DIELECTRIC PERMITTIVITY, EMW FILTERING AND MECHANICAL STRENGTH BEHAVIOUR OF Cu-PARTICLE/MICROWIRE-MESH REINFORCED UNSATURATED POLYESTER COMPOSITE IN 2-18GHz MICROWAVE REGION

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Electromagnetic wave filtering materials with conductive nano copper particles and micro-wire mesh was fabricated and analyzed for its performance. The principal aim of this research work is to evolve the importance of high purity copper nano particles into copper wire-mesh reinforced unsaturated polyester resin composite in electromagnetic wave filtering behaviour. Copper particles of 70 nm where prepared via electrolysis process. The prepared nano copper particles were surface activated by 3-Aminopropyletriethoxysilane (APTES). The copper micro wire-mesh is also treated by strong acid for more porous surfaceto ensure better adsorption. The unsaturated polyester copper/copper wire-mesh hybrid composites were prepared using hand layup process. The dielectric results show that additions of ultra fine copper particles into copper wire-mesh reinforced unsaturated polyester composite give maximum dielectric constant of 12. The wave attenuation coefficient and noise level of particle and wire-mesh reinforced unsaturated polyester composite gives 0.32 and -38 dB at frequency 16 GHz for composite contain copper wire-mesh reinforced along with 15 wt.% of treated copper particles.The tensile and flexural results of 85 MPa and 115 MPa were observed for composite, which contain 50 wt.% of micro-wire mesh and 10 wt.% of copper nano particles.

(Received October 21, 2018; Accepted February 18, 2019)

Keywords: Polymer composite, Cu-particle/wire-mesh, APTES, EM filtering, TEM

1. Introduction

Electromagnetic wave shielding or filtering needs special attention in electronic industries where electromagnetic interface plays crucial role [1]. These electromagnetic interfaces may reduce the life and performances of electronic devices to the greater extend [2]. Reducing these kinds of interfaces between electronic devices improves their performance to higher significant level. For preventing electromagnetic penetration conductive polymeric material could be used as a shielding element, where lot of electromagnetic radiation could be avoided. Conductive and magnetic particles could be used as attenuator within the polymers where conductive based shielding mechanism is required [3]. Conductive fabrics could be used as shielding elements within unsaturated polyester matrix, which may acts as EM blocker at higher frequencies. Conductive particle could be selected based on their availability, eco-friendly nature, cost and fabrication methodologies [4]. Copper woven conductive fabric could be used as primary matrix, which may reduce around 60% of electromagnetic wave penetration. Similarly additions of conductive copper particles take up charge of remaining 10-15% of attenuation. Overall around

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75% wave attenuation could be achieved [5]. The conductive copper particles could be surface activated using an activator 3-Aminopropyletriethoxylane. The surface modified particles surface could be identified by using FT-IR spectral analysis [6]. The conductive copper particles and fibre fabric reinforced composites could be prepared using hand layup method with recommended parameters [7]. The composite effectiveness could be justified in the form of dielectric, electro-magnetic shielding and mechanical behaviour using ASTM standards. Viswanthan et al [8] confirmed that adding copper and magnetic iron(III) oxide particle into epoxy resin increased the wave attenuation percentage up to 42%. They strongly recommended that surface modification process fetched significant results in overall microwave shielding effectiveness of composite. Around 10% of improvement was seen between as-received and surface modified copper and iron(III) oxide particlesin matrix. Arunprakash VR et al[9] concluded that additions of surface treated MWCNTs into epoxy resin attenuates the incoming microwaves up to 84dB in J band frequency. This larger attenuation was the reason of surface modified MWCNTs, which could attenuate more microwaves by conductive microwave shielding mechanism. In this work the importance of copper micro wire-mesh reinforced with copper particles for better microwave shielding effectiveness. These mechanically sound and electromagnetically shielding improved composites could be used in wave attenuation areas in electronic applications.

2. Experimental procedures

2.1. Materials

Unsaturated polyester resin used for this present study was maleic anhydride type having molecular weight of2400 g/mol, kinematic viscosity of 600 cps and density of 1.13 g/cm^{3}was procured from Huntsman India Ltd. Methyl Ethyl Ketone Peroxide of density 1.17 g/cm^3, molar mass of 210.1 g/mol and Cobalt naphthenate of molar mass 401.2 g/mol and density of 0.95 g/cm^3 werepurchased from Merck India Ltd. Pure, impure copper rod of diameter 10 mm and micro-wire mesh of Diameter 500 micron with cross gap distance of 1.2mm was selected for electrolysis and composite making was procured from Ms. Bandari metallic firm, Chennai, India. The surface modifier3-Aminopropyltriethoxysilane (APTES) was purchasedfrom Sigma Aldrich, USA.

2.2. Preparation of copper nano particles

The copper particle of 70 nm was made through electrolysis process; in which a liquid, Cu_2SO_4 was used as a dielectric medium and pure and impure copper electrodes are kept as cathode and anode. An 8 V DC supply was given to electrodes through a DC adapter for several hours. The adapter’s negative terminal was tied with cathode (pure copper rod) and the positive terminal was connected with anode (brass rod). The time duration of 1 h was used to spend to ensure the deposition of pure copper particle from electrolyte and brass rod. The settled down copper particles were collected from cathode and get washed with ethanol and dried at room temperature [10]. Figure 1 (a &b) shows the TEM images of electrolysis-made copper nano particles. The high resolution transmission electron microscopy used here was JEOL JEM S 2100, JAPAN. The particles showan irregular shape with sharp edges, which is due to the removal of atoms from Brass rod during process. Fig.1 (c) shows selected area X-ray diffraction peaks with circular dots, which confirmvery smaller grain size of Cu particles. Fig. 2 (d) shows HR-TEM image of prepared copper particle with highly textured crystalline nature of uniform atomic arrangements. Fig. 2 shows the EDAX graph of prepared copper particles with high purity nature. Only peaks related to copper is observed as maximum intensity, which confirms the high purity nature of particles.

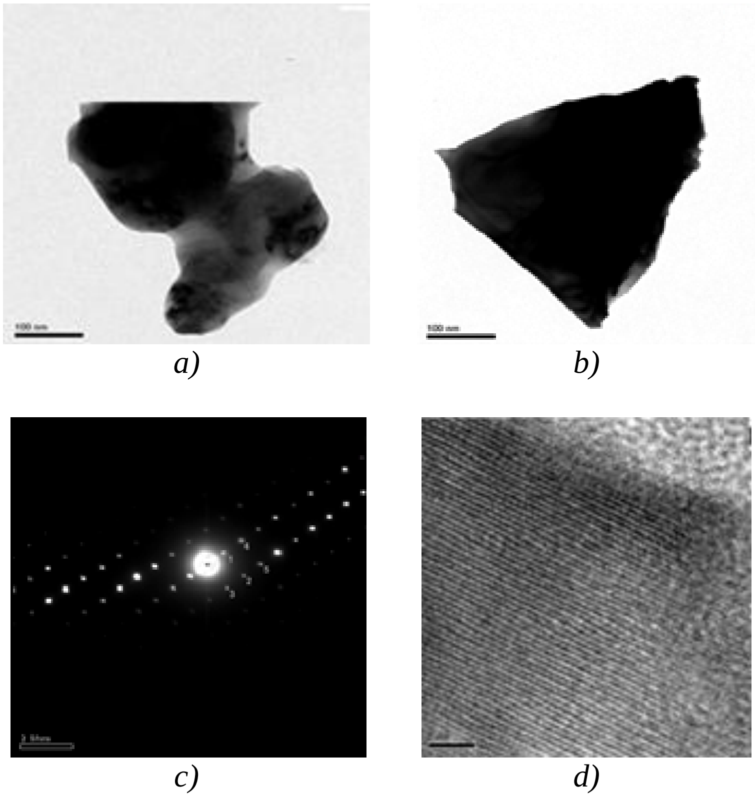


Fig. 1 TEM images of (a & b) fine Cu particles, (c) selected area diffraction and (d) HR-TEM image of Cu confirming high crystalline pattern.

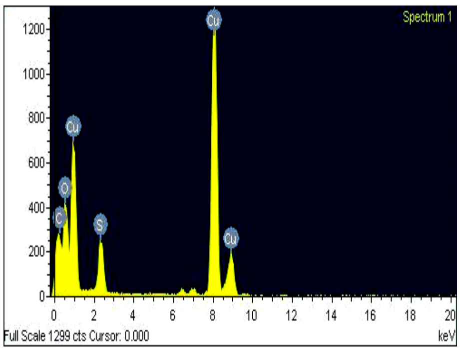
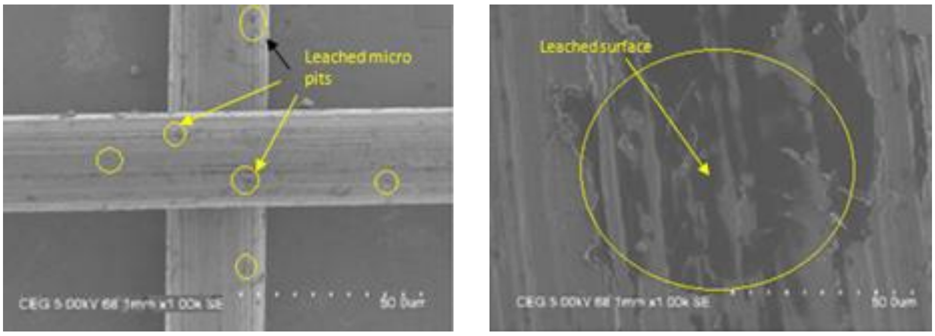


Fig. 2 EDAX report of pure copper particle.

2.3. Surface activation of copper nano particles and wire-mesh

The copper particles were surface treated by in-situ hydrolysis process, where particle's surface was activated using 3-aminopropyletriethoxysilane for better adhesion and dispersion of particles in matrix [11]. Similarly the copper micro wire-mesh was surface activated for more adsorption via acid etching process. In that the copper micro wire-mesh was immersed into high concentration (4N) of H_2SO_4 for 20 min to wash away the surface atoms. Fig.3(a & b) shows the SEM images of surface leached copper wire mesh. The scanning electron microscopy used here was a thermic SEM, HITACHI, S-1500, JAPAN.



a) *b)*

Fig. 3 Bleached surfaces of copper micro wire-mesh after acid etching.

2.4.Preparation of composite

A fixed quantity of unsaturated polyester was mixed with surface-activated copper nano particles with varying volume percentage. The mixed colloidal suspension was then stirred continuously until the degassing process completed. The resulted colloidal suspension was then mixed with 2 wt.% of Methyl ethyl ketone peroxide and 2 wt.% of Cobalt naphthenat and stirring process continued. The colloidal solution at the end of stirring process then poured into a 3mm silicon rubber mould and copper micro wire-mesh get laid one by one for about 50 wt.%. The poured resulted suspension was allowed to cure about 24 h and posy cured at 48 h. The final product was taken out from silicon mould and inspected for visual and dimensional errors. The tested sample specimens were prepared carefully based on ASTM standards.

3. Characterization of composites

The fabricated composites were tested for their effectiveness based on ASTM standards. The mechanical properties were evaluated using a tensile testing machine having load of 20 ton with cross head speed of 4.5 mm/min. The test specimens were hold with jaws using teflon gripper cloth for better tightness. The dielectric behaviour of fabricated composites was analyzed using a dielectric tester HIOKI, JAPAN with operating frequencies various from 50 Hz to 50 MHz. But in the current study the range of frequency was fixed from 50 Hz to 500 Hz. The microwave filtering effect was analyzed using a microwave probe meter setup with VSWR attachment. The attenuated frequency drop was calculated in the form of reflection loss and reflection coefficient.

4. Results and discussion

4.1. Dielectric permittivity

Fig. 4 shows the dielectric permittivity of various composites fabricated. The additions of copper micro wire-mesh and copper particles into polyester resin increased the dielectric permittivity of composite. The additions of continuous long copper wire-mesh into unsaturated polyester resin boosts the charge transfer within the composite under external frequency. The maximum dielectric permittivity of 12.0 was observed for composite contain 50 wt.% of copper wire-mesh and 15 wt% of copper nano particle. This huge improvement is the result of adding charge transfer ability copper material. Both continuous wire like structure and powdered form of copper enhanced better charge conductivity [12]. It is noted that at 100 Hz the composite posses maximum dielectric permittivity. This improvement is because of electronic polarization, which forms a continuous network for charge transfer. It is also observed that when particle volume increases the dielectric permittivity also increases. This is the result of improved and more active copper particle conductivity phenomenon [13]. The improvement in dielectric permittivity of 66%, 72%, 77%, 79% and 81% were observed for composites equipped with 50 wt.% of copper wire mesh and 1, 5, 10 & 15 wt.% of copper nano particles. Thus additions of surface-activated copper particles improved the permittivity of unsaturated polyester resin composites.

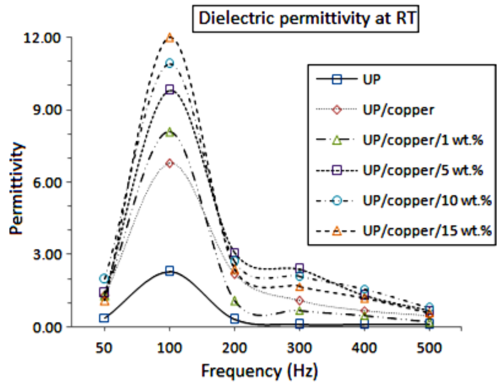


Fig. 4. Dielectric permittivity of copper wire-mesh and copper particle composites.

4.2. Electromagnetic wave attenuation

Fig. 5 shows the electromagnetic wave attenuation behaviour of surface-activated copper wire-mesh and copper nano particles-reinforced unsaturated polyester resin composites. Fig. 5(a) shows the wave attenuation coefficientof various composites fabricated. It is observed that the additions of copper wire-mesh and copper particles increase the wave attenuation percentage thereby giving lower attenuation coefficient. The very lowest attenuation coefficient of 0.32 was observed for composite, which contain 50 wt.% of copper wire-mesh and 15wt.% of copper nano particles. This very low coefficient is the result of multi reflection phenomenon.When microwave passes through the composite material the small dimensioned particles greatly attenuate the incoming microwaves. When more active sites are there the wave attenuation is more due to wave reflection phenomenon [14]. When multi-reflection is actively works on the shielding composite the attenuation also more [15].

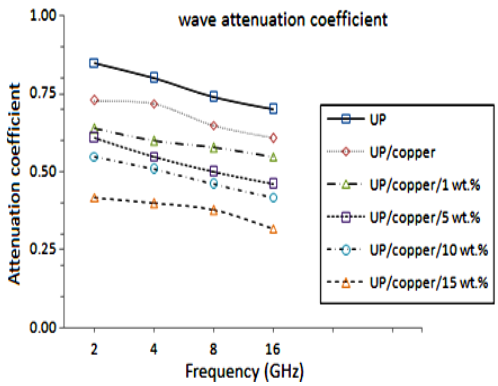


Fig. 5.Wave attenuation coefficient of composites in variable frequencies.

Fig. 6 shows the reflection loss of various composites fabricated. The maximum reflection loss of -38 dB was observed for composite, which contain 50 wt.% of copper wire-mesh and 15 wt.% of copper nano particles. This high reflection loss is the result of highly conductive copper wire-mesh and particles, which absorbs most of the incoming microwave. When the incoming microwave hits the shielding material the wave filtration starts in the form of reflection loss. It is observed that the increase of frequency improved the reflection loss within composite material. This phenomenon is the result of more amplitude of incoming microwave, which will improves the wave reflection within skin depth [16 & 17]. Similar improvements were observed in all composite designations.

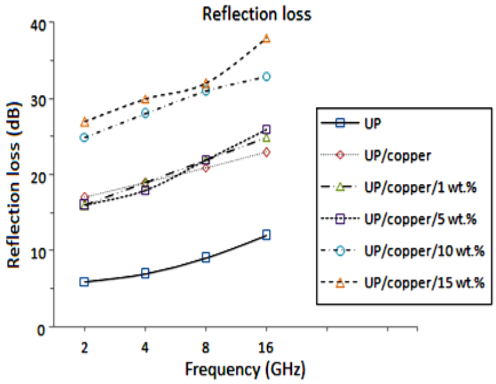


Fig. 6. Reflection loss of composites fabricated.

4.3. Mechanical properties of composites

Fig. 7 shows the tensile and flexural behaviour of copper composites. It is noted that additions of surface treated copper wire-mesh and copper nano particles improved the tensile and flexural strength of composite. The maximum tensile strength of 85 MPa was observed for composite, which contain 50 wt.% of micro-wire mesh and 10 wt.% of copper particles. Similarly the flexural strength of same designated composite is 115 MPa. This improvement is the results of improved load carrying capacitors on unsaturated polyester resin matrix. This micro-wire meshes acts as continuous load carriers in the matrix [18]. Similar way the copper particles act as crack suppressor while tensile and bending load was applied. The interfacial cracking was not initiated under external loading, which shows improved adhesion between matrix and wire-mesh. This improvement is because of surface activation on mesh surface [19]. It is observed that the composite, which contain 15 wt.% of copper nano particles decreased the mechanical properties. This is the result of agglomeration of very fine particles, which affects the uniform dispersion thereby lower tensile and flexural strengths were observed [20]. Figure 8 shows the SEM images of copper wire-mesh reinforced UP composites. The surface activated copper wire-mesh had very good adhesion with matrix, thus improved the load sharing phenomenon. There is no wire pull out is observed, and the colour of wire-mesh is like as colour of matrix. This indicates strong adhesion between reinforcement and matrix [21].

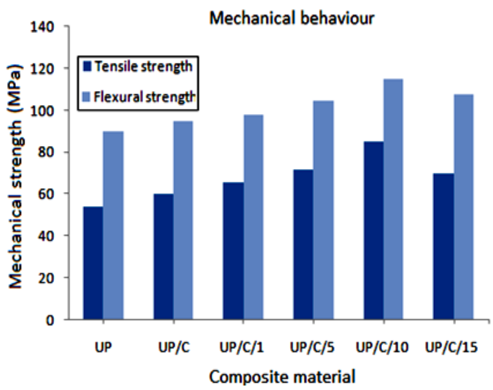
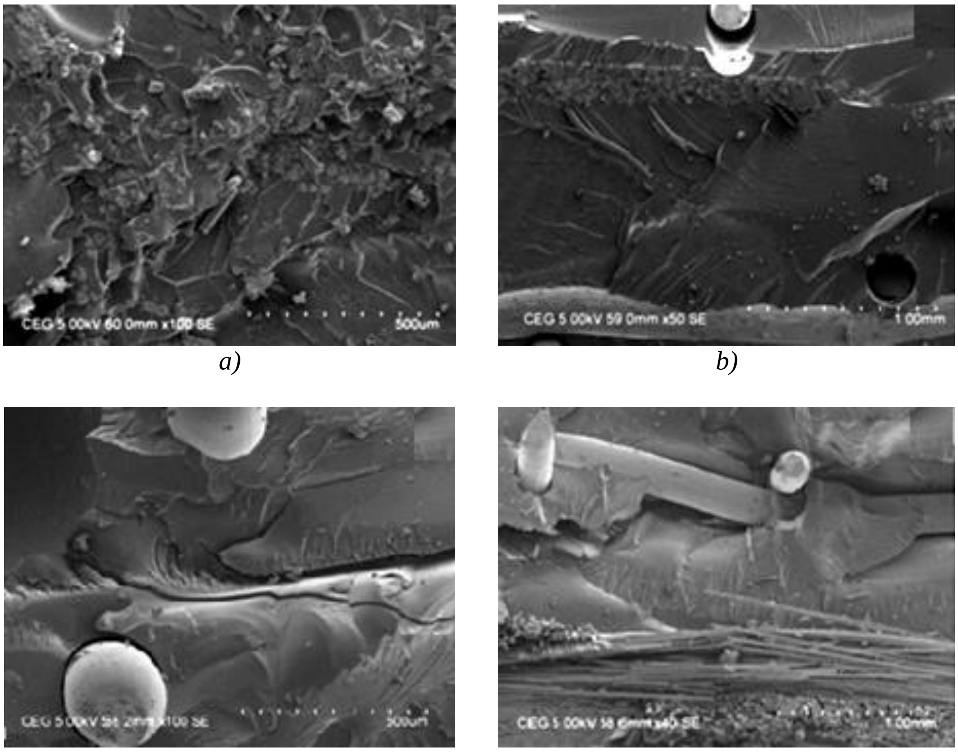


Fig. 7. Mechanical behaviour of composites.



a) b) c) d)

Fig. 8. SEM images of copper wire-mesh reinforced UP composites.

5. Conclusions

The copper wire-mesh reinforced copper nano particles dispersed unsaturated polyester resin composites were fabricated and analyzed. The additions of surface activated copper wire-mesh and copper nano particles improved the mechanical properties along with various novel properties. The additions of nano particles fetch more redefined dielectric behaviour under variable frequencies. The dielectric permittivity of composite greatly improved. Similarlythe microwave attenuation also increased via multi wave reflection phenomenon with in shielding material by the additions of copper nano particles.

The reflection loss is improved as particle volume increases. Same way the wave attenuation coefficient also increases with particles content. The tensile and flexural properties shows forward trend in their properties up to 10 wt.% of particle addition beyond this the strength got decreases. The SEM images show improved adhesion of reinforcements in UP matrix. Thus addition of copper wire mesh improved its mechanical, dielectric and micro wave filtering behaviour, whereas further addition of nano copper particles greatly improved the behaviour in all aspects of the composites. Thus nano particle additions along with micro-wire mesh would be worthy work in the field of electromagnetic wave filtering.

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