Indian Journal of Pure & Applied Physics
Vol. 60, June 2022, pp. 521-528

Dielectric Analysis of Single Walled Carbon Nanotubes Loaded SMC Composites Depending on Resin Type

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Received 28 February 2022; accepted 28 May 2022

This paper investigates effect of different industrial resins including vinyl ester and unsaturated polyester produced by sheet moulding compound (SMC) method on conductivity and dielectric properties of single walled carbon nanotubes (SWCNT) modified glass fiber reinforced polymer composites (GFRP), comparatively. Alternating current (AC) conductivities, complex dielectric permittivity, dissipation factor (DF), electric modulus of unsaturated polyester and vinyl ester resin-based composites was examined by impedance analyzer depending on frequency range from 10^{-2} Hz to 10^{7} Hz at room temperature. Cole–Cole approach was used to describe the impedance characteristics of tested samples. In order to clearly understand the effect of resin type, the glass fiber and carbon nanotube filling ratios in the produced materials were kept constant. AC conductivity results showed that VE composite exhibited better performance than UP composite in all frequency. The theoretical models including Power Law and Linear Regression (LR) algorithm also confirmed the experimental results about the conductivity of the composites. A comparison of the dielectric characteristics between SWCNT/VE and SWCNT/UP binary composites indicated that VE based composite exhibited superior dielectric properties compared to the UP based sample. In addition, scanning electron microscopy (SEM) images have been taken to observe distribution and organization of fillers in polymer composites.

Keywords: Single walled carbon nanotube; Unsaturated polyester; Vinylester; Conductivity; Dielectric permittivity; Electric modulus

Introduction

In recent years, the importance of composite materials used in the industry has been increasing with the development of composites with different matrices and reinforcing elements with desired properties[1]. The development, production, determination of material properties and application of composites with nano-sized reinforcement particles to different fields such as industry, automotive and aviation has made composites the focus of attention[2]. The choice of suitable resin type provides additional characteristic features to composite such as good strength, resistance to chemicals, flammability and improved electrical properties[3]. Understanding the relationship and differences between resins gives us the chance to use the power of creation for new applications while giving us clues to improve the efficiency of glass fiber reinforced polymer composites. The unsaturated polyester (UP) resins are versatile, low cost, ease to manufacture and have the ability to be modified during polymer chain formation. These features make it possible for the composite industry to have unlimited use in virtually all areas. Epoxy-based vinylester (VE) resin has relatively high mechanical and chemical properties (acidic and basic environment) with high corrosion resistance but its cost is higher than UP resin.

The adding of nanoscale particles at into these different types of resins has recently become an attractive topic for creating functional composites with improved properties as compared to conventional polymer composites[4]. It is well known that by adding carbon nanotubes to composite materials at low concentrations, the electrical conductivity of the polymer increases extraordinarily and even its conductivity reaches semiconductor levels in many applications such as electrostatic dissipation or electromagnetic radiation shielding[5]. The number of studies on the use of carbon nanotubes (CNTs) as a conductive additive in unsaturated polyester (UP) and vinyl ester (VE) based GFRP composites has increased considerably in the last two decades [6,7]. Lima *et al.*[8] stated that the electrical conductivity of polymer composites based on polyester at 1 wt. % loading rate of MWCNTs was 5.10^{-5} S/cm. Aviles *et al.*[9] obtained conductivity of

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10^{-1} S/m in 0.5 wt. % of multi walled carbon nanotubes-doped vinylester-based polymer composite. Samankan *et al.*[3] have investigated the effect of resin type on the properties of nanocomposites reinforced by 0.5 wt. % of multi walled carbon nanotubes.

The knowledge of dielectric characteristic also ensures beneficial information about properties of the composite for electronic applications. It is known that permittivity in propagation of electromagnetic waves in media with different dielectric properties is very important. Similarly, understanding the dielectric properties of the material is crucial for remote sensing systems in heterogeneous environments[10]. It has been reported that various resin types exhibit different dielectric behaviors in the literature. Chang *et al.*[11] achieved high dielectric constant and low dielectric losses in the epoxy-based MWCNT filled composites produced using microwave curing and thermal curing methods. The dielectric characteristics of multi-walled carbon nanotubes modified polyester resin based composite were investigated by Seng *et al.*[12] and they proved that MWCNTs filled polyester based composites with high dielectric loss were good candidates for EMI shielding materials. Belhimria *et al.* used electric modulus formalism to analyze Maxwell–Wagner–Sillars relaxation in carbon nanotubes/graphite/polyester ternary composites[13].

The effects of the resin type on the AC conductivity, complex dielectric permittivity, loss tangent and complex electric modulus characteristics of SWCNTs filled UP and VE resin based composites were analyzed depending on the frequency comparatively. In addition, the AC conductivity performance of the samples was fitted to a theoretical models power law and linear regression algorithm. SEM micrograph was also used to examine the dispersion states of the components and fillers both materials.

1 Material

Two types of polymer composites with wt. 1.2 % single walled carbon nanotubes (SWCNTs) have been prepared using sheet moulding compound (SMC) method. SMC is a ready-made molding compound containing reinforcing materials, resin, fillers, chemical thickeners, catalysts, mold release agents and components that extend shelf life. Fig. 1 shows production flow chart SWCNT reinforced SMC. Except for the glass fiber reinforcement, all SMC inputs in liquid and powder form were mixed in the mixer continuously or intermittently at the beginning of the production phase. The composition took the form of a paste. The obtained paste was transported onto non-stick polyethylene carrier foils. Glass fibres were cropped as homogeneously as possible on the dough laid on the foils. In order to ensure that the fibre material is more embedded and integrated into the dough, extra dough is laid on this dough. After that, dough was sent to the compression rollers so that there was no space between them. In order to relax dough and reach the ideal viscosity value, it was left in the oven for at least 2 days and kept in the mould which was heated ranging from 420 to 470 K by hot press method. A moulding time of 1.5 minutes was applied for every millimeter of material held at 160 kg / cm^2 mould pressure.

In order to clearly determine the effect of the resin type, the amount of other components in the manufactured composites was almost kept constant. Materials contain wt. 9.1 % additive, wt. 1.7% styrene, wt. 0.7% peroxide, wt. 1.8% zinc sulfide, wt. 31.2% calcium carbonate, wt. 0.9% thickening admixture, and wt. 27.2% glass fiber. The glass fiber FWR6 of 12.56 μm in diameter used for composite production was supplied from Sisecam Turkey. The unsaturated polyester resin 3417V and vinyl ester resin 701 were supplied from Poliya Polyester Turkey and SWCNT was obtained from OCSiAl. Both

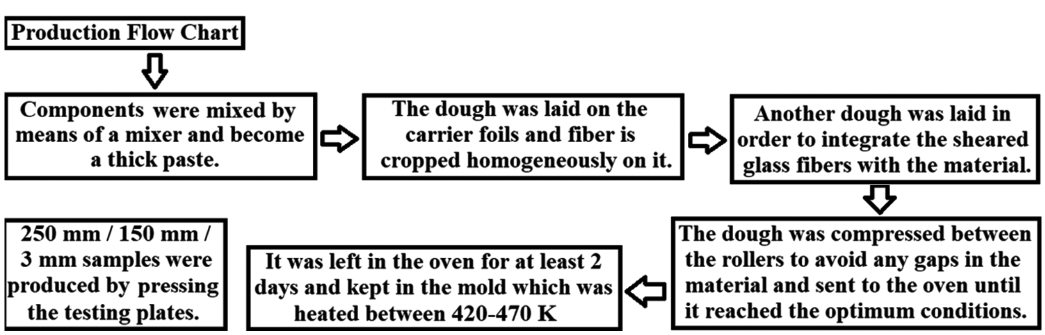

Fig. 1 — Production flow chart SWCNT reinforced SMC.

unsaturated polyester and vinyl ester were added to the mix wt. 27.3%.

2 Dielectric Analysis

A dielectric material consists of atoms or molecules that have one or more of the fundamental electrical polarizations. These mechanisms are electronic, atomic, dipole and interface polarizations that affect the dielectric behaviors of composite material[14]. A force is applied to the nucleus in the direction of the field and the electrons are repulsed in the opposite direction under an applied electric field. The electronic polarization occurs due to the opposite displacement of negative electrons and positive nuclei in the same atom[15]. The electronic polarization is dominant mechanism on dielectric properties of the material in the low frequency region. The effect of electronic polarization weakens with increasing frequency and disappears completely.

When the electron distribution in the molecules is not symmetrical, dipole moment caused by the deviations of these ions from their orbits is called atomic polarization. Atomic polarization is relatively weak and generally stable in the microwave frequency region similar to electronic polarization[16]. Dipole polarization arises from the orientations of molecular dipoles in the direction of the field when an electric field is applied[17]. The fillers, catalyst, additives or impurities migrates to the interfaces of polymer phase under electric field. These carriers are trapped at these regions. The source of interfacial polarization and dielectric relaxation is alignment of charge dipoles that create limited movement of charges.

The dielectric analysis of the samples is a process that includes frequency dependent capacitance C(f) and resistance R(f) measurements. The results obtained from these measurements are used to evaluate complex dielectric permittivity of the samples. The frequency dependent relative dielectric permittivity of the composite material is a complex formula consisting of two parts and is expressed as follows[15]:

$$\varepsilon^*(f) = \varepsilon'(f) - j\varepsilon''(f) \qquad \dots (1)$$

where $\varepsilon'(f)$ shows the real part of the dielectric permittivity and it is called dielectric constant of the material. It denotes the measure of the material's ability to polarize or store charge under an electric field. $\varepsilon''(f)$ represents the imaginary part and it is known loss factor. The dielectric loss factor describes the energy loss due to ionic conduction or polarization[18]. The real and imaginary parts of complex permittivity are described using the following formulas[13]:

$$\varepsilon'(f) = \frac{t}{A}\frac{1}{\varepsilon_0}C(f) \qquad \dots (2)$$

$$\varepsilon''(f) = \frac{t}{A}\frac{1}{\varepsilon_0}\frac{1}{\omega R(f)} \qquad \dots (3)$$

where t is sample thickness, A is area of the metal plates and ε_o is the permittivity of air (8.85×10^{-12} F/m). Plots of the real and imaginary parts of the complex permittivity of the SWCNT/UP and SWCNT/VE binary composites are shown in Fig. 2 (a,b). The polarization caused by the interfacial charges trapped between the SWCNT and the matrices via the Maxwell-Wagner-Sillars mechanism are thought to be the responsible mechanism for the high values of the dielectric constants at low

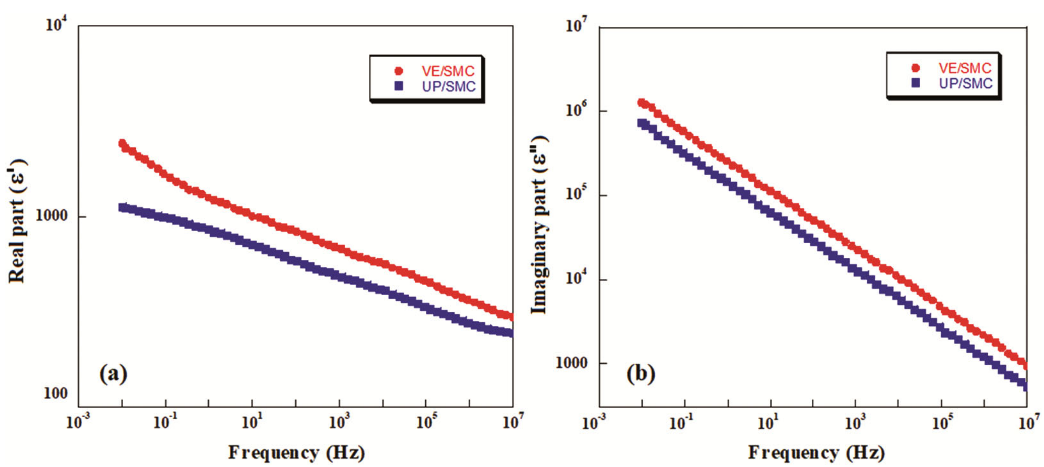

Fig. 2 — (a) Real part and (b) imaginary part of complex dielectric permittivity at room temperature.

frequency[19]. It is predicted that high dielectric permittivity values can be obtained even at low density SWCNT loadings that improve interfacial polarizations due to their high surface area and high aspect ratio. As the frequency increases, the polarization effect gradually decreases. However, micro capacitors become conductive due to increasing frequency, which reduces the dielectric constant. It can be seen that the dielectric constant of the vinylester resin composite is greater than that of the polyester resin composite for all frequency values. This difference can be explained that the better spatial distribution and the orientation of fillers in vinylester resin have produced more micro capacitors[11]. Another reason is the weak interaction between the polyester-based matrix and the surface of the carbon nanotube, as mentioned in the literature[20]. Similarly, imaginary part or dielectric losses are higher in the vinylester-based sample due to its high conductivity.

The loss tangent (tanδ) or dissipation factor (DF) is used to evaluate the magnitude of the amount of energy released as heat in the material. Loss tangent can be expressed as the ratio of the imaginary part of the complex dielectric constant to the real part[21].

$$\tan(\delta) = DF = \frac{\varepsilon''}{\varepsilon'} \qquad \ldots (4)$$

A comparison of the loss tangent characteristics between UP and VE based nanocomposites was shown in Fig. 3. The DF behaviors of the samples are very similar for both samples. Loss tangent values are relatively higher at low frequencies due to poor particle-resin bonding, impurities and material defects. These charge carriers are dragged or displaced in the material even under a small applied field and this situation is the main reason for loss tangent. It is seen that the materials show relaxation peaks at low frequencies. Polymer composites with high dielectric constant and tan delta value in the low frequency region (radio frequency region) can be used for charge storage devices, capacitors and electromagnetic shielding.

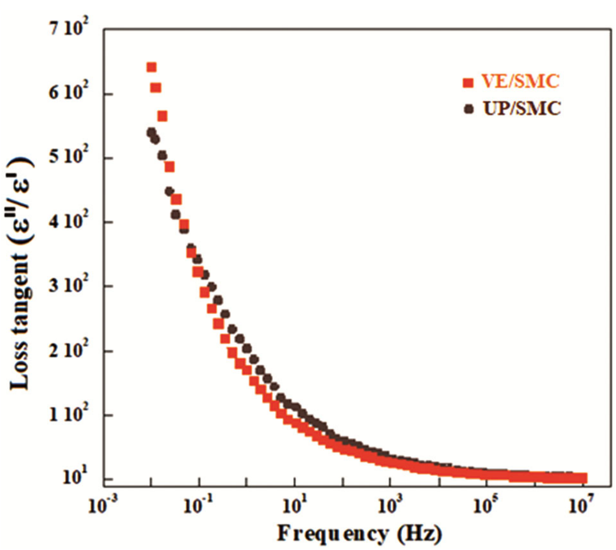

Fig. 3 — Loss tangents versus frequency at room temperature.

In addition to the dielectric analysis, the electrical responses of the samples are investigated by electrical modulus analysis. The electrical modulus analysis has been used in various studies to analyze the relaxation processes occurring in the dielectric responses of polymeric composite materials modified with conductive reinforcements such as carbon nanotubes[22,23]. Interfacial relaxation is suppressed by dielectric permittivity and conductivity in composites reinforced with a conductive component[24]. Especially in such cases, electric modulus formalism is used[25]. The inverse of the complex dielectric permittivity is described as the electrical modulus[26]:

$$M^* = \frac{1}{\varepsilon^*} = \frac{1}{\varepsilon' - j\varepsilon''} = M' + jM'' \qquad \ldots (5)$$

where M' and M" are the real and imaginary parts of the electric modulus, respectively. M' and M" are calculated using the values of ε ′ and ε ″ with the following formulas[27]:

$$M' = \frac{\varepsilon'}{[(\varepsilon')^2 + (\varepsilon'')^2]},\; M'' = \frac{\varepsilon''}{[(\varepsilon')^2 + (\varepsilon'')^2]} \qquad \ldots (6)$$

Figure 4 (a,b) shows real and imaginary electric modulus distributions of VE and UP based SWCNT reinforced composites. As seen in the Fig. 4 (a), the parameter M' is almost zero up to 10 kHz frequency. In this region, the value of M' seems to be independent of frequency. This indicates that the contribution of electrode polarization to M' can be neglected[28]. As the frequency increases, the M' also increases and starts to saturate at approximately 1 MHz frequency. This situation shows that the conduction mechanism includes short-range mobility of charge carriers. In addition, Yadav *et al.*[29] revealed that there is no restoring force that ensures the mobility of the charge carriers with the alternating field.

The imaginary part of electric modulus increases with frequency for both materials as illustrated in

Fig. 4 (b). It is shown that sharp M" peaks of samples form towards higher frequencies. The shape of graph is an asymmetrical situation created by interaction between fillers and matrix. This shows that the dielectric response is non Debye response.

The investigated relaxation process is thought to be due to the mechanism created by two different polarization types. The first of these mechanisms is polarization resulting from the dielectric nature of the material as a result of the reorientation of the dipole groups. Maxwell-Wagner-Sillars (MWS) polarization originated from the interface charges trapped between the SWCNT and the matrix is the other responsible mechanism.

Figure 5 shows the Cole-Cole plot of VE/SMC (a) and UP/SMC (b) specimens in the Argand plane. It is seen that both samples cannot form the semicircle that is shape characteristic of Cole-Cole graphs. The experimental data start from the origin indicates that the relaxation process caused by Maxwell-Wagner-Sillars (MWS) interaction dominates at low frequencies for both tested samples. The fact that the experimental shape is far from ideal semicircle behavior is explained by the skewness in the motion of the dipoles, which are activated to produce dipole polarization in a non-ideal environment[30]. However, especially VE/SMC sample tends to complete the semicircle in the frequency range of the experimental measurements. This implies that the VE/SMC sample has less viscous medium than the UP sample.

3 AC Conductivity Analysis

AC conductivity measurements were performed using the MFIA Impedance Analyzer in the range of 10^{-2} Hz-10^{7} Hz. The parallel metal plates with 22 mm length, 8 mm width and 3 mm thickness were used and polymer composites were placed between them. A sinusoidal AC voltage was applied across the samples at different frequencies. A voltage with amplitude of 1 V has been applied at room temperature. AC electrical conductivities (σ_{AC}) of specimens are calculated from complex impedance (Z^*) values using following formula[31]

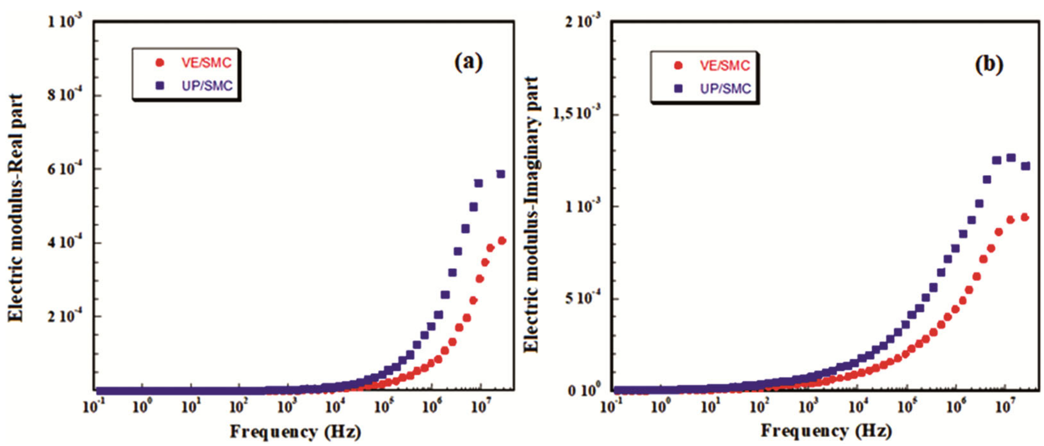

Fig. 4 — Real part (a) and imaginary part (b) of complex electrical modulus.

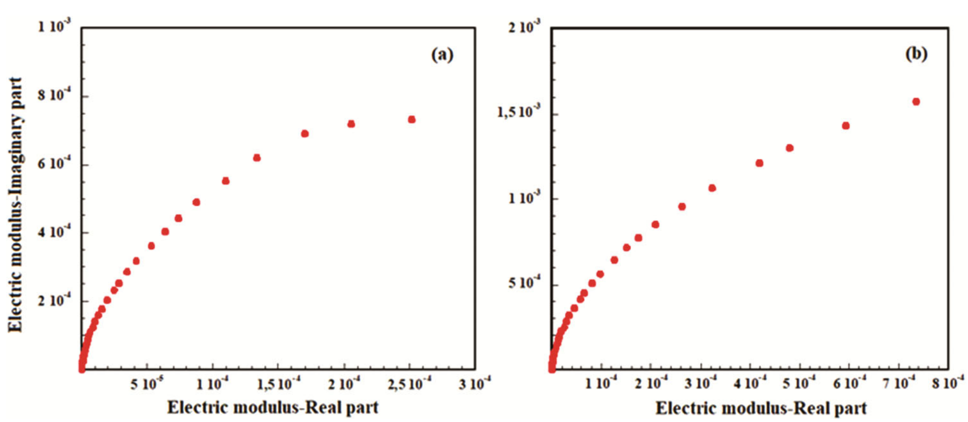

Fig. 5 — Cole-Cole diagrams of VE/SMC (a) and UP/SMC (b) tested samples.

$$\sigma_{AC} = \frac{1}{|Z^*|}\frac{t}{A} \quad \ldots (7)$$

The experimental and theoretical AC conductivities for unsaturated polyester and vinylester thermosetting resin based samples were plotted in Fig. 6. The calculated values show that the percolation threshold was exceeded for both samples and the conductivity values of the SWCNT doped materials increased to the semiconductor level. Although the slopes of the variation of the conductivity with frequency are different, it is seen that it increases linearly with frequency. It is shown that VE/SMC showed higher conductivity than UP/SMC in all frequency range. As a result of the chemical interaction of single walled carbon nanotubes with polyester resin, the formation of a sheath-like polymer layer occur around the SWCNTs, which creates high contact resistance at nanotube junctions. This chemical interaction between the amine functional groups of the nanotubes and the polyester resin may be responsible for the lower conductivity compared to the vinylester-based sample. It is predicted that the increase in conductivity at high frequencies for both samples is the hopping mechanism caused by the increased electric field mentioned in the literature[32]. The variation of alternating current (AC) conductivities of materials with frequency obeys to characteristic power law behavior described by Jonscher[33]. It can be written as

$$\sigma_{AC} = k.f^{s} \quad \ldots (8)$$

where k is constant, f is frequency and s is the frequency dependent exponent which is typically in the range of 0-1[34].

In this study, linear regression model have been also applied for the prediction of alternating current conductivity dependent on frequency for SWCNT filled vinylester and unsaturated polyester based polymer composites in addition to power law. The most basic linear regression model is a model created with a simple linear combination of input variables. More generally, the Linear Regression algorithm aims to model the probability distribution $(x|t)$, which can find target values corresponding to new input data[35].

$$y = \alpha_i + \sum_{i=1}^{N} \beta_i \varphi_i\,(x) \quad \ldots (9)$$

Here, α_i is the linear function constants, β_i is slope of the regression line and φ_i (x) is the basis functions of the model. The conductivity performance comparisons of the samples were made using the regression coefficient (R^2). Conductivity test data in the frequency range of 10^{-2} to 10^{7} Hz by experimental impedance spectroscopy method were used as input data for both samples. As seen in Fig. 6, the power law and linear regression algorithm were applied to the experimental conductivity data, comparatively. It can be concluded from graph that the experimental data of both samples almost obey the power law characteristics. The constant and exponents for power law method are given in Table 1.

The estimation results for UP/SMC material obtained with the linear regression model are in good agreement with the experimental results at low and mid-frequency values. However, there is some inconsistency especially in the extreme values of the frequency. Although the estimation results for VE/SMC were consistent with the experimental results at low frequencies, they did not provide the desired consistency towards to higher frequencies. This is also evident from the regression coefficient. The parameter R^2 that measures the accuracy of the model was found to be 0.65 for VE-based material and 0.69 for UP-based material.

We have compared two methods proposed to predict the alternating current-frequency behavior of

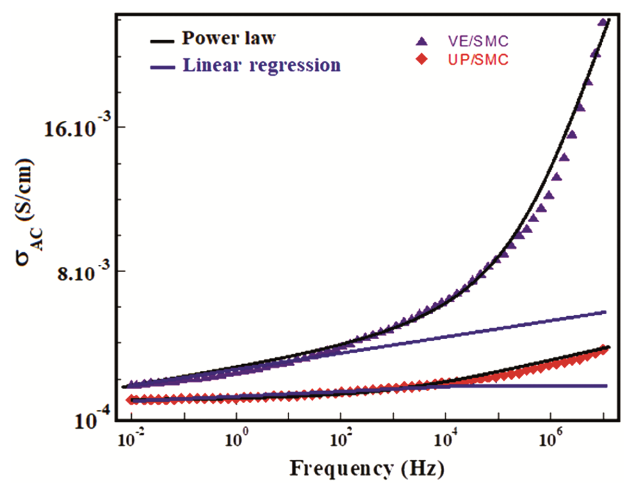

Fig. 6 — Variation of AC conductivities with the frequency for the specimens

Table 1 — Theoretical fit parameters applied to the experimental results.

Material	Constant (k)	Exponent (s)	Regression coefficient (R^2)
VE/SMC	$9{,}5.10^{-4}$	0,07	0,94
UP/SMC	$2{,}4.10^{-3}$	0,11	0,93

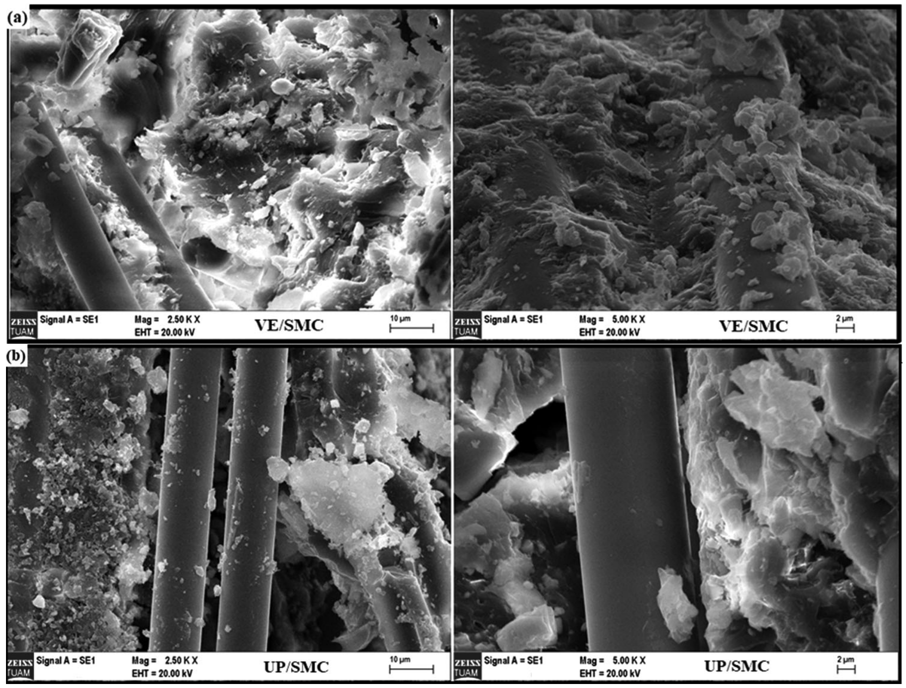

Fig. 7 — SEM images for the samples (a) VE/SMC, (b) UP/SMC.

composites. Results showed that the considered LR model is inappropriate to describe the AC conductivity-frequency relationship in our composites. The power law method gives more reliable and appropriate results for our produced samples.

4 SEM Analysis

Scanning electron microscope (SEM) images of vinylester and polyester resins based specimens are shown in Fig. 7. SEM results revealed that the components in the matrix showed non-homogeneous dispersion. The interaction of SWCNTs and components with the surrounding matrix is extremely critical for their mechanical strength. However, since this study was focused only on electrical properties of materials, the effect of the dispersion on electrical behaviors of the samples was considered. Occasional voids, matrices stacked on the glass fibers, and poor regional interface properties are the factors that disrupt the homogeneous distribution and lead poor dispersion within specimens. SWCNTs are embedded in the polymer matrix and formed agglomerations in certain regions. Although this situation negatively affects the mechanical properties, the randomly agglomerations of carbon nanotubes in certain regions of resin, especially in the vinylester-based sample, is seen as a plus in terms of conductivity mechanisms.

5 Conclusions

In this study, effect of SWCNTs on electrical properties of unsaturated polyester and vinylester thermosetting resins based composites was analyzed in terms of conductivity and dielectric properties. It is observed that with the addition of SWCNTs to the resins, both composites exceed the percolation threshold and become conductive with increasing number of nanoparticle in the matrices.

It is found from comparison of the AC conductivity versus frequency characteristics that the conductivity of the UP/SMC has lower conductivity compared to the vinylester-based sample. It is concluded that the effect of cross-links and high contact resistance at nanotube junctions in polyester, which is thought to be the cause of this situation, should be considered in further studies. The frequency dependence of the AC conductivity of the materials is also examined via theoretical models including power law and linear regression algorithm. It was found that the linear regression method could not be applied to our fabricated samples and the AC conductivities of the samples largely obeyed the power law method.

The variations of dielectric properties with frequency were plotted for both samples. It was observed that dielectric characteristics are high at low frequency regions and decreases towards high frequencies. It can be said that this situation is caused by polarization effects, which is the dominant mechanism in the matrices of composites at low frequencies. It is revealed from the impedance analysis results that the dielectric performance of VE/SMC are better than UP/SMC. The difference between VE and UP can be explained by the better spatial distribution and orientation of the fillers in the vinylester resin produces more microcapacitors. Consequently, the selection of the appropriate resin system is a critical parameter for producing a composite to be used for specific applications. By adding small percentages of SWCNTs into the polymeric matrices, improving the conductivity as well as the real and imaginary permittivity values showed that produced materials can be candidate for applications such as charge storage devices, electromagnetic interference (EMI) shielding.

Electric modulus analysis showed that electrical modulus behavior of the samples is similar and peaks form towards higher frequencies. However electrical response of VE/SMC is higher than that of UP/SMC at all frequencies. The asymmetrical behavior in the electrical response graphs due to chemical interactions between the filling material and the matrix indicates the non-Debye model. Cole-Cole diagrams also indicate that VE/SMC has less viscous medium than the UP sample and its components show a more homogeneous distribution.

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