)$$

Where P_I (E_I) and P_T (E_T) are the power (electric field) of incident and the transmitted electromagnetic radiations, respectively. For a mono layer of shielding material, the total electromagnetic interference shielding effectiveness (EMI SE_T) calculated from Eq. (1) is described as the sum of the contribution due to reflection (SE_R), absorption (SE_A), and multiple reflections (SE_M) as the following [29,30]:

$$SE_T = SE_R + SE_A + SE_M (dB) \quad (2)$$

$$SE_R = 20 \log \left| \frac{1+n^2}{4n} \right| (dB) \quad (3)$$

$$SE_A = 20 \text{Im}(k)d \log e (dB) \quad (4)$$

$$SE_M = 20 \log \left| \frac{1-(1+n^2) \exp(2ikd)}{1+n^2} \right| (dB) \quad (5)$$

Here, n is the refractive index of shielding material and $\text{Im}(k)$ is the imaginary part of wave vector in the shielding material. The S_{11} (or S_{22}) and S_{12} (or S_{21}) are the scattering parameters (S -parameters) of the two-port vector network analyser (VNA) system. They represent the reflection and transmission coefficients, respectively. The transmittance (T), reflectance (R), and absorbance (A) through the shielding material can be described as below:

$$T = \left| \frac{E_T}{E_I} \right|^2 = |S_{12}|^2 (= |S_{21}|^2) \quad (6)$$

$$R = \left| \frac{E_R}{E_I} \right|^2 = |S_{11}|^2 (= |S_{22}|^2) \quad (7)$$

$$A = 1 - R - T \quad (8)$$

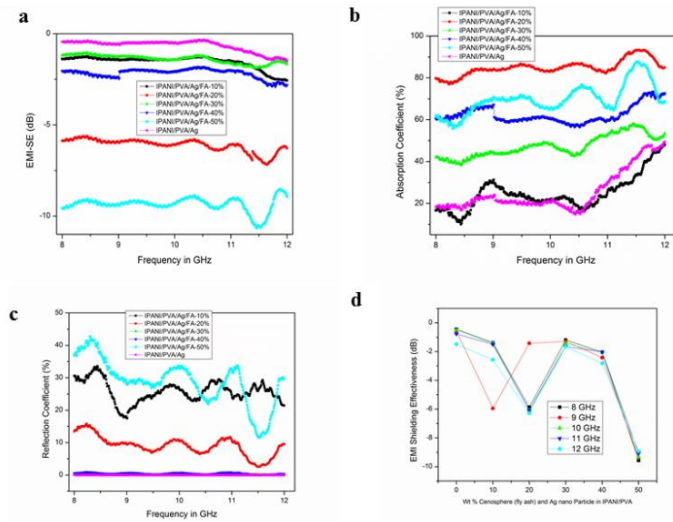


Fig.5.(a) IPANI/PVA/Ag –fly ash EMI SE (dB) X-Band; (b) Absorption coefficient of IPANI/PVA/Ag–Fly ash X-Band; (c) Reflection coefficient of IPANI/PVA/Ag –Fly ash X-Band; (d) EMISE of wt % of Fly ash and Ag particle in IPANI/PVA at X band

Fig.4. (a) shows the EMI-SE versus frequency for different wt % interfacial polyaniline/PVA/Ag – fly ash composites. We observed that the 50 wt% composite shows excellent EMI shielding effectiveness. Particularly a stabilized EMI SE is observed for the lower weight percentages of fly ash cenosphere and silver nanoparticles (10, 20, 30, 40 and 50 wt %) throughout the X-band frequency range. Except for a certain range of frequencies, EMI-SE of composites is independent of frequency. It is also reported that EMI-SE of a composite depends not only on conductivity, but also on permittivity of the composite. Thus the composition of the sample, nature of the dispersed phase, its particle size and shape etc are important factors and can affect the EM radiation absorption and EMI-SE. The increased conductivity of the composite resulted in such an efficient EMI SE values and it may be a result of the long range charge transport and increased number of relaxation modes. Altogether the composites show very effective shielding in the entire X-band. The absorption dominated shielding effectiveness (SE) confirms the relevant application of the interfacial polyaniline/PVA/Ag-fly ash composite for shielding purposes in the X-band frequencies.

Fig.4.(b) reveals that the IPANI/PVA/Ag-Fly ash composites with the fly ash cenosphere, nanoparticles in the conducting free standing polymer matrix 87% (attenuation, –9.0 dB) of the energy of the incident radiation at 8 GHz and 65% (attenuation, –4.5 dB) at 12 GHz. The absorption resonance frequency is positioned outside or at the lower limit of the X-band. At around 9.5 GHz (frequency commonly used by tracking radars), the absorption of energy of the incident radiation was about 80% (attenuation, –7.0 dB) and at 8.0 GHz (the lower limit of the X-band) the absorption was 87% (attenuation, –9.0 dB). There are two types of absorbing materials: resonant or narrowband, and wideband absorbers. Resonant absorbers are usually made of a single layer of absorbent material and are more common, while different materials in layered arrangements are combined to make wideband absorbers. The resonance peak of most absorbent materials is around 9.5 GHz. The frequency of the resonance peak can be changed by changing the arrangement of layers in a multi-layer absorbent material enabling it to absorb over a wide frequency band.

The variation of reflection coefficient of the IPANI/PVA/Ag-Fly ash composites for different concentrations of fly cenosphere nanoparticles at X-band frequencies are given in Fig.4. (c). the behaviour of reflection coefficient of microwaves from these samples is observed to be dependent on frequency. With an increase in the concentration of these nanoparticles, the microwave reflection coefficient also increases but the nature of the curve remains same. With an increase in concentrations of fly ash cenosphere particles, there is an increase in the reflection coefficient. This nature of the peak is maintained up to a 50 wt % amount of fly ash cenosphere nanoparticles. The identical condition could be explained by the nullification of intensity of incident and reflected

microwaves at the absorber's surface.

Fig.4. (d) depicts the deviation of Shielding effectiveness values v/s weight percentage of fly ash in IPANI/PVA/Ag at fixed frequencies viz., at 8, 9, 10, 11 and 12 GHz. From the graph it has come to notice that the values of EMI shielding effectiveness of all conducting polymer composite samples increases till the 50 wt % fly ash in IPANI/PVA/Ag conducting free standing film composites increases because of fly ash being present and silver nanoparticles acts as tiny antennas to shield the radiation.

Conclusions:

We prepared nano sized hybrid free standing film material were prepared by mixing varying % amount of fly ash (10.20.30,40 and 50% wt) in silver/interfacial polyaniline as nano filler with polyvinyl alcohol as binder matrix and their microwave absorption studies were compared. Prepared nano sized hybrid free standing films were characterized by using several physical methods viz., FTIR, XRD and SEM. These techniques reveals the homogeneous distribution of silver and fly ash nano particles this is confirmed from EMISE measurements. It was observed that nanosized silver/fly ash/ interfacial polyaniline/PVA free standing films have good electromagnetic absorbing properties. However, the nanosized silver/fly ash (50% wt of FA) IPANI-filled sample with good conductivity attributed excellent electromagnetic absorbing performance.

Acknowledgements

The authors are acknowledge to Vision Group on Science and Technology, Government of Karnataka, Bangalore, for providing financial assistance in the form of a research and establishment of infrastructure (No.:VGST/K-FIST(L₁)/GRD-363/2014-2015 dated 02 Jan. 2015). We are also thankful to SAIF, Cochin University, Cochin, for XRD, IR spectral data and SEM images.

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