Journal of Metallurgy and Materials Science, Vol. 62, No. 3-4, July-December 2020, pp. 71-80
Printed in India, © NML, ISSN 0972-4257

Tuning surface resistivity and thermal conductivity of water resistant fly ash waste based polymer composite via tailoring the interfacial polarization

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Abstract: High performance environment friendly industrial inorganic waste (fly ash) waste based water resistant electrical insulating hybrid composites are fabricated with very high surface and volume resistivity. Dielectric constant of hybrid composite was decreased significantly from 10900 to 4.98, 5.25, 5.00, 5.81, and 6.69 for filler concentrations of 10, 20, 30, 50 and 60% in epoxy matrix, respectively. Very high surface resistivity of 10^{13} Ohms/sq. and volume resistivity of 10^{14} to 10^{15} Ohms-cm with ultra-low water absorption of 0.14 % were achieved . Thermal conductivity analysis shows a slight increase in the thermal conductivity of the composite sheet and reaches the value of 0.4387 W/mK. Such high resistivity is attributed due to low dielectric constant and interfacial polarization and low water absorption in the samples. Our approach presents new, adaptable and cost-effective means for effectively utilizing waste as eco-friendly electrical and thermal insulating sheet and lowering the thermal loss in microelectronics.

INTRODUCTION

Fabrication of electrical insulator with high resistance to electric current has gained huge interest for separating electrical conductors and building electrical insulating panels. Previously, many materials e.g. wood, glass, fabrics, mineral oil, and ceramics are used for electrical insulating purposes [1,2]. With the growth of electrical/electronics industry, there is high need for an alternative material that possessed the desired electrical, thermal, and mechanical properties. New high performance engineering polymeric materials with additional properties such as ease of fabrication, lighter weight, low cost and excellent insulation properties are the most desirable and demanding materials for electrical applications[1-6]. In addition, the interface between filler and matrix has long been a critical problem that affects the thermal conductivity and electric properties of the polymer composites. Industrial waste from thermal power plant especially fly ash of about 150 millions of tons generated across the world every year[7]. Moreover, enviro-friendly utilization of fly ash become global challenge as dumping the fly ash into the open environment creates air, land and water pollutions. Heavy metal presented in fly ash are most important sorts of contaminant in the environment that produced severe diseases such as lung cancer, anemia, dermatitis and skin cancer as their small sizes penetrate into different organs of the human system and also create permanent respiratory disorder [8-9]. In addition, dumping of fly ash into the environment creates air, land and water pollutions. Presently, fly ash are utilized in to cement manufacturing, filling of low lying area, construction of road and embankment, making fly ash brick, ash pond dyke raising and also in agriculture area with certain conditions.[10] Various researches have been carried out across the world to find an economical and suitable way to use fly ash, the industrial waste for some application[7]. However, fly ash solidification in cement and disposal at landfills is not an optimized solution due to variation of fly ash characteristics. Fly ash as basement material for road construction is also limited to 5 % in the United States and Japan, due to releasing of heavy metals from solidification matrix. Moreover, to preserve the toxic element including fly ash numerous thermoplastics and thermosets are used [11]. Quan et al. have studied the role of fly ash in the performance of fly ash concrete [12]. Lingling et. al have also studied fired bricks with replacing clay by fly ash in high volume ratio and shown that fired fly ash bricks has high compressive strength, low water absorption, no cracking due to lime, no frost and high resistance to frost melting. The properties of fired bricks were improved by pulverized fly ash [13]. However, the sintering temperature of bricks with high

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volume of fly ash was about 1050^0C, which is very high and energy consuming. PVC resin and fly ash based polymer composite and fly ash/ polyurea (FA/PU) composites have been also reported by Nidal H. et al and Jing Qiao et al, respectively [14-15]. They have reported thermal properties and loss of moduli and compared with those of neat polymer. However, dielectric properties, surface resistivity and ac conductivity are still not reported for fly ash based polymer composites. H. Khan et al. have shown that SiR (Silicone Rubber) composites with addition of fillers are used as high voltage (HV) electrical and non electrical application, [16] and Masahiro Kozako et. al. have improved both thermal and electrical insulation properties of epoxy based composites using nanocomposite technique [17]. In addition, the rapid development of micro-electronic technology, and highly integration of the electric component cause the high heat flux and release of high heat from electronic component creates severe problem to electronic devices. Moreover, the heat dissipation is also an important issue for electronic devices and controlling them by tuning the thermal conductivity and dielectric loss tangent is very important. However, to the best of our knowledge investigation of fly ash based polymer composite with epoxy system as electrical insulation with superior dielectric and high surface/volume resistivity through lowering the interfacial polarization and water absorption are not reported till date.

In this work, water resistant fly ash-based polymer-based composites are prepared in epoxy matrix. Dielectric constant, dissipation factor, electrical conductivity, surface and volume resistivity and water absorption of the fly ash waste polymer composite with variation of filler concentration have studied in details. Physical, chemical and dielectric properties of fly ash industrial waste powder was also investigated.

EXPERIMENTAL SECTION

Fabrication of electrical insulating sheet using fly ash waste.

The inorganic fly ash waste powder is collected from Sarni, district of Madhya Pradesh state of India. Physiochemical properties such as pH, density, porosity and electrical conductivity of fly ash waste powder were measured. The synthesis of polymer composites were carried out using hand operated compressive moulding machine at room temperature. The prepared solution was poured in specific mould frame and then allowed to cure for 6 hours at room temperature.

Characterization and measurements.

Elemental analysis was performed using S8 Tiger X-Ray fluorescence spectrometer (XRF). Morphology of the fly ash powder was identified using scanning electron microscope (SEM) using JEOL, Model :NeoScope, JCM6000. Dielectric constant and dissipation factor of fly ash waste powder and their polymer composites were measured using Keysight LCR meter (E4980A) in the frequency range 20Hz–2MHz at room temperature. Surface and volume resistivity are measured using Tektronix Electrometer Model 8009 Resistivity Test Fixture as per ASTM D-257 standard. Water absorption study of fly ash waste epoxy composites was performed as per ASTM D 570-98 method.

RESULTS AND DISCUSSIONS

Physicochemical analysis of fly ash powder.

The physio-chemical analysis through various parameters such as pH value, bulk density, specific gravity, conductivity and porosity of fly ash waste powder were measured and calculated as per standard procedures. Bulk density of the fly ash sample was calculated using IS: 2386-1963 method and the calculated value is found to be 1.04 g/cm^3. Specific gravity of fly ash was calculated using the IS CODE 2720 Part 3 and found to be 1.39. The pH of fly ash sample of 7.04 and conductivity of 2.06 µS/cm were obtained using ASTM standard. The detail results are shown in Table No. 1.The porosity was calculated using the known value of bulk density and specific gravity and porosity of fly ash waste sample was found to be 25.68 %.

Table 1: Physicochemical properties of fly ash waste powder

S No.	Parameter	Value
1	pH	7.04
2	Bulk Density	1.04 g/cm^3
3	Specific Gravity	1.39
4	Electrical Conductivity	2.06 µS/cm
5	Porosity	25.68 %

Scanning electron microscopy (SEM) of fly ash sample.

The morphology of the fly ash powder sample is recorded using scanning electron microscope studies and results are shown in the shown in Fig.1 (A-C). It is evident that the fly ash sample has cenospheres morphology and size in the range of 3 -12 µm. The morphological results was well correlated with the characteristic morphologies of coal fly ash sample [18-20].

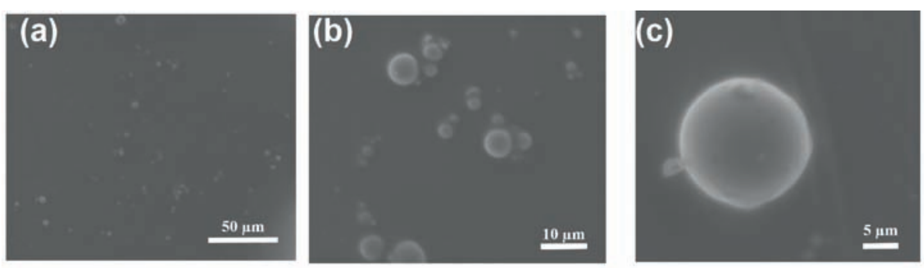

Fig.1. (a) low (b) mid and (c) high magnification of the SEM images of the fly ash waste powder sample

X-Ray Florescence (XRF) studies.

Wavelength dispersive X-ray fluorescence (WD-XRF) is a well-known technique for fast, and accurate quantitative determinations of matrix elements over a wide concentration range. To perform the elemental analysis of the fly ash sample, a pellet is prepared using the 5g fly ash and 5 gm boric acid.[21] The results obtained by using WD-XRF analysis is shown in Fig 2A and Table 2. It is clearly visible that fly ash has SiO_2 (57.3 %), CaO (1.12 %), Fe_2O_3 (3.73 %), Al_2O_3 (27.8 %), MgO (0.967 %), SO_3 (0.243 %) , Na_2O, (0.210 %), K_2O (3.00 %) TiO_2 (1.92 %), P_2O_5 (0.232 %) and various elements including heavy elements such as Zn, Mn, Cu, Ni, Pb, and Zr concentration in ppm range are also detected. The results are displayed in Fig 2B.

Table 2: XRF of fly ash waste powder showing elemental composition of the fly ash particulates

S. No.	Oxides	% in fly ash
1.	SiO_2	57.3 %
2.	CaO	1.12 %
3.	Al_2O_3	27.8 %
4.	Fe_2O_3	3.73 %
4.	MgO	0.967 %
5.	SO_3	0.243 %
6.	Na_2O	0.210 %
7.	K_2O	3.00 %
8.	TiO_2	1.92 %
9.	P_2O_5	0.232 %

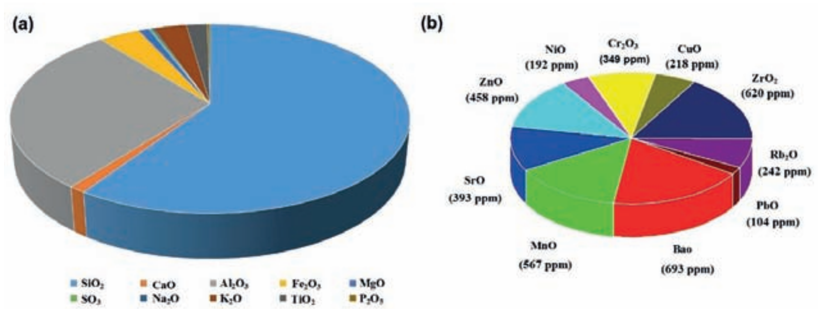

Fig. 2. (a)WD- XRF results of the fly ash waste powder showing elemental composition (b) heavy element presented in the fly ash samples

Water absorption of Fly ash polymer composite sheet.

Electrical insulating sheet prepared from fly ash with various content (10, 20, 30, 50 and 60 wt %) is shown in the Fig 3. Calculation of water absorption of fly ash polymer composite was carried out through initial and final weight of fly ash polymer composite samples before and after fully immersion in distill water bath for 24 hours are measured and calculated.

Fig. 3. Original image of fly ash waste based polymer composite (FAPC) samples with various filler concentration

Samples are conditioned at 50°C for 24 hours before testing the water absorption of samples. Water absorption measured for 10, 20, 30, 50 and 60 wt% fly ash waste based polymer composite samples is shown in Fig 4 a and b.

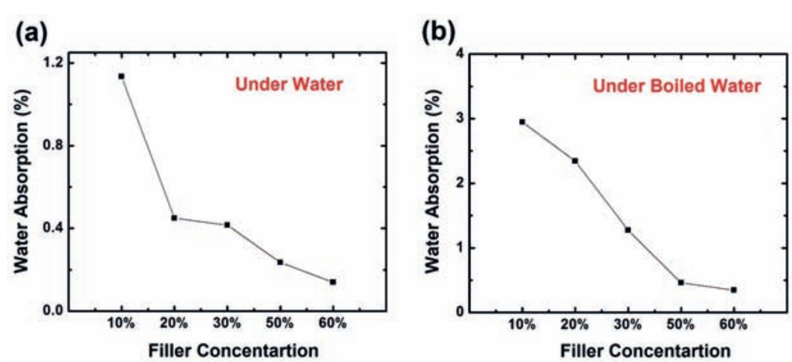

Fig. 4 (a) Water absorption (b) Water absorption under boiled water of fly ash waste hybrid polymer composites.

It is clearly seen that as the weight amount of the fly ash waste powder in epoxy matrix system increases as the water absorption decreases. The water absorption of value 1.13, 0.44, 0.416, 0.84 and 0.14 % were obtained for fly ash polymer composite sheet containing 10, 20, 30, 50 and 60% of filler amount (fly ash) for 24 hour water immersion test. Similar procedure was repeated for boiling water absorption test and fly ash composite samples was immersed in boiling distill water for 2 hours. Boiling water absorption test shows that water absorption of 2.94, 2.34, 1.27, 0.45 and 0.34 % were obtained for fly ash polymer composite samples of filler concentration 10, 20, 30, 50 and 60 % of fly ash. It has been observed that very low water absorption upto 0.14 % and 0.34 % was obtained for sample containing 60 % wt of fly ash in water absorption and boiling water absorption test, respectively. The results show that the prepared polymer fly ash polymer composites are water resistant and suitable for high end application towards fabrication of electrical insulations sheet and other construction materials.

Scanning electron microscopy (SEM) of fly ash polymer composite.

The morphology of fly ash based polymer composites are analysed using SEM technique. Figure 5 (a-d) depict the SEM micrographs of industrial fly ash polymer composites samples. SEM technique is a strong method to see the interfacial bonding of inorganic particulates with the polymer. To investigate the interfacial bonding of prepared polymer composite sample, SEM of fracture sample has been used. Fig 5 clearly indicate that fly ash are randomly distributed with the epoxy system and no leakage or weak bonding with polymer is observed[3].

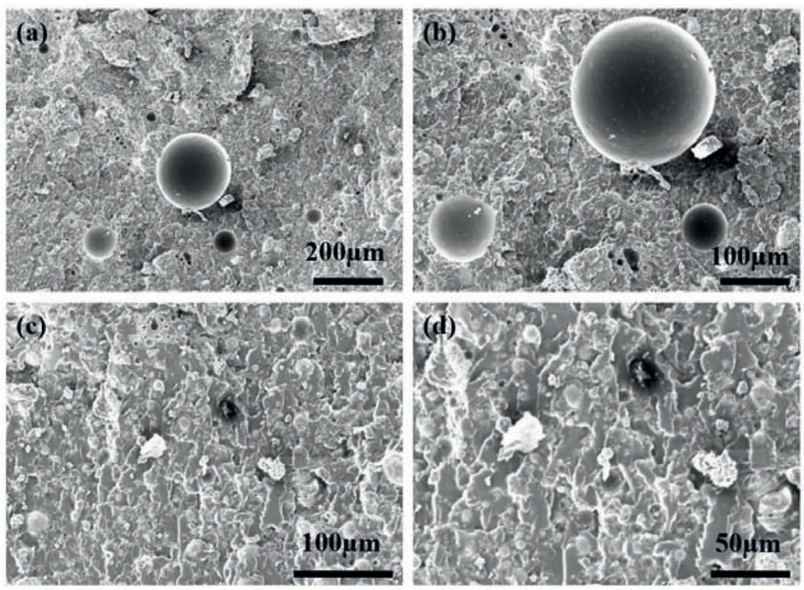

Fig. 5.(a) low and (b-d) high magnified SEM images of the fly ash based hybrid polymer composite.

Dielectric and Electrical Analysis.

Dielectric constant is important parameter to design the electrical insulating sheet and it the ratio of the capacitance formed by two plates with a material between them to the capacitance of the same plates with air as the dielectric. Dissipation factor has also important role to fabricate high performance electrical insulating sheet and electronic components and it is equivalent to the ratio of current dissipated into heat to the current transmitted. Dielectric constant (ε′) and dissipation factor (tan δ) for fly ash powder was measured in the wide frequencies range of 20 Hz –2 MHz at RT and the obtained results are shown in Fig. 6(a) and (b), respectively.

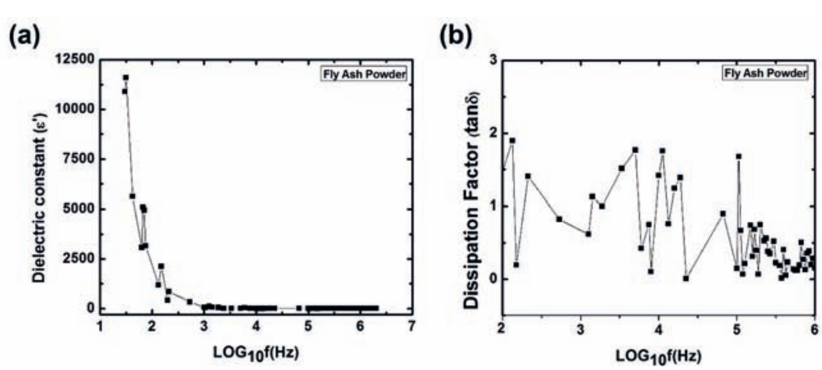

Fig. 6 Variation of (a) Dielectric constant and (b) dissipation factor with frequency for fly ash sample

Fly ash waste powder sample exhibited a very high dielectric constant of about ~10900 at RT at lower frequency region (29 Hz) which attains a low constant value of 6.16 at higher frequencies side (2 MHz) (Fig. 6 (a)). The observed dielectric constant showed that fly ash may be used as capacitor for energy storage, however to fabricate the electrical insulating sheet such high value may add capacitance effect which is not desirable for fabricating electrical insulating sheet. S. C. Raghavendra et. al. have reported that fly ash has a dielectric constant of order 10^4.[22] The dielectric constant (ε′) of the materials was calculated by the relation

$$\varepsilon' = \frac{C_p \, x \, t}{\varepsilon_0 \, x \, A} \qquad (1)$$

Where, ε_0 is permittivity of free space, A is the cross sectional area, t is the thickness, and C_p is the capacitance of the sample [3]. Dissipation factor (tan δ) is given by

$$\text{Tan } \delta = \frac{\varepsilon''}{\varepsilon'} \qquad (2)$$

Where, ε″ is the imaginary part of dielectric constant [23-26]. Fig 6(b) present the variation of dissipation factor of fly ash with applied frequency and a tan d of ~ 1.8 was detected at low frequency side. Dielectric constant, dissipation factor and ac conductivity of the fabricated fly ash polymer composite samples consisting filler concentration varying from 10-60 wt % fly ash powder were measured under same condition in range of 20 Hz to 2 MHz at RT.

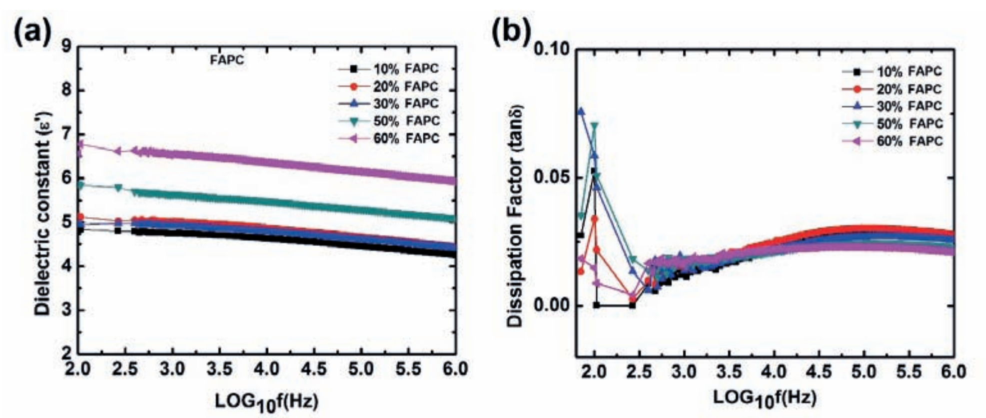

Fig. 7. Variation of (a) dielectric constant and (b) dissipation factor with frequency for fly ash waste polymer composite with various filler concentration.

Fig 7a and 7b show the variation of dielectric constant and tangent loss with applied frequencies of polymer composite samples. As the frequency increases the dielectric constant of all composite sample found to be decreases. It is interesting to note that dielectric constant of fly ash polymer composite samples dramatically decreases from 10900 to 6.69 for 60 % fly ash filler concentration sample. Dielectric constant of fly ash polymer composite with 10, 20, 30, 50 and 60 % fly ash content shows dielectric constant of 4.98, 5.25, 5.00, 5.81, and 6.69 respectively. It is clearly visible that as the fly ash concentration increases in the epoxy resin, the value of dielectric constant increases. However, very minute decrease is detected for 30 % fly ash content polymer composite sample comparative to 20 % fly ash content sample. Such remarkable decrease in the fly ash based composite is due to the low dielectric constant of epoxy resin. Dielectric loss of the polymer composite with various filler concentration was also measured in the same frequency range of 20 Hz to 2MHz. Dielectric loss were also found to be decreases with increase of frequency and found to be very small of about 0.002 for 60 % fly ash based polymer composite sample. The calculated ac conductivity of all fabricated samples and variation of ac conductivity with applied frequency is shown in the Fig 8.

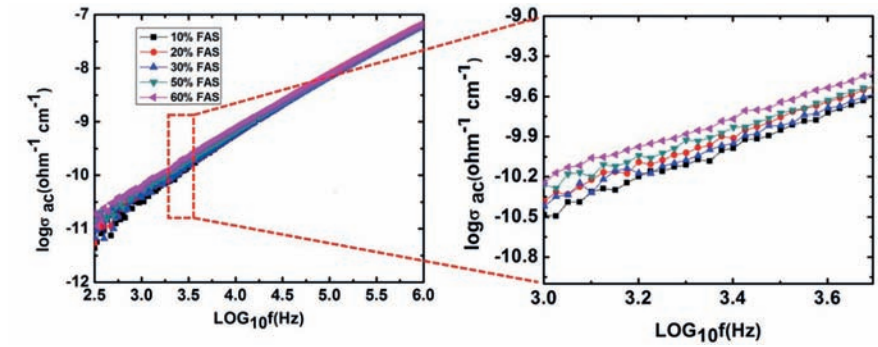

Fig. 8. Variation of ac conductivity with frequency for fly ash waste polymer composite with various filler concentration (right side shows enlarge view of ac conductivity).

AC conductivity increases with increase of the fly ash waste concentration in the polymer composites and ac conductivity has low value of 1.27 x $10^{-14}\Omega^{-1}$ cm^{-1} in low frequency region and high 2.96 x $10^{-10}\Omega^{-1}$ cm^{-1} at higher frequency side (2 MHz) for 60 % fly ash hybrid polymer composite sample.

Surface and Volume Resistivity Studies

Surface and Volume resistivity of the fabricated fly ash based polymer composite samples are measured as per ASTM D257 standard. The samples are prepared in square shape of 100 mm dimension to measure both surface/ volume resistivity. Surface resistivity of the fly ash based polymer composite samples was found to in range of 10^{13} ohms/sq (Fig 9a).

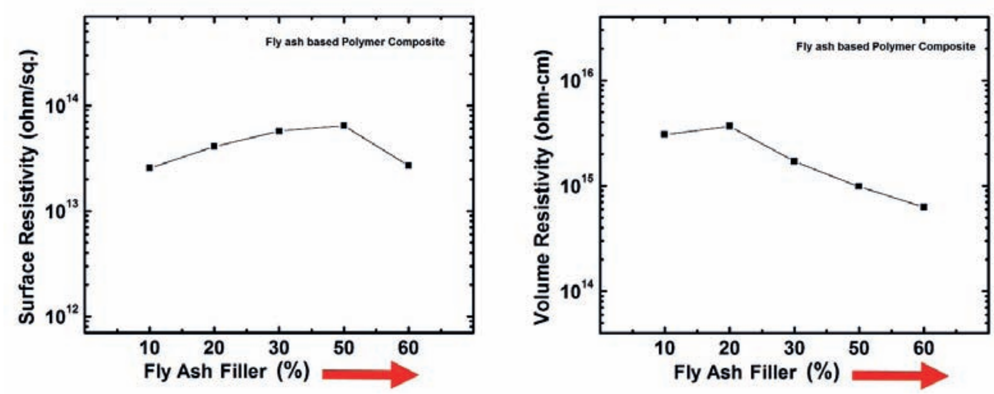

Fig.9 (a) Variation of surface and (b) volume resistivity of fly ash waste polymer composite with various filler concentration.

The results indicate that surface resistivity increases with increase of filler concentration till 50 % filler content and decrease to 10^{13} for 60 % fly ash amount. Further, volume resistivity of the fly ash polymer composite samples was found in range of 10^{14} to 10^{15} ohms-cm for the filer concentration of fly ash from 10, 20, 30, 50 and 60% in polymer composites (Fig 9b). It is observed that volume resistivity slightly decreases with increase of filler concentration (Figure 8b). The observed high value of surface and volume resistivity of fabricated polymer composite samples shows their suitability for electrical insulation application. Roy E. Bickelhaupt et al has reported that ash producing by plants may have resistivity greater than 10^{12} ohm-cm [27]. Osarenmwinda J.O. et al. have observed that composite materials prepared using sawdust:palm kernel shell has insulation resistance of 490MΩ [28].To the best of our knowledge such polymer composite with high value of surface and volume resistivity are not reported till date. The higher resistivity is basically attributed due to low water absorption and weak interfacial polarization between polymer and fly ash particles.

THERMAL CONDUCTIVITY ANALYSIS

Thermal conductivity of the developed composites sheet with fly ash filler loading up to 60 % were evaluated and measured. Thermal conductivity set up based on hot wire method using KEM QTM-710, Japan is used. The obtained thermal conductivity of the polymer composites samples having filler concentration of 10, 20, 30, 50 and 60 % fly ash exhibited thermal conductivity of 0.1765, 0.278, 0.3164, 0.3925 and 0.4387 W/mK (Fig 10).

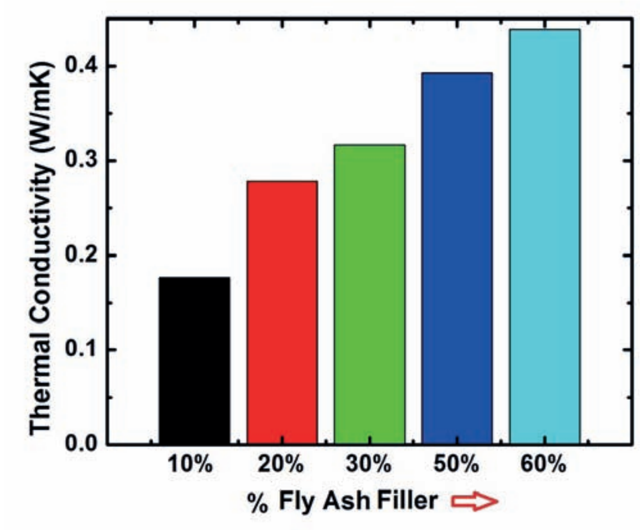

Fig. 10. Thermal conductivity of the fly ash polymer composite sample.

As the filler increases in the epoxy resin, the thermal conductivity increases and reached a maximum value of 0.4387 for 60 % filler condition. We found the spherical shape of fly ash create a path way in epoxy resin matrix, resulting slight enhancement of the thermal conductivity polymer composites. The low value of thermal conductivity enable the product to use in thermal insulating materials specially it reduces the high heat flux and release of high heat from electronic component in micro-electronic technology cause by the integration of the electronic components.

CONCLUSIONS

In summary, this study disclose a facile method for developing environmentally friendly industrial inorganics fly ash based moisture resistant electrical insulating hybrid polymer composite using compressive moulding techniques. Very high dielectric constant of 10900 at low frequency and high dissipation loss factor at RT were measured of fly ash waste powder. Various fly ash polymer composite with filler concentration of 10, 20, 30, 50 and 60 % wt ratio were fabricated. Very low water absorption of 0.14 % was observed from developed sheet. Effect of filler concentration on density, water absorption, dielectric constant, surface/volume resistivity were analyzed in details. Dielectric constant of fly ash based hybrid polymer composites were found to be decreased with respect to applied frequency and increases with fly ash waste powder. Surface resistivity about 10^{13} ohms/sq and volume resistivity in the range of 10^{14} to 10^{15} ohms-cm were measured from the fly ash based polymer composite samples. Low thermal conductivity of the fabricated composite was obtained and slight increase in the thermal conductivity was observed with increase in the filler loading. The high resistivity is discussed in the light of low water absorption and interfacial polarization between polymer and fly ash particles. Our work not just only demonstrates a promising way to utilize industrial waste to fabricate electrical insulating sheet but also greatly broadens the eco-friendly recycling method of industrial waste.

ACKNOWLEDGEMENTS

Authors are very thankful to the Dr. A. K. Srivastava, Director, CSIR-AMPRI, Bhopal for giving permission to publish the work. Authors are thankful to Mr. Ravi Patidar, Mr. Ashwin and Mr. Deepak Kashyap for collecting the raw materials and SEM measurements. Dr. M. K Gupta is grateful to CSIR, India for granting MLP -116 and MLP-115 projects.

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