

THERMAL EXPERIMENTAL ANALYSIS FOR DIELECTRIC CHARACTERIZATION OF HIGH DENSITY POLYETHYLENE NANOCOMPOSITES

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Abstract. *The importance of nanoparticles in controlling physical properties of polymeric nanocomposite materials leads us to study effects of these nanoparticles on electric and dielectric properties of polymers in industry. In this research, the dielectric behaviour of High-Density Polyethylene (HDPE) nanocomposites materials that filled with nanoparticles of clay or fumed silica has been investigated at various frequencies (10 Hz–1 kHz) and temperatures (20–60 °C). Dielectric spectroscopy has been used to characterize ionic conduction, then, the effects of nanoparticles concentration on the dielectric losses and capacitive charge of the new nanocomposites can be stated. Capacitive charge and loss tangent in high density polyethylene nanocomposites are measured by dielectric spectroscopy. Different dielectric behaviour has been observed depending on type and concentration of nanoparticles under variant thermal conditions.*

Keywords

Dielectric properties, high density polyethylene, insulation, nano-composite, nanoparticles, polymers.

1. Introduction

Among the various types of polymeric dielectrics, High-Density PolyEthylene (HDPE) has been standing out as a raw material for the production of insulators, spacers, and also as a coating for cable conductors used in electrical power distribution networks. For this type of application, the dielectric strength is one of the properties that must be taken into account in order to check

the ability to withstand high electric fields. Dielectric strength is defined as a relationship between the breakdown voltage and the dielectric thickness, representing the maximum field which the material can support indefinitely for a specific experimental setup. The use of high purity polymers in engineering applications is technologically not viable. This problem leads to the development of formulations with additives in order to protect the polymers against losses in their properties (for example, mechanical and thermo-mechanical) during the processing stages or in service. These additives used in polymers for electrical insulation may or may not harm the electric properties [1], [2], [3], [4] and [5]. Polymer composite, compared with conventional single-phase insulation, can improve dielectric properties. Non-linear conductivity in the insulating polymer has been achieved by the introduction of inorganic semi-conductor in particulate.

The polymer composite with field dependent conductivity can be used to improve the distribution of electric field. For different application, the different composites are used. For example, the silicon rubber and EPDM filled with non-linear fillers are used as electric stress grading materials in cable joints and terminations, and the epoxy composites filled with carborundum (SiC) are used for grading the electric field distribution at the end of windings in electric machines. The polymer composite filled with non-linear inorganic fillers, as one kind of non-linear dielectrics, can be called "smart insulating materials", due to the function of grading electric field and restraining the formation of space charge. The properties of the non-linear materials are dependent on the basic materials, and also dependent on the fillers. In any engineering application, it is vital that the selected materials exhibit an appropriate combination of properties throughout the design lifetime of the

plant. For polymers, macroscopic properties are determined by two factors; microstructure and composition [6], [7], [8], [9] and [10]. For example, the growth of spherulites may lead to increase or decrease in electrical breakdown strength, depending upon the precise structure of these objects.

The composition of a polymeric material can be varied in many ways, through the addition of antioxidant, plasticizers, crosslinkers, fillers, etc. The addition of inorganic filler to an elastomer will increase its stiffness, albeit at the expense of reduced elongation at break [10], [11], [12], [13] and [14]. As of now, work is underway to examine the physical properties of nanocomposite materials composed of nanoparticles and their compounds stabilized within a polymeric dielectric matrix. In recent years polymer nanocomposites have attracted wide interest with regard to enhancing polymer properties and extending their utility. It has been found that the dielectric properties have a close relationship with the interfacial behaviour between the fillers and the polymer matrix in such composites. The electric and optic properties of these materials have been demonstrated to be highly dependent on the size, structure, and concentration of the nanoparticles, as well as on the type of polymeric matrix [15], [16], [17], [18] and [19].

Great expectations have been focused on effects and importance of costless nanoparticles [20], [21], [22], [23], [24], [25], [26], [27] and [28]. However, it has been concerned in this paper about the effect of types of costless nanoparticles on the electrical properties of a polymeric nanocomposite. With a continual progress in polymer nanocomposites, this research depicts the effects of types and concentration of costless nanoparticles in electrical properties of industrial polymer material. All the experimental results of dielectric spectroscopy have been investigated and discussed to detect all nanoparticles effects on electrical properties of nanocomposite industrial material which fabricated; like High Density PolyEthylene (HDPE) with various nanoparticles of clay and fumed SiO_2 .

2. Experimental Setup

HIOKI 3522-50 LCR Hi-tester device measured electrical parameters of nanocomposite solid dielectric insulation specimens at various frequencies: $|Z|$, $|Y|$, Θ , R_p (DCR), R_s (ESR, DCR), G , X , B , C_p , C_s , L_p , L_s , D ($\tan \delta$), and Q . Specification of LCR is Power supply: 100, 120, 220 or 240 V ($\pm 10\%$) AC (selectable), 50/60 Hz, Frequency: DC, 1 mHz to 100 kHz, Display Screen: LCD with backlight/99999 (full 5 digits), Basic Accuracy: Z : $\pm 0.08\%$ rdg. Θ : $\pm 0.05^\circ$, and External DC bias ± 40 V max.(option)

(3522-50 used alone ± 10 V max./using 9268 ± 40 V max.).

Finally, all dielectric properties for pure and nanocomposite industrial materials can be measured using HIOKI 3522-50 LCR Hi-tester device. Figure 1 shows HIOKI 3522-50 LCR Hi-tester device for measuring characterization of nanocomposite insulation industrial materials.



Fig. 1: HIOKI 3522-50 LCR Hi-tester device.

3. Preparation of Nanocomposites and Characterization

The industrial materials studied here are high density polyethylene which has been formulated utilizing variant concentrations of nanoparticles of clay and fumed silica. High density polyethylene nanocomposites have been prepared and fabricated by using recent nanotechnology procedures and devices for melting pure high density polyethylene grains, mixing and penetrating nanoparticles inside the base matrix HDPE by modern ultrasonic devices. Most of all nanocomposite materials are commercial and available already in the manufacturing of High-Voltage (HV) industrial products and their properties detailed in Tab. 1.

Tab. 1: Electric and dielectric properties of pure and nanocomposite materials.

Materials	Dielectric constant at 1 kHz	Resistivity ($\mu\text{-m}$)
Pure HDPE	2.3	10^{15}
HDPE + 1 wt% clay	2.23	10^{16}
HDPE + 5 wt% clay	1.99	$10^{16} - 10^{19}$
HDPE + 10 wt% clay	1.76	$10^{19} - 10^{21}$
HDPE + 1 wt% SiO_2	2.32	10^{14}
HDPE + 5 wt% SiO_2	2.39	$10^{14} - 10^{12}$
HDPE + 10 wt% SiO_2	2.49	$10^{12} - 10^{10}$

SEM images illustrate penetration of nanoparticles in polymeric nanocomposites; thus, Fig. 2 shows SEM

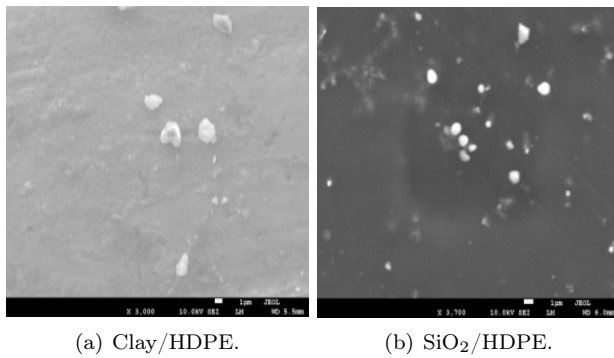


Fig. 2: SEM images for high density polyethylene nanocomposites.

images that illustrate the penetration of cost-fewer nanoparticles in high density polyethylene nanocomposites. It has flakes like morphology with high surface area. Also, it illustrates that the nanoparticles are uniformly dispersed in the polymer matrix.

4. Results and Discussion

Dielectric Spectroscopy is a powerful experimental method to investigate the dynamical behaviour of a sample through the analysis of its frequency-dependent dielectric response. This technique is based on the measurement of the capacitance as a function of frequency of a sample sandwiched between two electrodes. The $\tan \delta$, and capacitance (C) was measured as a function of frequency in the range 10 Hz to 50 kHz at variant temperatures for all the test samples. The measurements were made using high-resolution dielectric spectroscopy.

4.1. Effect of Nanoparticles at 20 °C

Figure 3 shows loss tangent as a function of frequency for clay/HDPE nanocomposites at room temperature (20 °C). This figure illustrates the loss tangent of clay/HDPE nanocomposites increases with increasing clay nanoparticles concentration up to 1 wt%, especially at low frequencies, but it decreases with increasing clay nanoparticles concentration up to 10 wt%. In addition, Fig. 4 shows loss tangent as a function of frequency for (20 °C). The loss tangent of SiO₂/HDPE nanocomposites decreases with increasing fumed silica concentration nanoparticles up to 1 wt%, especially at high frequencies but it increases with increasing fumed silica concentration nanoparticles (1–10 wt%).

Figure 5 shows capacitance as a function of frequency for clay/HDPE nanocomposites at room temperature (20 °C). It is clear that the measured capacitance of clay/HDPE nanocomposites increases with in-

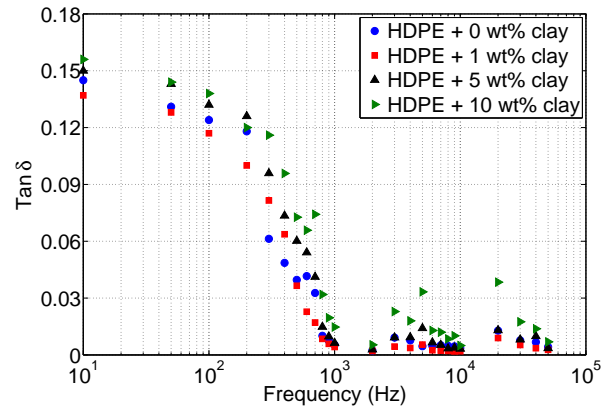


Fig. 3: Measured loss tangent for clay/HDPE nanocomposites at $T = 20$ °C.

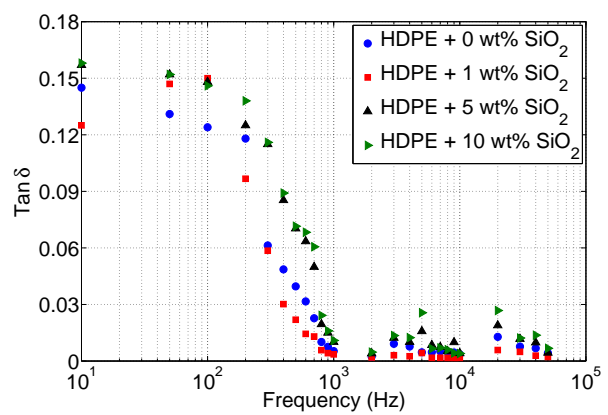


Fig. 4: Measured loss tangent for SiO₂/HDPE nanocomposites at $T = 20$ °C.

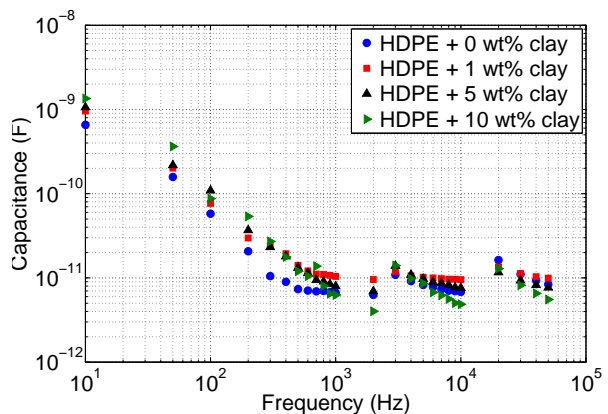


Fig. 5: Measured capacitance clay/HDPE nanocomposites at $T = 20$ °C.

creasing clay concentration nanoparticles up to 10 wt%. On the other hand, Fig. 6 shows capacitance as a function of frequency for SiO₂/HDPE nanocomposites at room temperature (20 °C). Furthermore, the measured capacitance of SiO₂/HDPE nanocomposites increases with increasing fumed silica concentration nanoparticles up to 5 wt% but it decreases with increasing fumed silica concentration nanoparticles up to 10 wt%.

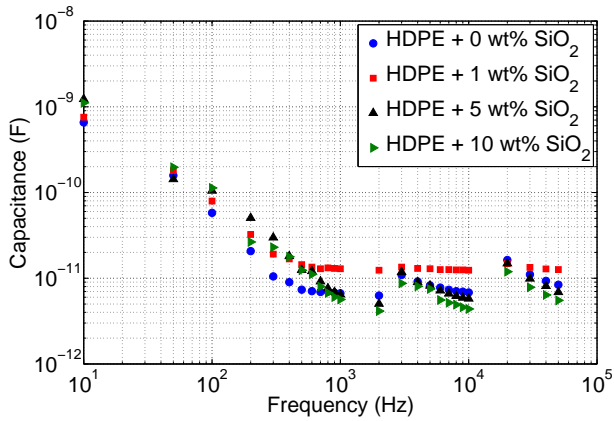


Fig. 6: Measured capacitance SiO₂/HDPE nanocomposites at $T = 20\text{ }^{\circ}\text{C}$.

4.2. Effect of Nanoparticles on HDPE Characterization at 40 °C

Figure 7 shows loss tangent as a function of frequency for clay/HDPE nanocomposites at a temperature (40 °C). The loss tangent of clay/HDPE nanocomposites increases with increasing clay nanoparticles concentration up to 10 wt%, especially at low frequencies. Figure 8 shows loss tangent as a function of frequency for SiO₂/HDPE nanocomposites at a temperature (40 °C), the measured loss tangent of SiO₂/HDPE nanocomposites increases with high fumed silica concentration nanoparticles up to 1 wt%, especially at low frequencies. Noting that the loss tangent of SiO₂/HDPE nanocomposites decreases with increasing fumed silica concentration nanoparticles (1–10 wt%).

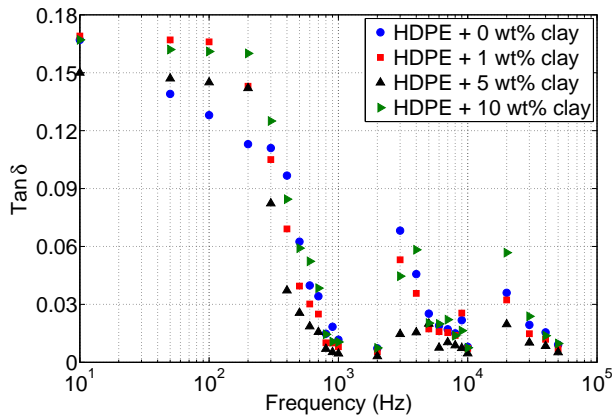


Fig. 7: Measured loss tangent for clay/ HDPE nanocomposites at $T = 40\text{ }^{\circ}\text{C}$.

Figure 9 shows capacitance as a function of frequency for clay/HDPE nanocomposites at a temperature (40 °C). The measured capacitance of clay/HDPE nanocomposites decreases with increasing clay concentration nanoparticles. Similarly, Fig. 10 shows the

measured capacitance of SiO₂/HDPE nanocomposites decreases with increasing fumed silica concentration nanoparticles capacitance as a function of frequency at a temperature (40 °C).

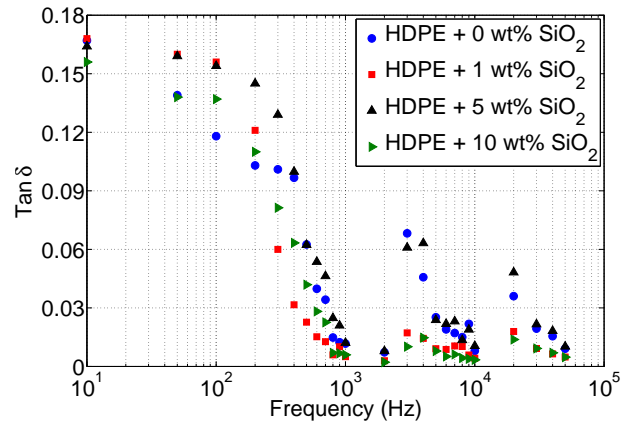


Fig. 8: Measured loss tangent for SiO₂/HDPE nanocomposites at $T = 40\text{ }^{\circ}\text{C}$.

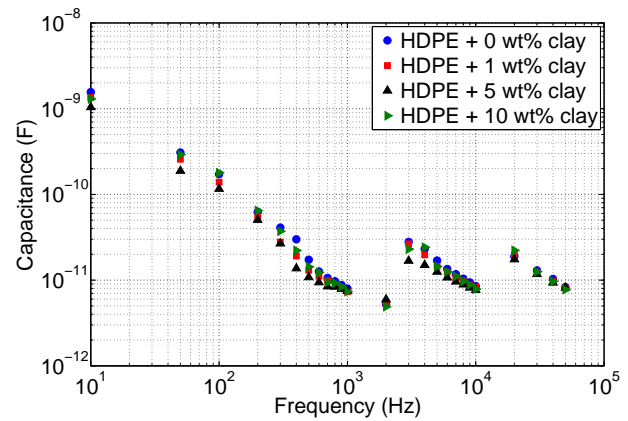


Fig. 9: Measured capacitance for clay/HDPE nanocomposites at $T = 40\text{ }^{\circ}\text{C}$.

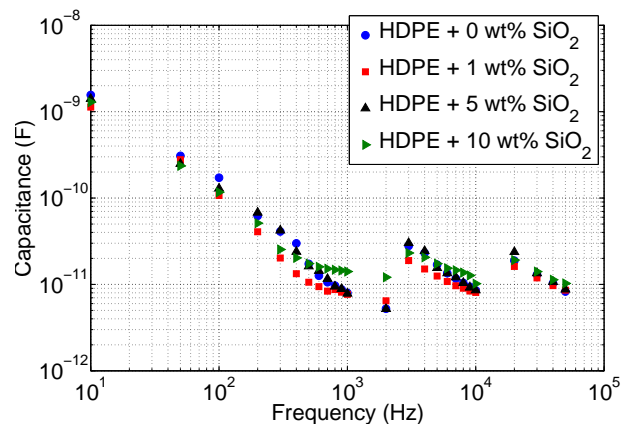


Fig. 10: Measured capacitance for SiO₂/HDPE nanocomposites at $T = 40\text{ }^{\circ}\text{C}$.

4.3. Effect of Nanoparticles at 60 °C

Figure 11 shows loss tangent as a function of frequency for clay/HDPE nanocomposites at a temperature (60 °C). The loss tangent of clay/HDPE nanocomposites decreases with high clay nanoparticles concentration up to 10 wt%, especially at low frequencies. On the other hand, Fig. 12 shows loss tangent as a function of frequency for SiO₂/HDPE nanocomposites at a temperature (60 °C), moreover, it is noticed that the loss tangent of SiO₂/HDPE nanocomposites decreases with high fumed silica concentration nanoparticles up to 10 wt%, specially, at low frequencies.

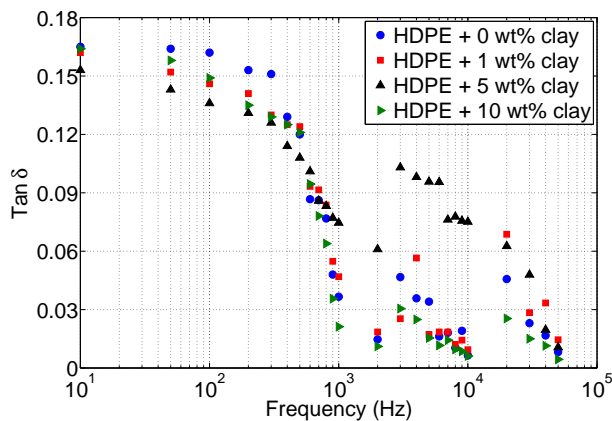


Fig. 11: Measured loss tangent for clay/HDPE nanocomposites at $T = 60$ °C.

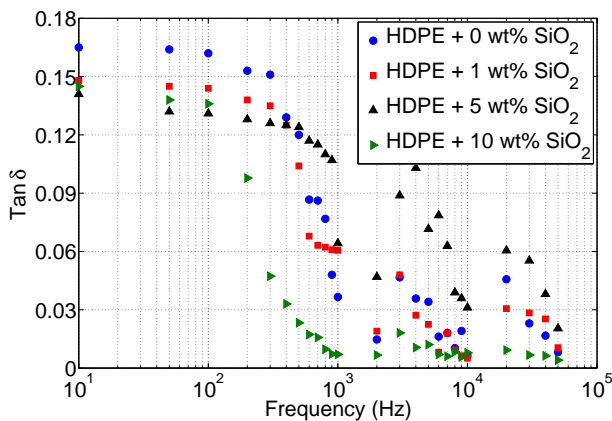


Fig. 12: Measured loss tangent for SiO₂/HDPE nanocomposites at $T = 60$ °C.

Figure 13 shows capacitance as a function of frequency for clay/HDPE nanocomposites at a temperature (60 °C). It illustrates the measured capacitance of clay/HDPE nanocomposites increases with increasing clay concentration nanoparticles up to 10 wt%. On the other hand, Fig. 14 shows capacitance as a function of frequency for SiO₂/HDPE nanocomposites at a temperature (60 °C). Moreover, it is illustrated that the capacitance of SiO₂/HDPE nanocomposites increases

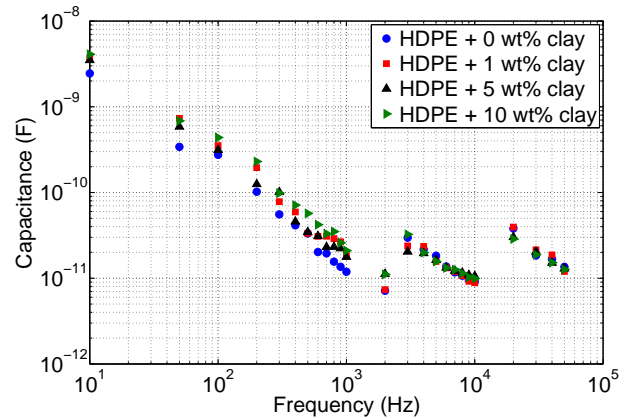


Fig. 13: Measured capacitance for clay/HDPE nanocomposites at $T = 60$ °C.

with high fumed silica concentration nanoparticles up to 1 wt%, in addition, it decreases with the growth of fumed silica concentration nanoparticles (5–10 wt%).

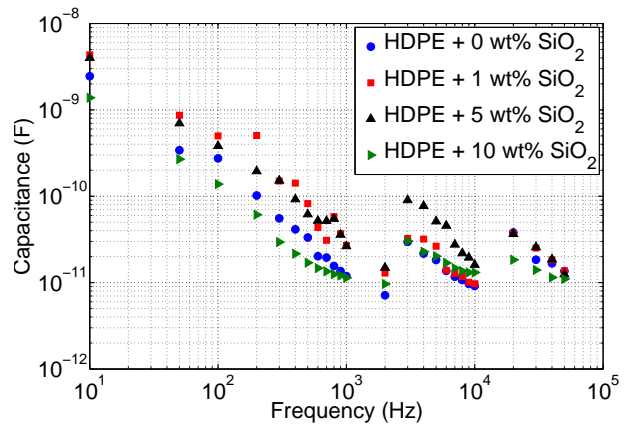


Fig. 14: Measured capacitance for SiO₂/HDPE nanocomposites at $T = 60$ °C.

4.4. Trends of Nanoparticles under Thermal Conditions

All depicted results have cleared that adding fumed silica increases permittivity of High density polyethylene insulation materials; furthermore, adding clay has decreases permittivity of High density polyethylene insulation materials as shown in Tab. 1. Physical interface between high density polyethylene and nanoparticles has been affected on capacitance and dielectric loss angle curves under normal thermal conditions (20 °C) that are pointed out in (Fig. 3, Fig. 4, Fig. 5, Fig. 6). Therefore, the loss tangent of clay/HDPE nanocomposites increases with increasing clay nanoparticles concentration up to 1 wt%, specially, at low frequencies but it decreases with the raise of clay nanoparticles concentration up to 10 wt%. Moreover, the loss tangent of SiO₂/HDPE nanocomposites decreases with fumed sil-

ica concentration nanoparticles raised up to 1 wt%, especially, in case of high frequencies but it increases with increasing fumed silica concentration nanoparticles (1–10 wt%). The measured capacitance of clay/HDPE nanocomposites increases with increasing clay concentration nanoparticles up to 10 wt%. Furthermore, the measured capacitance of SiO₂/HDPE nanocomposites increases with increasing fumed silica concentration nanoparticles up to 5 wt% but it decreases with increasing fumed silica concentration nanoparticles up to 10 wt%.

Changing thermal conditions can be affected by physical interface between high density polyethylene and nanoparticles and so will be affected by capacitance and dielectric loss angle curves as pointed out in (Fig. 7, Fig. 8, Fig. 9 and Fig. 10) for high thermal condition (40 °C). Thus, the loss tangent of clay/HDPE nanocomposites increases with increasing clay nanoparticles concentration up to 10 wt%, specially, at low frequencies. Moreover, the measured loss tangent of SiO₂/HDPE nanocomposites increases with increasing fumed silica concentration nanoparticles up to 1 wt%. Noting that the loss tangent of SiO₂/HDPE nanocomposites decreases with the higher concentration of fumed silica nanoparticles (1–10 wt%). Also, the measured capacitance of clay/HDPE nanocomposites decreases with the addition of clay concentration nanoparticles up to 10 wt%. It is clear that the measured capacitance of SiO₂/HDPE nanocomposites raises with the concentration of the fumed silica nanoparticles up to 1 wt% and also increases with the addition of fumed silica concentration nanoparticles up to 10 wt%.

Finally, the effect of raising thermal conditions up to (60 °C) on physical interface between high density polyethylene and nanoparticles is pointed out in (Fig. 11, Fig. 12, Fig. 13 and Fig. 14) where loss tangent and capacitance of new nanocomposite materials are reported for different concentration weights of modified nanoparticles concentration at (60 °C) temperature. Thus, the loss tangent of clay/HDPE nanocomposites decreases with clay nanoparticles concentration raised up to 10 wt%, especially in case of low frequencies. It is noticed that the loss tangent of SiO₂/HDPE nanocomposites decreases with fumed silica concentration nanoparticles up to 10 wt%, especially in case of low frequencies. The measured capacitance of clay/HDPE nanocomposites increases with the addition of the clay nanoparticles concentration up to 10 wt%. The capacitance of SiO₂/HDPE nanocomposites increases with fumed silica concentration nanoparticles increased up to 1 wt% but it decreases with the addition of fumed silica nanoparticles (5–10 wt%).

5. Conclusion

Adding fumed silica has increased permittivity of the new high density polyethylene nanocomposite materials, but adding clay has decreased permittivity of the new high density polyethylene nanocomposite materials.

Nanoparticles can be controlled in the loss tangent, and capacitance of new high density polyethylene nanocomposites depend on type and concentration of nanoparticles in nanocomposites according to physical interface that created between high density polyethylene and nanoparticles.

Thermal environment is an effective parameter for increasing and decreasing the loss tangent and capacitance of new high density polyethylene nanocomposites with respect to the type and concentration of nanoparticles. Thermal environment conditions are affected by physical interface created between high density polyethylene and nanoparticles because clay nanoparticles are more efficient than fumed silica nanoparticles for decreasing charging capacitance and loss tangent performance under room thermal conditions. However, under the high thermal conditions, the performance of charging capacitance and loss tangent are changed gradually.

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