



Full Length Research Article

**STUDIES OF NICKEL AND COPPER-COATED CENOSPHERE-POLYMER COMPOSITES FOR
ELECTROMAGNETIC INTERFERENCE SHIELDING**

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ABSTRACT

Recently, electronic devices such as televisions, office automated appliances, computers, etc., have been using housing as a shield to reduce Electro Magnetic Interference (EMI) and Radio Frequency Interference (RFI). Polymer housing is conductive and increases the shielding effectiveness. This study involves preparing polymer-composites and determining their effectiveness as EMI-shielding materials. Non-conductive, hollow, spherical, fly-ash cenosphere particles (45-100 μm) have been used for electroless surface-coating with nickel and copper in order to make them electrically conductive. The metal-coated cenospheres (2% and 5% by weight) are blended with Polycarbonate (PC) to produce polymer-metal-coated cenosphere composites. The surface morphological analysis, chemical composition and phase analysis of the metal-coated cenospheres have been carried out using Scanning Electron Microscope (SEM), Energy Dispersive X-ray (EDX) and X-Ray Diffraction (XRD), respectively. Both nickel and copper are well-deposited on the pre-treated and activated cenosphere surfaces. EMI-shielding effectiveness of Polycarbonate-5% Nickel-Coated Cenosphere (PC-NCC) composites and Polycarbonate-5% Copper-Coated Cenosphere (PC-CCC) composites have been studied by means of coaxial holder method.

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INTRODUCTION

Electro Magnetic Interference (EMI) is a topic of current interest. EMI is a process by which disruptive electromagnetic energy is transmitted from one electronic device to another via radiated or conducted paths, or both. The interference sources may be internal or external to the electronic system and they may propagate by radiation or conduction (John Noto *et al.*, 2010). As technology advances, the need to integrate large number of electronic systems into automobiles, airplanes, ship, etc., has dramatically increased. These systems include Control Area Networks (CAN), safety systems, communications, mobile media, infotainment systems including wireless headsets, DC motors and controllers. Placing a large number of electronic systems in a confined space poses the problem of keeping the EMI of these systems from interfering with each other.

In an electronic system, EMI can adversely affect the performance of an integrated circuit internally as well as that of the other electronic components in close proximity. Typical examples of EMI include corona discharge, producing noise and disturbance around high-voltage transmission lines; distortion of TV picture observed when driven under high-voltage line; occurrence of loud static noise in a computer speaker if a cell phone is placed near the computer and when the cell phone begins to ring; hampering of airplane ground/air traffic control system and communication systems by using cell phones/laptops/CD players during take-off or landing of airplanes. If airbag, cruise control, anti-lock braking or other electronically-controlled assemblies are adversely affected by EMI, operation of the vehicle or its safety can become critical. Electro Static Discharge (ESD) also is an EMI problem which starts with a very slow build-up of energy, and upon reaching the threshold, rapid breakdown may occur. It is this fast breakdown that causes EMI problems in modern electronic systems. If the electronic system design is unable to reject all the unwanted signals, then the equipment must be protected or shielded, in order to obtain the desired performance or to

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prevent catastrophic failure. Shielding is more cost-effective when combined with other suppression techniques. Shielding can usually be installed after the design is complete. However, it is much more cost-effective and generally, it cannot be added easily once the device has gone beyond the prototype stage. The use of shielding may take many forms, from Radio Frequency (RF) gaskets to Board-Level Shielding (BLS). Shielding from the deleterious effects of EMI is achievable by using EMI-shielding materials which provide an electrically-conductive seal for the electronic equipment openings and housing the covers to prevent or restrict electromagnetic interference. There are many types of EMI-shielding materials such as electrically-conductive elastomers, Silicone Rubber or Fluoro Silicone Rubber-Based Materials, Metallic Materials and Metal-Coated Plastics.

A cenosphere is a light, inert, hollow sphere made largely of silica and alumina, and filled with air or inert gas, typically produced as a by-product of coal combustion at thermal power plants. In order to derive all the benefits of cenospheres and to explore its usability as EMI-shielding materials (after providing a conductive, thin metal coating on its surface), this study examines the potential of developing a silver-coated cenosphere-polymer composite. It is expected that coated cenospheres or microspheres would enable significant reduction of RF interference in much the same way as traditional solid silver/nickel particles, by developing chains of contacting particles into a vast network within the chosen polymer matrix, such as polycarbonate. Coated cenospheres can also be incorporated into plastics, polymers, composites and rubber compounds during the forming or molding stage, thus providing excellent EMI shielding as an integral part of the finished product.

There are many methods available to coat ceramics such as electroplating, hot dipping, metal spraying, vacuum metalizing, electroless plating, etc. Among these methods, electroless plating may be considered more suitable for the present study, since the substrates are in the form of powder (cenosphere) and the coating method is expected to provide with a thin, controllable and homogenized coating. Electroless coating provides additional benefits; it involves deposition of metal on the surface of a material by means of a reducing-chemical bath. In recent years, the plastic industry has been increasingly involved with electronic devices such as television, office automatic instruments, computers, etc. Because of the need for these housings to attenuate EMI and Radio Frequency Interference (RFI), the technique of imparting shielding to EMI-transparent polymers has received a great deal of attention. Polymer housing must be made conductive to exhibit the required shielding effectiveness (Xiang Cheng Luo and Chung, 1998). Polycarbonate (PC) are loaded with conductive fillers such as metal-coated cenospheres to make conductive PC composites possess effective EMI shielding.

Then, the process ability, Shielding Effectiveness (SE) values and morphology of PC/metal-coated cenosphere composites are studied. To prepare electrically conductive composites that contain conductive filler dispersed in an insulating polymer matrix, compression moulding and twin-screw extruder have been used in the present study. It is proposed to make use of waste from coal-fired thermal power plants, like cenosphere,

to function as filler for making polymer composites. The cenosphere is to be coated with metal by electroless processing. Finally, the metal-coated cenospheres, in combination with the polycarbonate matrix, have to be evaluated for their physical, chemical, structural and electrical properties, and to correlate these properties with their performance as suitable EMI shields. The objectives of this study are:

- To carry out complete characterization of cenospheres obtained from thermal power plants.
- To prepare metal-coated cenosphere by electroless processing followed by comprehensive characterization.
- To prepare and modify polymer blends, add metal-coated cenosphere, determine relevant properties to function as a material polymer matrix composites.
- To determine materials and electrical properties of polymers, fillers and blends of polymers-metal-coated cenospheres as composites.
- To evaluate the suitability of prepared polymer- matrix composites for their potential to perform as EMI-shielding materials and to characterize them.

Very little information is available in the literature on metal-coated cenospheres and on the compatibility of these materials as fillers in polymer composites as well as their usefulness as EMI-shielding materials. In this study, it has been proposed to make use of cenosphere as waste from coal-fired thermal power plants for making polymer composites. The cenospheres are to be coated with metals like Nickel and Copper by electroless processing. Finally, polymer composites are derived from the metal-coated cenospheres by combining with the polycarbonate matrix and then, subjected them to comprehensive characterization and evaluation of their suitability to perform as effective EMI-shielding materials.

MATERIALS AND METHODS

Cenospheres are subjected to acid wash before carrying out the coating process in order to remove impurities like bimetallic salts. Fly-ash cenosphere particles, supplied by Raichur Coal Plant, India, are used for electroless Nickel and Copper coating. Concentrated Sulfuric acid and distilled water are the materials required for pretreatment of cenospheres. Hot air oven is required for drying. The acid-washed cenosphere particles are then ready to be used. The chemicals used for electroless Ni coating of fly-ash cenosphere particles include SnCl_2 (anhydrous min. 99%), PdCl_2 (99.9%, metal basis), Pd (59.78%), NiSO_4 , NaH_2PO_2 , $\text{NH}_2\text{CH}_2\text{COOH}$, CH_3COONa . Electroless Ni coating requires two-step process, involving sensitization and activation, to make them suitable for subsequent Ni deposition. Acidic SnCl_2 solution is used as sensitizer, whereas acidic PdCl_2 solution is used as activator. Sn^{2+} can reduce Pd^{2+} ions since standard oxidation-reduction potential of $\text{Sn}^{4+}/\text{Sn}^{2+}$ couple (0.15 V) is less than that of $\text{Pd}^{2+}/\text{Pd}^0$ couple (0.987 V). The electroless Nickel-coating bath consists of the following components:

Nickel sulphate is an ideal source of Nickel ions and the most preferred reducing agent is sodium hypophosphite. Sodium acetate is used to adjust the pH. A pH of 5-6 is maintained. Under the catalytic action of Pd^0 , Ni cations get deposited onto the surface by capturing electrons furnished by a common reducing agent (NaH_2PO_2).

The electroless copper-coating bath consists of the following components:

Copper sulphate is used as the source of copper ions and the reducing agent is Formaldehyde. Formaldehyde is oxidized to Formic acid in the presence of a strong alkali, generating two electrons which reduce cupric ions to metallic copper. In order to prevent the formation of Copper hydroxide precipitate, the complexing agent Sodium potassium tartarate is added. Other complexing agents used are EDTA and a variety of alpha hydroxy carboxylic acids. Sodium hydroxide flakes are added to maintain the pH between 10 and 12. In the electroless Copper-coating bath, under catalytic action of Pd⁰, Copper cations get deposited onto the surface by capturing electrons furnished by the reducing agent Formaldehyde.

The various types of composites that were prepared are Polycarbonate (PC), Nickel-Coated Cenosphere Composites (NCC) and Copper-Coated Cenosphere Composites (CCC), Uncoated cenosphere particles, Twin-Screw Extruder for Compounding of PC/NCC/CCC and Compression Moulding, After compounding, a batch of composites are sent into the frame, delivered into a pair of plates, and pressed by compression moulding at 260°C (Table 1) and at the pressure 150 kgf/cm².

Table 1. Conditions for compounding of composites

PC/NCC/CCC Composition (wt.%)	Processing Temperature (°C)
98/2	260
95/5	255

When the blends are completely melted, the ultimate pressure is maintained for 10 min, and then cooled by water at the rate of about 10°C/min until the temperature fell below 50°C. The main measuring method for EMI- Shielding Effectiveness is the coaxial-holder method. This method uses a specimen holder, transmission line (signal generator) and a vector network (data) analyzer. The sample material is placed and fixed in the flanged circular coaxial transmission line holder. By measuring the S-parameters, S₁₁ and S₂₁ – reflection and transmission coefficients, it is possible to determine the contribution of the absorption and the reflection at the total shielding effectiveness. Generally, the maximum operating frequency is around 1 GHz. This measuring system is compact and allows automation and data proceeding by computer control. The difficulty of this measurement method arises from the sample preparation.

EXPERIMENTAL

Electroless Coating of Cenosphere with Nickel

The as-received fly-ash cenosphere particles were stirred in the acidic SnCl₂ bath, containing 5 g/L of SnCl₂ and 30 ml/L of conc. HCl acid for 1 h (sensitization step). The sensitized particles were filtered off and then, transferred to acidic PdCl₂ bath containing 0.1 g/L PdCl₂, 25 ml/L HCl and 5 ml/L HNO₃ and stirred in this bath for 1 h (activation step). The activated particles were filtered off and washed thoroughly with de-ionized water. The particles were then transferred to the coating bath involving 25 g/L NiSO₄, 30 g/L NH₂CH₂COOH, 30 g/L NaH₂PO₂ and 20 g/L CH₃COONa for actual Ni-

deposition for 1 h at 80°C. The Ni-coated particles were dried in vacuum oven at 110°C for 1 h.

Electroless Coating of Cenosphere with Copper

Similar procedure was carried out for the sensitization and activation steps. The activated particles were filtered off and washed thoroughly with de-ionized water. The particles were then transferred to the coating bath involving 12g/l of Copper sulphate, 30 g/l of Sodium potassium tartarate and 5 ml of Formaldehyde for actual Cu-deposition for 1 hour at room temperature. The Copper-coated particles were dried in a vacuum oven at 110°C for 1 h.

Preparation of PC-NCC/CCC Composites

The twin-screw extruder machine was preheated for two hours. When the temperature attained the set values, PC was used to clean the extruder for 10 to 15 min. The PC-NCC/CCC was loaded to the machine by controlling the four-step temperature at 260± 2°C each and the rotating speed at about 110 rpm. When the material content was increased, the temperature decreased. Compositions of the composites prepared were 98/2 and 95/5 (% weight).

Characterization of Uncoated and Metal-Coated Cenospheres

Characterization studies were done for uncoated and metal-coated cenospheres using Scanning Electron Microscope (SEM), Energy Dispersive X-ray (EDX) and X-Ray Diffraction (XRD).

Characterization using SEM Analysis

There are two basic types of Scanning Electron

Microscope (SEM), one which is regular type requiring a conductive sample and the other is an environmental SEM that can be used to examine a non-conductive sample without being coated with a conductive material. In this work, the former was used. About 1 spatula of each of the raw material sample was taken and spread on an aluminium stub. The sample was blown carefully on the stub; it was kept in a sputter coat unit. Gold coating was done to increase the conductivity. When the electrons struck the sample, the conductivity increased and the sample under study could be clearly viewed in the SEM. Acetone was added so that the sample stuck on the aluminium stub. The samples were mounted in the SEM and the lid was closed. A vacuum of 0.975×10⁻⁵ torr was applied. The equipment was started and the analysis was carried over. The image was finally obtained and the results were interpreted.

Characterization using EDX Analysis

The gold-coated specimen was bombarded with an electron beam inside the SEM. The bombarding electrons collided with the specimen atom's own electrons, knocking some of them off in the process. The amount of energy released by transferring electron depended on the shell it was transferred from and also the one it was transferred to. Furthermore, the atom of every element released X-rays with unique amount of

energy during the transferring process. Thus, by measuring the amount of energy present in the X-rays being released by a specimen during electron-beam bombardment, the identity of the atom from which the X-ray emitted could be established.

Characterization using XRD Analysis

Powdered sample was taken on a sample holder and back-loaded. While loading, the sample was compacted, spread uniformly over the specimen holder and then, kept inside XRD set up. Back-loading technique was done to avoid preferred orientation effects. When the powder was compacted on the sample holder, the natural orientation of the sample was disturbed. This orientation is called preferred orientation. The back surface of the sample was disturbed whereas, the front surface of the sample was not disturbed. Hence, proper peaks and orientation of the sample could be observed on the front surface.

Shielding-Effectiveness Test

Coaxial-holder method was used for testing the shielding effectiveness of the composites. Composites were cut according to the dimensions needed to load into the coaxial jig. Initially, sample 'a' was loaded into the coaxial jig. One end of the jig was connected to the signal generator and the other end was connected to the network analyser. In the signal generator, the power level was set to -27 dB which was maintained constant for all the measurements with different input frequencies, ranging from 100 KHz to 1 GHz and respective dB output values were noted. The measurements were repeated with samples 'b' and 'c' loaded together in the jig with the above frequency ranges and the dB values were noted. The difference in the output dB values with shield (sample 'a') and without shield (samples 'b' and 'c' together) were calculated.

RESULTS AND DISCUSSION

SEM and EDX Analyses of Uncoated Cenosphere

SEM micrograph of uncoated fly-ash cenosphere particles are presented in Fig. 1. The as-received cenosphere particles have spherical surface morphology, evident in Fig. 1.

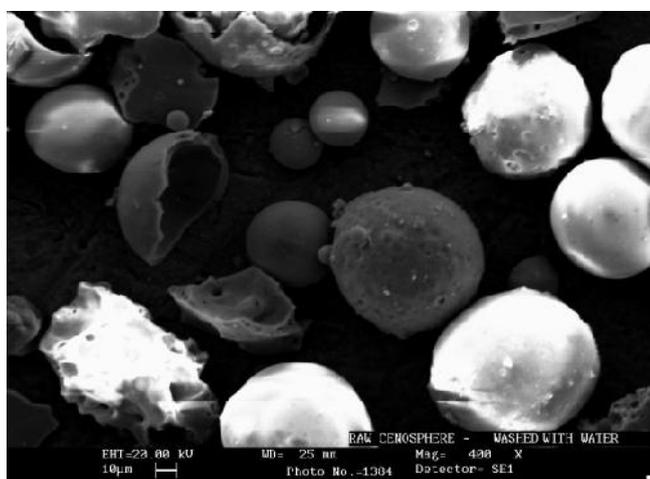


Fig. 1. SEM Micrograph of as-received Cenosphere Particles at 400 Magnification

It can also be noted that the as-received cenosphere contains broken particles. Therefore, cenospheres must be filtered to separate the broken particles and this was done using 45μm mesh. The chemical composition of fly-ash cenosphere particles mainly composed of mixture of oxides such as SiO₂, Al₂O₃ and Fe₂O₃ as indicated by the EDX analysis (Fig. 2). Various trace elements, such as K, Ca, Mg, Ti and C are also present. No Ni or Cu is detected for uncoated particles.

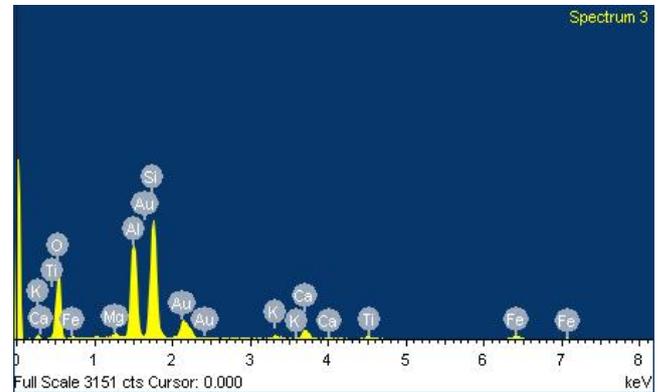


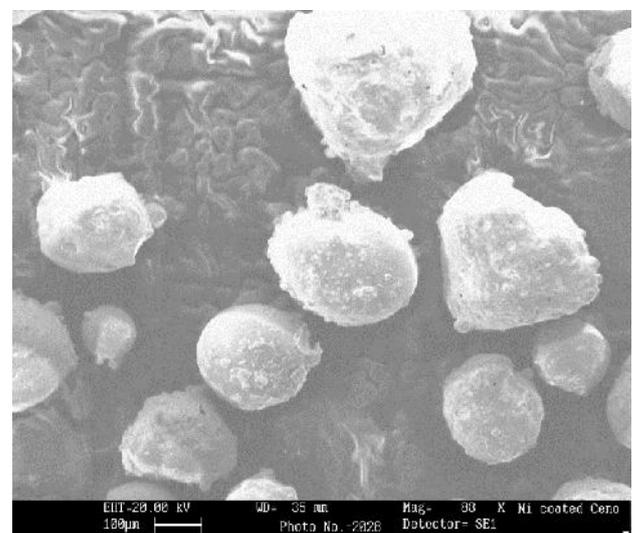
Fig. 2. EDX Analysis of Uncoated Fly-Ash Cenosphere Particles

XRD Analysis of Uncoated Cenosphere

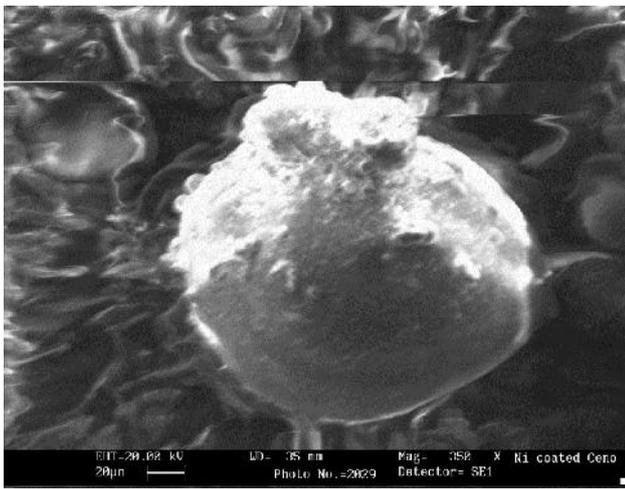
The XRD pattern was also obtained for uncoated cenosphere particles in which the major peaks of Mullite and Quartz could be seen (not shown in this paper). The intensities of the peaks indicate the crystallinity. The peak intensities are very low and the noise level is high indicating that the cenosphere is highly amorphous in nature.

SEM Analysis of Nickel-Coated Cenospheres

SEM micrographs of Ni-coated fly-ash cenosphere particles are shown in Fig. 3. It is also to be noted that even the Ni-coated particles exhibit spherical surface morphology, indicating the uniform nature of the Ni coating obtained by this technique. The uniform coating is evident at low and high magnifications, where some clusters are also seen to be deposited on the surface.



(a)



(b)

Fig. 3. SEM Micrographs of Ni-Coated Cenosphere Particles at (a) 88 × Magnifications and (b) 350 × Magnifications

At high magnification (Fig. 3 (b)), the Ni-coated surface reveals the presence of nanoparticles. It appears that during the Ni coating over the sensitized and activated cenosphere particles, surface develops through Ni nanoparticles formation.

EDX Analysis of Nickel-Coated Cenospheres

EDX analysis of Ni-coated cenosphere particle surface (Fig. 4) confirms the presence of Ni along with the other underlying substrate elements such as Si, Fe, Al and K. It is also to be noted that the peak corresponding to Ni was not present in Fig. 2 and it appears only after electroless coating process, indicating successful Ni deposition on the cenosphere particle surface by the present electroless technique. Thus, SEM and EDX analyses suggest the successful Ni deposition on the surface of fly-ash cenosphere particles by the electroless process.

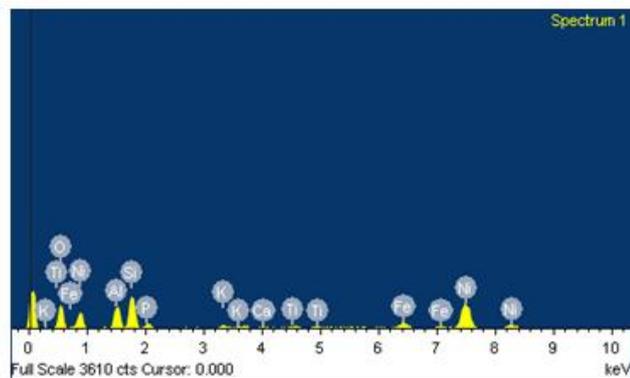


Fig. 4. EDX Analysis of Ni-coated Fly-ash Cenosphere Particles

XRD Analysis of Nickel-Coated Cenospheres

The XRD pattern obtained is shown in Fig. 5, in which the major peaks of Ni can be seen. The Nickel peak confirms that Nickel has been successfully coated over the surface of the cenospheres. The sharpness of the peaks indicates the crystallinity. Peak corresponding to silicon oxide belongs to the cenosphere.

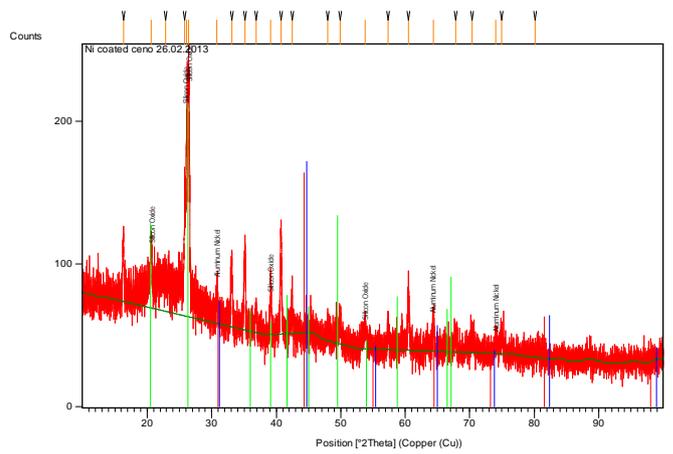


Fig. 5. XRD Pattern Obtained for Ni-Coated Fly-Ash Cenosphere Particles

Thickness Analysis via SEM of Nickel-Coated Cenospheres

Scanning Electron Microscope is used to measure the Nickel-coating thickness by mounting the Ni-coated cenosphere particles using acrylic. The moulded sample is polished using emery paper (240µ, 320µ, 400µ, 600µ, 800µ and 1000µ) and then, diamond- polished and gold-coated. The sample has been studied under SEM and its thickness is measured, as shown in Fig. 6. The point to point measurement shows the thickness of Ni coating developed on the surface of fly-ash cenosphere which has been found to be 1.39 µm.

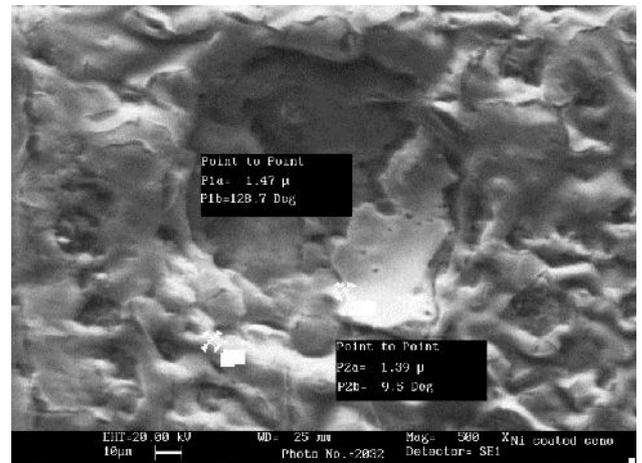


Fig. 6. SEM Micrographs Showing the Thickness of Nickel Coating Developed on the Surface of Fly-ash Cenosphere

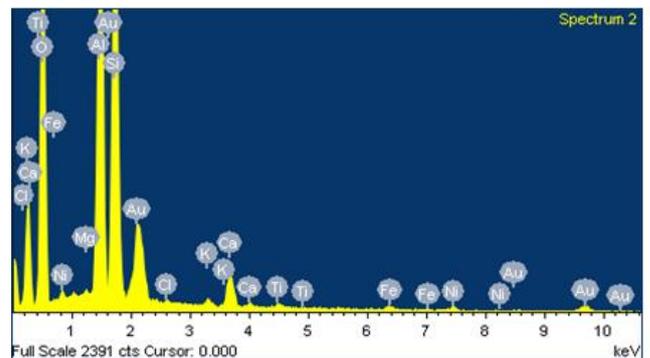
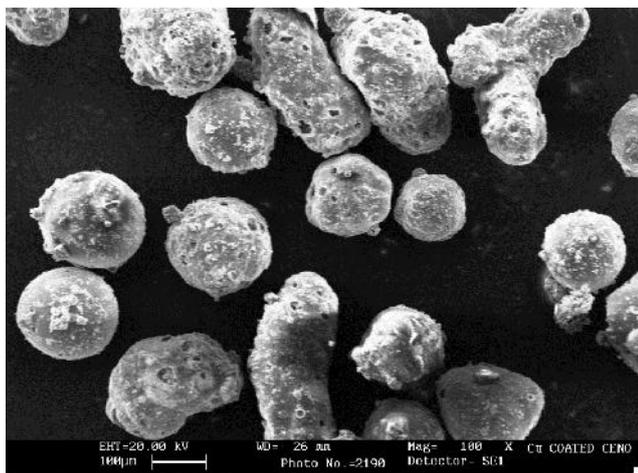


Fig. 7. EDX Analysis Confirms that Thickness Measured is Actually the thickness of Ni-Coating and not the thickness of the Cenosphere

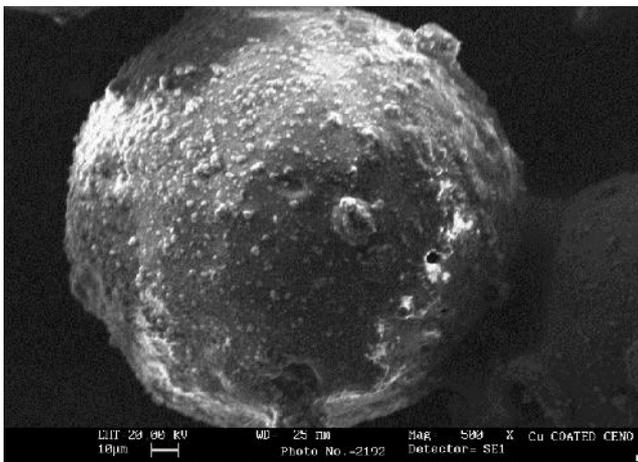
It is evident from Fig. 7 that thickness measured is actually the thickness of Ni coating and not the thickness of the cenosphere.

SEM/ EDX Analysis of Copper-Coated Cenospheres

Fig. 8 shows SEM micrographs of Cu-coated cenosphere particles at 100X and 500X magnifications. The cenospheres have retained their spherical shape even after copper coating. It appears that during electroless metal deposition, uniform copper coatings have been developed over the activated cenosphere particle surface.



(a)



(b)

Fig. 8. SEM Micrographs of Cu-Coated Cenosphere Particles at (a) 100 × Magnifications and (b) 500 × Magnifications

EDX Analysis of Copper-Coated Cenospheres

EDX analysis of Cu-coated cenosphere particle surface (Fig. 9) shows the prominence presence of Cu along with the other underlying substrate elements such as Si, Fe, Al and K. It is also to be noted that peak corresponding to Cu was not present in Fig. 2 and it appears only after electroless coating process, indicating successful Cu deposition on the cenosphere particle surface by the present electroless technique. At high magnification (Fig. 8 (b)), the Cu-coated surface revealed the presence of nanoparticles (figure not given). It appears that during the Cu coating over the sensitized and activated cenosphere particles, surface develops through Cu nanoparticles formation.

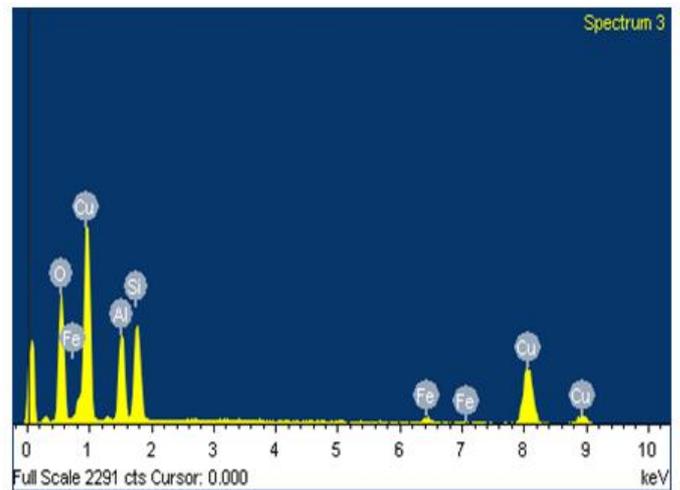


Fig. 9. EDX Analysis of Cu-Coated Fly-Ash Cenosphere Particles Showing the Presence of Cu as a Major Element on the Particle Surface Indicating Successful Cu Coating of Fly-Ash Cenosphere Particles

Thus, SEM and EDX analyses suggest the successful Cu deposition on the surface of fly-ash cenosphere particles by the present electroless process.

XRD analysis of Copper-Coated Cenospheres

The XRD is shown in Fig. 10, where the major peaks of Cu can be seen. The Copper peak confirms that Copper has been successfully coated over the surface of the cenospheres. The sharpness of the Copper peaks indicates crystallinity of the coating. Peak corresponding to silicon oxide belong to the cenosphere which is non-crystalline in nature.

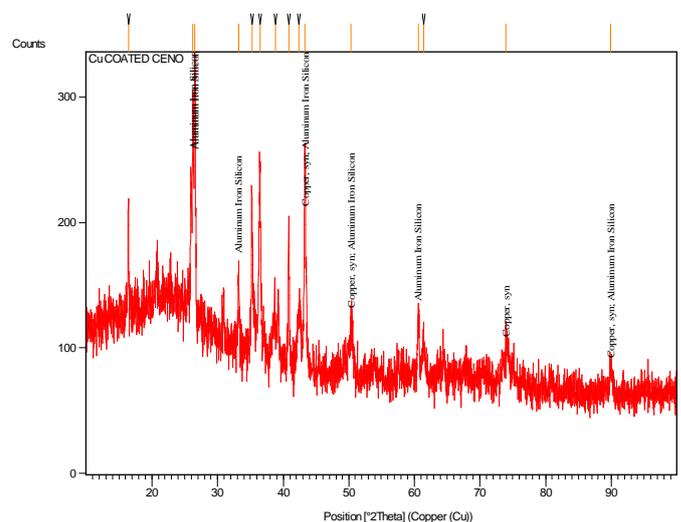
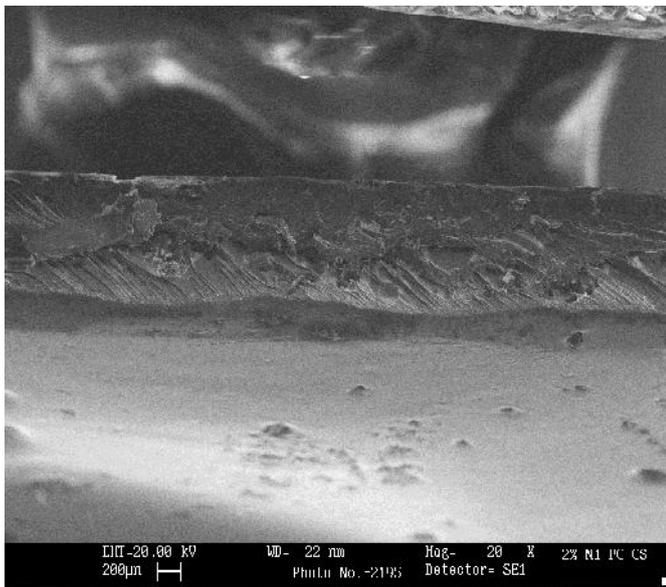


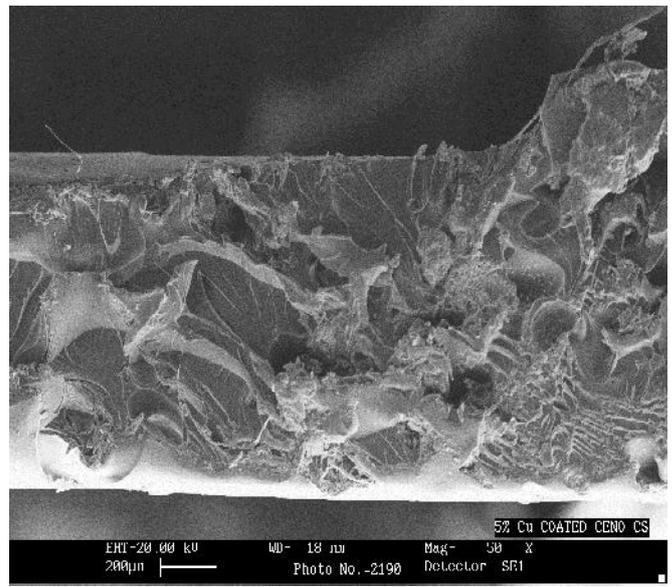
Fig. 10. XRD Pattern Obtained for Cu-Coated Fly-Ash Cenosphere Particles

PC-NCC/CCC Composites

From the SEM images in Fig.11, it can be observed that coated cenosphere particles are embedded inside the polymer matrix and the distributions of cenosphere particles are non-uniform which may be attributed to non-uniform mixing.

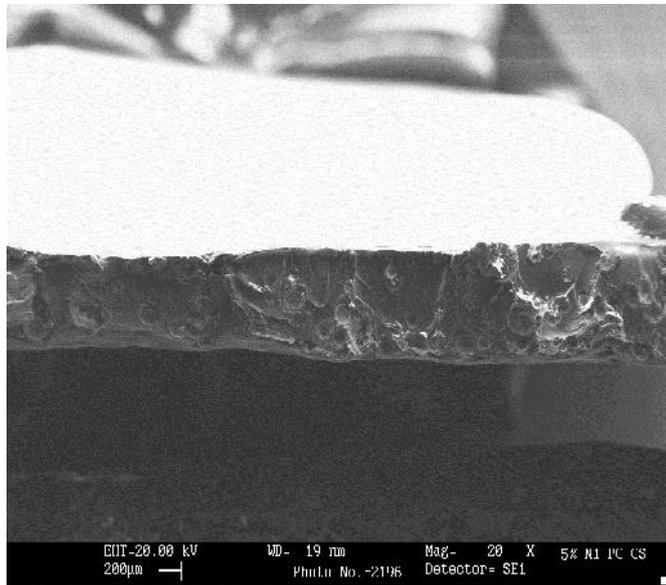


(a)

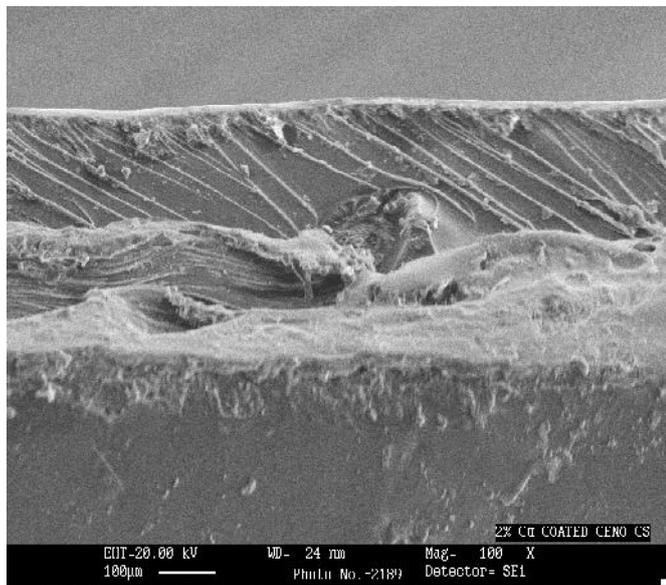


(d)

Fig. 11. Representative SEM images of (a & b) 2% and 5% NCC-PC Composites, respectively. (c & d) 2% and 5% CCC-PC Composites, respectively



(b)



(c)

Shielding Effectiveness Test Results

5% PC-NCC Composites

Shielding Effectiveness of 5% PC/NCC composites were tested (as described in chapter 4) and the readings obtained are tabulated below.

Table 2. Shielding effectiveness readings of 5% PC/NCC composites

Frequency (Hz)	Shielding Effectiveness (SE) with shield (dB)	Shielding Effectiveness (SE) without shield (dB)	Shielding Effectiveness (SE) of 5% PC/NCC, dB (SE with shield – SE without shield)
100K	-84.35	-93.12	8.77
200K	-78.83	-87.43	8.6
500K	-70.79	-79.47	8.68
1M	-64.82	-73.58	8.76
2M	-60.11	-69.22	9.11
5M	-51.51	-60.65	9.14
10M	-35.37	-52.46	15.28
20M	-42.96	-50.65	7.69
50M	-35.63	-42.63	7
100M	-34.82	-40.58	5.76
200M	-30.61	-34.16	3.55
500M	-32.81	-31.42	-1.39
1G	-46.55	-28.75	-17.8

From Table 2, it can be observed that the shielding effectiveness is more or less constant at lower frequencies and increases to maximum of 10 MHz and gradually decreases at higher frequencies. At very high frequency above 500 MHz, there is no shielding effectiveness. The overall Shielding effectiveness of 5% PC/NCC between 100 KHz to 200 MHz is 8.39 dB (The dB values at 500 MHz and 1 GHz have not been included).

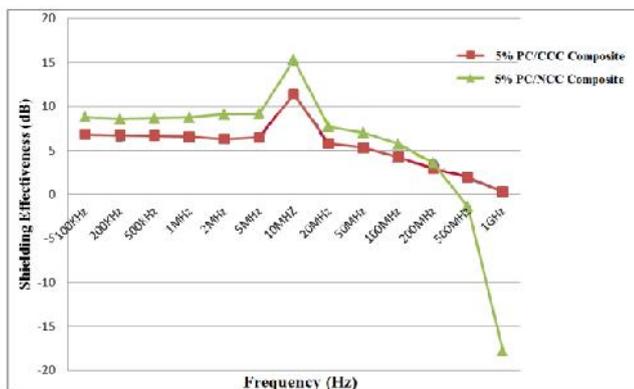
5% PC-CCC Composites

Shielding Effectiveness of 5% PC/CCC composites were tested and the readings obtained are tabulated below:

Table 3. Shielding effectiveness readings of 5%PC/CCC composites

Frequency (Hz)	Shielding Effectiveness (SE) - with shield (dB)	Shielding Effectiveness (SE) - without shield (dB)	Shielding Effectiveness (SE) Cu, dB = SE of with shield – SE of without shield
100K	-86.48	-93.28	6.8
200K	-80.15	-86.85	6.7
500K	-72.04	-78.68	6.64
1M	-65.90	-72.44	6.54
2M	-61.24	-67.55	6.31
5M	-52.72	-59.21	6.49
10M	-38.26	-49.68	11.42
20M	-43.68	-49.45	5.77
50M	-36.13	-41.42	5.29
100M	-35.28	-39.51	4.23
200M	-30.67	-33.58	2.91
500M	-28.90	-30.85	1.95
1G	-27.93	-28.23	0.3

From Table 3, it can be observed that the shielding effectiveness more or less constant at lower frequencies and increase to a maximum at 10 MHz and gradually decreases at higher frequencies. The overall Shielding effectiveness of 5% PC/CCC between 100 KHz to 200 MHz is 6.28 dB. A plot showing the EMI Shielding Effectiveness at various frequencies (ranging between 100 KHz and 1 GHz) for the two composites (PC/ 5% Nickel/ 5% Copper-Coated Cenospheres) is given in Fig. 12.

**Fig. 12. Shielding Effectiveness of PC-NCC/CCC (5% metal-coated cenospheres) Composites**

From Fig. 12, it can be interpreted that the shielding effectiveness of the two composites shows maximum at 10 MHz. Shielding effectiveness of 5% Polycarbonate-Nickel-coated cenosphere composites shows better EMI shielding capabilities compared to 5% Polycarbonate-Copper-coated cenosphere composites.

Conclusions

From the characterization of metal-coated cenospheres, the following conclusions were drawn:

From the SEM analysis:

- The coated and uncoated cenospheres had true spherical morphology.
- Cross-section analysis showed the uniformity of the metal coating deposition on cenosphere surfaces and allowed the determination of their thicknesses. The thicknesses' varied between 1 and 1.5 μm .

From the EDX analysis:

- Both Nickel and Copper were successfully deposited on the cenosphere particles. The chemical compositions of the coating were revealed by detailed SEM and EDX analyses.
- The results of metal-coating compositions quantitatively revealed that the metals were well- deposited on the cenosphere surfaces. The thickness of the coatings were very small ($\sim 1.5\mu\text{m}$).

From the phase analysis by XRD:

- Phase analysis by using XRD revealed the presence of Nickel and Copper in their elemental form. In addition, presence of their compounds with oxygen and other elements were also found. That is, the coatings were not totally elemental.

From the Shielding Effectiveness Test:

- The Shielding effectiveness of 5% PC/NCC/CCC composites between 100 KHz to 200MHz was 8.39, 5.68 and 6.28 dB, respectively.
- From the shielding effectiveness of all the two composites, it could be concluded that the EMI- Shielding effectiveness of 5% Polycarbonate- Nickel-coated cenosphere composites was better than the shielding capabilities of 5% Polycarbonate-Copper-coated cenosphere composites. From the EMI-Shielding effectiveness vs Frequency plot, the effectiveness of both the composites had increased to a maximum of 10-15 dB at 10 MHz.

REFERENCES

- John Noto, Gary Fenical and Colin Tong. 2010 Automotive EMI Shielding-Controlling Automotive Electronic Emissions and Susceptibility with Proper EMI Suppression Methods.
- Xiang Cheng Luo and D.D.L. Chung. 1998. Electromagnetic Interference Shielding using Continuous Carbon-Fiber, Carbon-Matrix and Polymer-Matrix Composites. Composite Materials Research Laboratory, State University of New York at Buffalo, Buffalo, NY, USA.
- Rajendra Kumar Goyal and Amol Kadam. 2010. Polyphenylene Sulphide/Graphite Composites for EMI-Shielding Applications. *Adv. Mat. Lett.* 1(2): 143-147.
- Das, N.C., Yamazaki, S., Hikosaka, M., Chaki, T.K., Khastgir, D. and Chakraborty, A. 2005. Electrical Conductivity and Electro Magnetic Interference Shielding Effectiveness of Polyaniline-Ethylene Vinyl Acetate Composites. *Polym. Int.* 54: 256.
- Das, N.C., Khastgir, D., Chaki, T.K. and Chakraborty, A. 2000. Electromagnetic Interference Shielding Effectiveness of Carbon Black and Carbon Fibre Filled EVA and NR Based Composites. *Composites Part A, Elsevier Science.* 31(10): 1069-1081.
- Agarwala, R.C. and Vijaya Agarwala. Electroless Alloy/Composite Coatings: A Review. 2003. *Sadhana.* 28: 475-493.
- Xiaozheng Yu and ZhigangShen. The Electromagnetic Shielding of Ni Films Deposited on Cenosphere Particles by Magnetron Sputtering Method. 2009. *Journal of Magnetism and Magnetic Materials.* 321(18): 2890-2895.

Paunovic, M. and Schlesinger, M. 1998. *Fundamentals of Electrochemical Deposition*. Wiley Inc., New York. p. 133.

Xinrui Xu, Xiaojun Luo, Hanrui Zhuang, Wenlan Li and Baolin Zhang. 2003. Electroless Silver Coating on Fine Copper Powder and its Effects on Oxidation Resistance. *Mater. Lett.* 57: 3987-3991.

Supriya, M., Sashikala, M., Manish S. Khodwe, Soundarajan Krishnan and Vynatheya, S. 2013. Studies on the Potential of Silver-Coated Cenosphere-Polycarbonate Composites for Electromagnetic Interference Shielding. *International Journal of Recent Scientific Research.* 4(11): 1736-1742.
