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ASSESSMENT OF THE DIELECTRIC PROPERTIES OF POLYESTER/METAKAOLIN COMPOSITE

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Abstract

One of the promising and exciting avenues for developing effective and efficient dielectric materials with high energy density and low cost is the recent approach of introducing inorganic nanoparticles into polymer matrices to form dielectric polymer composites, which finds application in the electrical energy storage, electronic and power device productions. Therefore, this study explored the potential of metakaolin (prepared from kaolin sourced from Kankara, Nigeria) in influencing the polyester composite dielectric properties, being a material found to be environmentally friendly and affordable. The dielectric constants and dissipation factors were used to assess the impact of introducing metakaolin in the polyester matrices. A precision LCR meter was used at room temperature, over a frequency range of 20 Hz to 1 MHz. The metakaolin used was found to be composed of 41.54 % Al2O3 and 55.28 % SiO2. The study's outcome indicates that the dielectric constant and dissipation factor increased with the metakaolin loading due to interfacial polarization but was found to decrease with increasing frequency. However, the composite with 10 %wt metakaolin was found to have shown the highest dielectric constant, based on this study, indicating an excellent electrical property, validating its use for devices like capacitors.

Keywords: *Polyester; Metakaolin; Composite; Dielectric; Dissipation factor; Frequency.*

Introduction

A dielectric material is a nonconducting substance, such as an insulator. However, the word "dielectric" is usually employed in considering the effect of alternating electric fields on a material. In contrast, the term "insulator" is most often used to express the material's electrical nature when exposed to withstand a high electric field. Dielectric materials usually find application in control and storage of electrical charges and electrical energies, including developing modern electronics and electric power systems [1,2] motors, and cables [3]. Excellent dielectric materials with high stiffness and thermal stability have traditionally been used in ceramics such as mica, silicon dioxide, and $BaTiO_3$. However, these dielectric materials' applicability for a practical electronic device is mostly impeded by their high-density, poor flexibility, and challenging processing conditions. With these limitations, polymeric based composite has been extensively explored to serve as alternative materials in dielectrics. They can offer tunable dielectric properties, processing advantages with mechanical flexibility, and the capability of molding it into different configurations for electronic devices that have reduced weight and volume [4,5].

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There has been renewed interest in extensively exploring composites that combine service and electrical properties' stability in recent decades. It usually involves a polymer matrix with low cost and ferroelectric ceramics of high dielectric permittivity. The composite’s dielectric constant increase is usually attributed to the inorganic fillers' high dielectric constant since the composite dielectric constant is a volume average of those of the constituents. Although the ferroelectric ceramic materials’ dielectric constant can be very high, its volume fraction in the composite has to be within the percolation threshold. The values for fillers with a random distribution of grain size are typically below the volume fraction of 60% [6–8]. The composite's functional and structural applications have been studied to achieve the integrated phases' enhanced performance to those of the individual components. Besides the interfacial advantage between materials, the matrix's properties, and the filler characteristics, such as geometry, nature, volume ratio, and orientation, can significantly influence the composite's performance [9].

Kaolinite (aluminum silicate hydroxide) is one of the common clay minerals formed by feldspar weathering. It has a chemical makeup of $Al_2Si_2O_5(OH)_4$. It is a layered silicate mineral with one tetrahedral sheet linked through oxygen atoms to one octahedral sheet of alumina octahedral [10]. It is an environmentally benign aluminosilicate mineral insoluble in water [11]. Chemical investigation of Kankara kaolin in Katsina State, Nigeria, has been reported in the literature [12,13]. It has relatively high Al/Si, possessed tetrahedral and hexagonal, euhedral morphology [14]. The target applications of Kankara kaolin were water treatment [15], pharmaceuticals [11], and catalyst support [3–5,16]. Metakaolin is a chemical phase that is produced upon subjecting kaolinite to a thermal treatment process. The temperature range depends on the kaolin (kaolinite with minor impurities) characteristics like particle size and degree of crystallinity.

A further survey of the literature indicates that much work investigates composite development interested in the high dielectric properties. Some of these works include Yan et al. [17], whose studies showed that the addition of $CaTiO_3$ lowered the dielectric loss of $CaCu_3Ti_4O_{12}$ ceramics remarkably, especially at low frequencies, while the giant dielectric constant remained. Vijaya et al. [18] also reveal that using industrial waste to reinforce polypropylene yields more dielectric strength than polyester reinforcement. Another work is the Xu et al. [18] report, which reveals that a rise in the aramid/epoxy composites’ strain results in a rise in the dielectric constant of composites’ dielectric constants increases while the dielectric loss factor or tangent did not display any change. However, the pure epoxy resin and glass fiber/epoxy composite's dielectric properties remain broadly stable when being stretched. The performance of aramid/epoxy composite was identified due to its crystal orientation, unlike others that are amorphous in structure [18].

In recent reports, Wu et al. [19] studies revealed that the introduction of 5 wt% titanium dioxide decorated multi-walled carbon nanotubes (MWCNTs-TiO2) on a polylactide (PLA) nanocomposites via the use of sol-gel approach raised the dielectric constant of the composite by 8.3 times higher than that of pure which increases from 3.2 (i.e., pure PLA) to 26.6, while the displaying a low value (0.2) of dielectric loss remains at 1000 Hz. The findings indicate that the modified composite possesses good insulating layers for the suppression of dielectric loss effectively. Torgut [20] studies further indicated an increase in frequency results in decreased composites’ dielectric constant and loss factor. Further findings from this author indicated no significant change in dielectric constant, and loss factor was recorded for voltage change. The introduction of graphene on Poly(Maleic anhydride-N-isopropyl acrylamide) copolymer via free radical polymerization method yields a composite considerably risen the dielectric constant and loss factor of 244 % and 387 %, respectively [20]. In the study of Acharya & Pradhan [21], the dielectric properties of synthetic and natural fiber reinforced epoxy composites were investigated. The fibers used for reinforcing epoxy with different fiber loadings include bamboo and glass fibers. The studies indicated that the treated fiber composites exhibited lower dielectric properties due to the natural fiber's decreased hydrophilicity. The chopped glass fibers reinforced epoxy composites were found to be the highest dielectric constant among all tested composites samples in the study.

However, this work investigated the effect of metakaolin loading on the dielectric properties of the polyester composite. This study evaluated the use of varying weight percent of metakaolin in a polyester-based composite matrix at 25 ^{o}C, with varying frequencies, while accounting for its dielectric behavior, respectively. Dielectric constant, loss factor, and dissipation factor were used to account for the composites' dielectric behavior.

Materials and Methods

Materials

An unsaturated polyester resin (YUKALAC Type 157 BQTN-EX) with specific gravity and viscosity of 1.10 ± 0.02 and 4.5–5.0 Poise (25°C), respectively, were used in this study. Methyl-ethyl-ketone-peroxide type A (MEPOXE) was employed as a catalyst, while cobalt naphthenate was used as the accelerator in this study. The metakaolin used in this study was obtained from Kankara kaolin, which was locally sourced in Kankara LGA in Katsina, Nigeria.

Preparation of metakaolin

The method reported in the literature [5,22] was employed to prepare the metakaolin in the study.

(a) Kankara kaolinite clay's beneficiation

The raw kaolin clay collected from the Kankara (a community in the Katsina State of Nigeria) was beneficiated by soaking it in water for three (3) days with periodic stirring in a plastic vessel to break up small lumps present in the clay and for the removal of soluble impurities present in the clay. The suspended impurities were removed via the decantation approach on a daily basis. The resulting mixture was allowed to settle for 24 h to obtain a thick clay mass. After settling, it was decanted and dried in the open air for two (2) days. The beneficiated clay was ground and sieved using a 75-micron sieve[22].

(b) Beneficiated Kankara kaolin clay's calcination

Fig. 1. Calcined kaolinite clay

The dried kaolinite powder was loaded into crucibles and calcined in an electric furnace for the period of 5 h at the temperature of 750 °C to obtain the more reactive phase of kaolin known as metakaolin (Fig. 1). The resulting metakaolin was analyzed for elemental composition using a spectrometer X-Supreme 8000 XRF (an Oxford Instrument).

The chemical equation expressing changes involved in the calcination process is presented in equation (1).

$$Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O \rightarrow Al_2O_3 \cdot 2SiO_2 + 2H_2O \quad (1)$$

This equation displayed the mechanism involved in the calcination of the kaolin to have metakaolin.

Composite preparation

The method reported by Gaiya *et al.* [23] was adopted for the preparation of unsaturated polyester composite. Materials were weighed using an electronic weighing-balance. In synthesizing reinforced polyester composite, the polyester's mass was varied with that of the metakaolin filler to give a total of 100 grams. The filler was added into the polyester resin and stirred continuously with a mixer until a uniform mixture is achieved. After that, 1ml of Methyl-ethyl-ketone-peroxide (MEKP) (i.e., catalyst) was added with the aid of a disposable syringe. These components were then mixed for about 2 minutes, after which 1.5 ml of cobalt naphthanate (i.e., accelerator) was added and stirred for 2 minutes.

Table 1. Formulation Table for Polyester/Metakaolin Composite

Specimen	Metakaolin Composition (wt %)	Unsaturated polyester (ml)	MEKP (ml)	Cobalt Naphthanate Accelerator (ml)
A	0	1×10^2	1	1.5
B	2	98	1	1.5
C	4	96	1	1.5
D	6	94	1	1.5
E	8	92	1	1.5
F	10	90	1	1.5

The next stage involved pouring the mixture into a metal mold, where it was allowed to cure. This procedure was repeated for other specimens with varying weight percentages of the metakaolin filler (Fig. 2). A control sample was produced without adding the filler. Table 1 presents the formulation matrix employed in preparing the polyester/metakaolin composite (PMC) production.

Composite Testing*: Determination of dielectric property*

The samples produced for the dielectric property test were 3.6 mm thick and 230 mm, diameter. A programmable LCR Bridge (HM8118) was employed to measure the sample's capacitance, Cs, and the loss tangent, tan (δ) at frequencies ranging from 20-1000 Hz. The sample was fixed between two electrodes. Equation (2) was used in determining the dielectric constant, ε^{l};

$$\varepsilon' = \frac{C_s d}{\varepsilon_o A} \quad (2)$$

Where ε_o is the constant of free space (8.85 E-12 F/m), A is the effective cross-sectional area, and d is the thickness of the sample [17,24]. The dielectric loss factor ε^{ll} was calculated from equation (3).

$$\varepsilon'' = \varepsilon' \tan(\delta) \quad (3)$$

Where δ is the phase angle between the electric field and the polarization of the dielectric [24].

Results and Discussions

Elemental composition *of metakaolin*

The prepared metakaolin characterized for elemental composition using spectrometer X-Supreme 8000 XRF (an Oxford instrument) is shown in Fig 2.

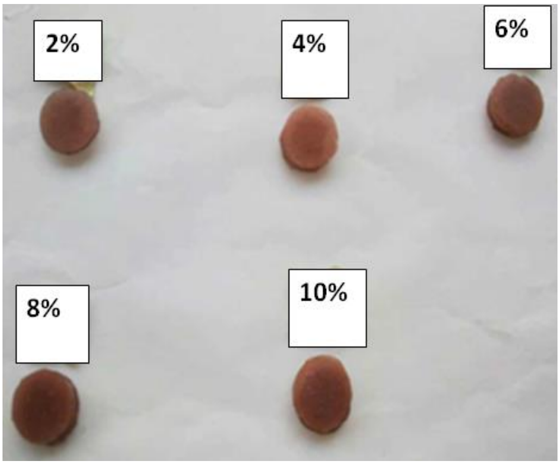

Fig. 2. Metakaolin/Polyester composite

The study of results presented in Table 2 shows that the metakaolinization of the Kankara kaolin achieved by calcination/dehydroxylation was evident by enrichment of Al_2O_3 and SiO_2 at 41.536 % *and* 55.284 %, respectively, with fewer impurities. The impurities were fewer due to the large composition claim as 97 % for the products, silica, and alumina, while others were 3 %.

Table 2. Elemental compositions of metakaolin

Element	Metakaolin (%)
MgO	0.743
Al_2O_3	41.536
SiO_2	55.284
SO_3	0.150
Cl	0.010
K_2O	1.123
CaO	0.229
TiO_2	0.084
Cr_2O_3	0.002
Mn_2O_3	0.011
Fe_2O_3	0.815
ZnO	0.009
ZrO	0.004
SiO_2/Al_2O_3	3.370

Upon thermal treatment, the water was liberated to form an amorphous aluminosilicate called metakaolin. There is an increase in Si-O-Si groups. Even though the metakaolin is dehydroxylated by calcination, it can be hydroxylated when in contact with the atmosphere; this can increase hydroxyl groups, affecting the relaxation phenomenon [7,14].

Effect of frequency *on the dielectric property of the unsaturated polyester/metakaolin composite*

For PMC with different metakaolinite loading, the dielectric constant falls with rising frequency (Fig. 3), which can be attributed to an interfacial relaxation [3,25,26]. The findings confirm that the dielectric constant of the composite has a negative correlation with frequency. However, it may be related to electronic polarization for the higher frequencies [4,24]. Generally, strong interfacial interaction was beneficial to limit the dipoles' polarization movement because the dielectric value depends on the dipoles' presence. If dipoles are present, it would permit electrons' flow, thereby decreasing the resistance value hence a small dielectric [26,27].

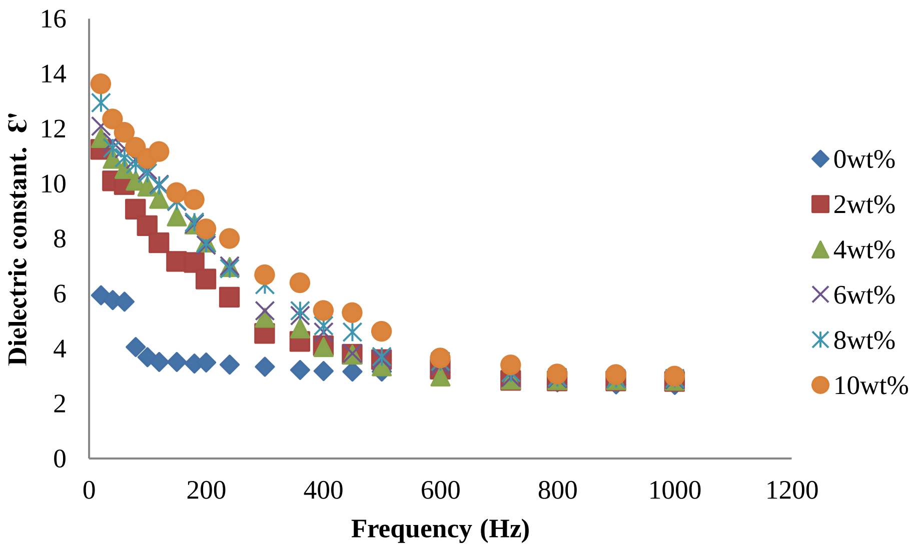

Fig. 3. Polyester/metakaolin composite's dielectric constant variation with frequency

The study of results presented in Fig. 3 reveals that an increase in the metakaolin loading content (MLC) increases the dielectric constant of composite (DCC). This observation implies that the composite's dielectric constant showed a positive correlation with MLC in the polyester matrix. The permittivity enhancement is attributed to interfacial polarization, also known as the Maxwell–Wagner–Sillars (MWS) effect or polarization. This deduction was because fillers have a higher permittivity by nature than the base polymers, causing interfacial polarization. In turn, the interfacial polarization provides information about charge trapping associated with internal surfaces and relaxation processes associated with dipole reorientation [28]. The control sample exhibited the lowest dielectric constants because of the absence of permanent dipoles since the matrix was composed only of carbon and hydrogen atoms. All loadings are significantly appreciated with high dielectric capacity at a lower frequency but reduced with increasing frequency. PMC with 10 wt% showed the highest dielectric-frequency trend, validating dielectric dependency on frequency. The highest dielectric constant value obtained is 13.63, which was about 5 times higher than its pure form.

Effect of metakaolin loading on dielectric property of unsaturated Polyester/Metakaolin composite

It is clear from Fig. 4 that the dielectric strength increased with the metakaolin loading's increase [10]. This trend can be attributed to metakaolin's total surface area and its continuity [29].

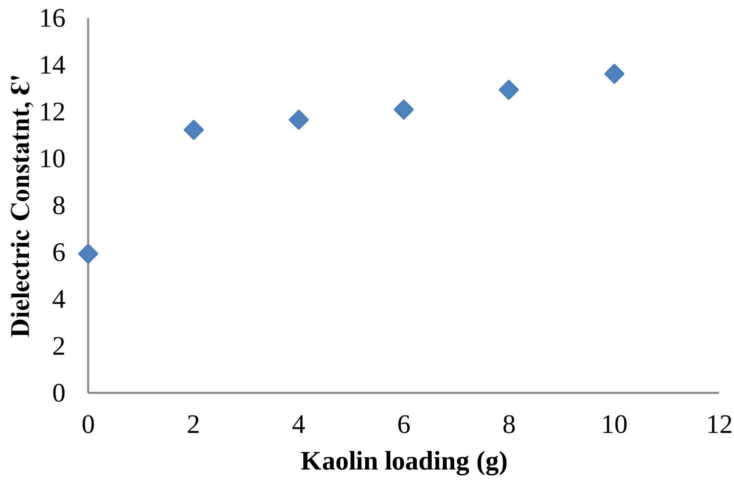

Fig. 4. Variation of the dielectric constant of polyester/metakaolin composite with kaolin loading at 20Hz.

As the material density rises, it exhibits greater resistance to flow hence a high dielectric constant [27]. The dielectric strength values showed dependency on the higher loading of metakaolin.

Effect of frequency on the dissipation factor of unsaturated polyester/metakaolin composite

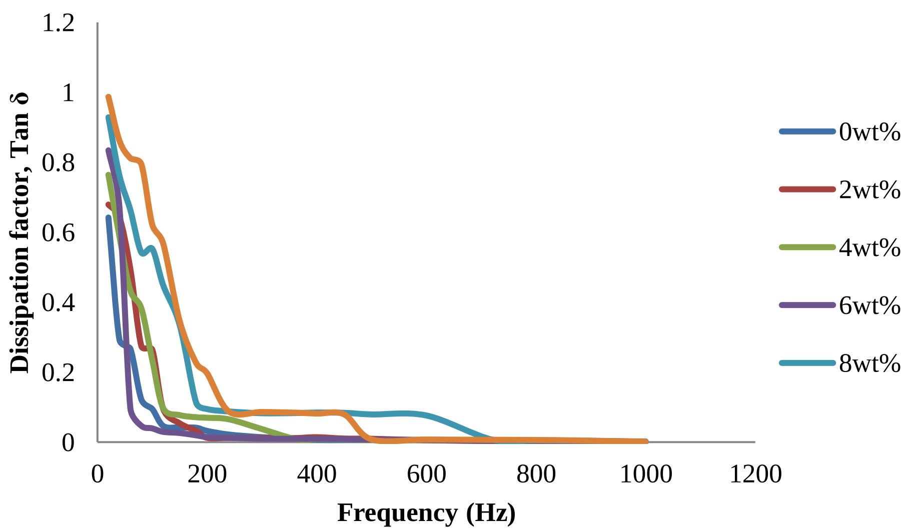

Fig. 5. Variation of the dissipation factor of polyester/metakaolin composite with kaolin loading at 20Hz.

When an alternating voltage is applied to an insulating medium, the capacitive current leads the voltage by 90º. When impurities such as moisture, contaminants, or oxidation byproducts are present within the insulating medium, dielectric losses are created, which causes the current to deviate from the ideal 90º phase shift from the applied voltage. This shift from 90º is measured as the Dissipation factor (tangent of loss factor δ).

From the results presented in Fig 5, it was deduced that the dissipation factor, δ, which is inversely frequency-dependent, decreasing with an increase in frequency [4]. When the metakaolin loading was increased, it showed a significant increase in the low-frequency region's behavior than the pure matrix material at a given frequency. As the frequency increases between 20 and 200 Hz, the dissipation factor decreases suddenly for all loadings. At a higher frequency (>200 Hz), the dissipation factor linearizes at tan δ<0.2.

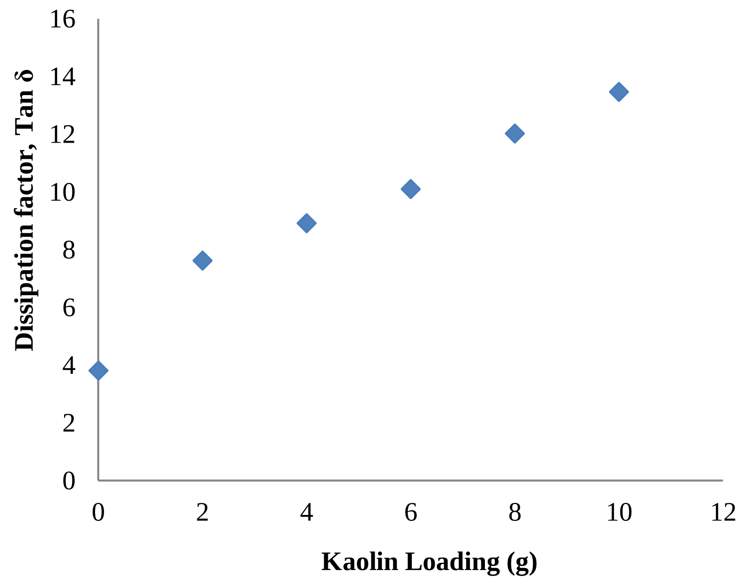

Fig. 6. Variation of the dissipation factor of polyester/metakaolin composite with kaolin loading at 20 Hz.

Effect *of metakaolin loading on the dissipation factor of unsaturated polyester/metakaolin composite*

The study of results presented in Fig 6 reveals that a rise in the dissipation factor was obtained for increasing kaolin loading at 20 Hz (frequency). At the metakaolin loading of 2 wt%, which corresponds to a dielectric constant of 11.668, the dissipation loss is within the matrix's neighborhood. However, there is an increase in the dissipation factor with increasing loading, which shows a good agreement with the report of Yan *et al.* [17]. This observation can also be attributed to the samples' interfacial polarization and increases with a rise in loading.

This study confirms that the composite's dielectric constant shows a negative correlation with frequency while a positive correlation with metakaolin loading content in the polyester matrix. Also, the dissipation factor of composite showed a positive correlation with the metakaolin content and a negative correlation with frequency. This observation confirms the dissipation factor's direct relation with the composites materials' dielectric constant, which agreed with the literature [17,24] mathematical relations report.

Conclusions

In this study, the dielectric properties of varying metakaolin/polyester composite matrices were studied as a function of frequency and metakaolin loading. The results revealed that the produced Kankara metakaolin was rich in alumina and silica with 97% purity. The best dielectric improvement obtained for the composite was achieved with increasing loading of metakaolin was obtained at the highest loading (10 wt%). Further investigation displayed that the composites' dielectric constants decreased with a frequency increase from 20 Hz to 1MHz. The highest dielectric constant obtained for this composite material was 13.63, indicating an excellent electrical property.

Also, this study was able to identify a metakaolin obtained Kankara kaolin to be a material with the potential positive influence of significantly improving the polyester dielectric properties and to increase its affordability due to its low cost potentially. We expect that these results presented in this study would help promote polyester-based composite materials to practical applications like capacitors.

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