Journal of Materials and Environmental Science
ISSN : 2028-2508
CODEN : JMESCN

J. Mater. Environ. Sci., 2021, Volume 12, Issue 03, Page 406-417

http://www.jmaterenvironsci.com

Experimental Effect of Filler Variation on the polarizability of polymer Matrix Composites Developed from Orange Peel Particulates.

A. D. Omah [1,2], E.E. Ugwuogbo [1], P.O. Offor [1], I.Y. Suleiman [1], S.V. Egoigwe [3], U.C. Ogbuefi [3]**

[1]Department of Metallurgical & Materials Engineering, University of Nigeria, Nsukka, Nigeria.
[2]African Centre of Excellence, ACE-SPED, University of Nigeria, Nsukka, Nigeria.
[3]Department of Electrical Engineering, University of Nigeria, Nsukka, Nigeria.
***Corresponding author, Email address: augustine.omah@unn.edu.ng*

Received 29 Jan 2021,
Revised 25 March 2021,
Accepted 27 March 2021

Keywords
- ✓ *Orange Peel*
- ✓ Composite,
- ✓ Dielectric,
- ✓ Polyester,
- ✓ Particulate.

**Corresponding author
augustine.omah@unn.edu.ng
Phone: +2348033863639

Abstract

This study investigated the dielectric behaviour of orange peel (OP) reinforced polyester composites. Orange peels (OP) were sourced from Nsukka environs as agro – waste materials. The peels were washed and air dried, after which they were pulverized and sieved to obtain 300 µm particulates. The resin was blended with the agro-waste materials in appropriate ratio. Composites having 10, 20, 30, 40 and 50% weight fraction of OP were made using hand layup method. The nature and dispersion characteristics of the OP particles in the matrix was revealed using scanning electron microscope (SEM), while the elemental compositions of the particulates were revealed by EDS analysis and the phases at the diffraction peaks of the X-ray diffractometer (XRD). The degradation temperature/thermal stability of the samples were quantified using Thermo-Gravimetric Analysis (TGA). Dielectric and physical properties were determined on the samples. Results revealed that the dielectric strength and dielectric constant had their optimum values at 40 and 20 wt% respectively. Resistivity decreased as the filler loading increased and a more enhanced property was obtained at the 10 wt% composition. It was also observed that the 50 wt% sample has the highest moisture content and water absorption values. The properties determined were observed to be equivalent to some typically used dielectrics. Thus, they can be employed in areas of application as served by other orthodox dielectrics.

1. Introduction

A dielectric is a material that can be described by its electric dipole structure which shows a separation of positive and negative electrically charged molecular or atomic entities; i.e. it is an electrically insulating material which can be polarized under an electric field. Due to dipole interactions with electric fields, dielectric materials store electric charges for capacitors [1]. It also provides an understanding of the storage and dissipation of electric and magnetic fields in materials and its potential applications in electrical insulation, sensor devices and circuit components [2].

From the concept of polarization, when a dielectric is placed in an electric field, the charged entities (electrons and protons) of the atoms or molecules reorient themselves and become polarized; thus, the insulating medium due to pressure stores energy that becomes available when the electric field is removed. The polarization of a dielectric is analogues to the polarization of a magnetized piece of iron. Hence, just like in the case of a magnet, a certain amount of polarization remains when the polarizing force is removed.

Dielectric measurement is a significant tool to understand and analyze the response of a material to electric field. It provides the electrical or magnetic characteristics of the materials, which is an essential parameter to predict the use of the material in many applications. The polarizability of the material is expressed by permittivity, which is a complex number often referred to as dielectric constant [3]. Dielectric constant values vary from a little more than 1 for air to 100 or more for certain ceramics containing titanium oxide. The dielectric constant of certain materials (like: glass, mica, porcelain, etc.), that serves as an insulation medium in certain devices and circuits, are within the range of about 2 to 9. The ability of a dielectric to withstand electric fields without losing insulating properties is known as its dielectric strength [4]. The need for enhanced dielectrics which can find applications in electrical insulation and circuit components is the motivation behind the present study which tests for the dielectric properties of composite material developed from orange peel (OP) particulates.

Insulation material is any material that is a poor conductor of heat or electricity i.e. it can be used to suppress the flow of heat or electricity. The insulation resistance of a dielectric material is the combined effect of volume and surface resistance [1]. Cost implication has constituted a great obstacle to engineers in meeting the demand for insulation materials despite its vital role in electrical system or industry. Thus, this work was aimed at developing a composite material with improved electrical insulation properties, low cost implications and minimum environmental pollution. This work therefore used locally sourced material (OP particulate) to meet these necessary demands.

The combination of two or more materials with different properties to give rise to entirely new material with characteristics different from the individual components is referred to as a composite [5]. The distinctive physical phases of the constituents are immiscible with each other and therefore do not form a new chemical compound. A composite therefore is any material combination system that reveals distinctive improved properties that are preferred to or better than the properties of the constituent components. It is a material system with enhanced properties that are absent in the individual elements. Composite materials are characterized into two basic classifications namely: classification based on matrix i.e. polymer matrix composite (PMCs), metal matrix composites (MMCs), and ceramic matrix composites (CMCs); and classification based on reinforcement i.e. fiber-reinforced (continuous and discontinuous) composites, particulate reinforced composites, whisker reinforced composites, and flake-reinforced composites. The term natural fiber or particulates covers a wide range of plant, animal and mineral fibers used as fillers. Availability of these natural fillers, easy of manufacturing and no or low cost implications have led to the use of locally available inexpensive natural particulate fillers like orange peels as reinforcement material in polymer matrix.

Orange peel (OP) is the outer protective layer of an orange that can be peeled off. Thus, orange peels are obtained by peeling off the outer layer of the orange fruit. The peels can be processed and used as reinforcements for polymer composites production and for other domestic applications. Orange is a citrus fruit largely found in Southeast Asia. It is acidic with a pH range of 2.9 – 4.0 [6].

Poor waste management can give rise to deleterious health implications with accompanying economic waste and maintenance costs. The total annihilation or control of the dangerous effects of littered wastes which includes wastes from agricultural produce is now a prime target in developing nations, even as governments, researchers and scientist of such nations are trying to recover the ecological damage done by these wastes by embracing the technology of conversion of waste into wealth [7]. OP wastes are principally used during extraction of some oily substance used for some chemical preparations. To complement its primary usage, orange peel wastes is now employed in composites technology for dielectric applications.

Due to the continual alarming increase in human population and the progressive technological demand by the populace, there is a high need for a cost effective, eco-friendly and light weight electrical insulating materials for engineering applications. The adverse effect of many agro–waste materials that litter the environment, marring the beauty of the landscape due to wrong waste handling, poor waste disposal and inability to recycle them into more useful products is a call for major attention [8]. Thus, this work aimed at investigating the potency of orange peel polyester composite for application such as: power transmission insulators, high performance circuit board, etc. Some works on agro–waste based composites include:

V.S. Aigbodion *et al* [9] carried out research on orange peel particulate in their published work titled, "Development of High-Density Polyethylene/Orange Peels Particulate Bio-Composite" where they established that the orange peels waste could be used as a biodegradable eco-friendly reinforcement.

"Optimization of Water Absorption Properties of Orange Peel Particulate-based Epoxy Composite Using Grey Relational Analysis" by the authors: Oluwaseyi A. Ajibade, Johnson O. Agunsoye, and Sunday A. Oke, where they determined the water absorption capabilities of orange peel particles of different compositions [10].

Vijaya Kumar Nimmagadda *et al* [11], investigated the dielectric properties of industrial waste reinforced particulate polymer composites. Results revealed that coupling agent treated composites produced improved dielectric strength due to improvement in compatibility between matrix and reinforcement interface.

Despite the above-mentioned studies by researchers on industrial and agro – waste materials like orange peels as reinforcing fillers for composites, the novelty of orange waste-based polymer composites in terms of dielectric properties, has not been well investigated, for it is still a scarce information in literature. This implies that orange peel particulate composite dielectric properties have not been well studied. Based on this, this study targeted the development of a very good insulator from orange peel particles through the determination of the dielectric properties of the orange peel particulate composite using polyester as the matrix.

2. Methodology

2.1 Sourcing and preparation of materials

Orange peels were sourced from Nsukka locality as waste and processed into fine particles; Polyester resin (with a melt flow index of 2.5 - 3.5 g/min, and density, 0.926 g/cm^3), Cobalt Napthanate (accelerator), and Methyl Ethyl Ketone Peroxide (catalyst) were all procured from a vendor Chemical shop. Equipment used include: Scanning electron microscope (SEM) JEOL JSM-6480LV, Dielectric tester by fosters transformer Ltd., SDT Q600 V20.9 for TGA, Micro-ohm-meter by DV POWER, Kaise insulation test model SK5010 was used in measuring the resistivity of the polyester composites. Figure 1 and 2 denotes the orange peels and the fabricated samples respectively.

The orange peels were procured from Nsukka market, washed and sun-dried for one week. It was pulverized after drying using a conventional grinding machine and sieved into particle size of 300 μm. The resin with 0.1% catalyst and accelerator was then blended with the reinforcing material to ensure an efficient and proper coating of the agro-waste with the binder. The resin to fibre ratio for every 100wt% of the composite composition is 9:1, 4:1; 7:3; 3:2 and 1:1. The mixture was cast into the moulds to form the composites. The produced composites were examined under the following property tests: dielectric strength, dielectric constant, resistivity, water absorption capacity and moisture content.

Figure 1: Orange peels

Figure 2: cast samples

2.2 Experiments

The samples were cut to dimensions and were immersed in water for 24 houurs and then palmed dried with a cloth, and weighed to the nearest 0.0001g. The weights (M_1) and (M_2) of the samples were measured before and after the water absorption and after the drying process respectively. The water absorbed (W_A) was calculated as percentage weight gain using equation 1 below according ASTM D570-98 [12].

$$W_A = \frac{M_2 - M_1}{M_2} \times 100 \qquad \textbf{Eqn. 1}$$

The samples for the moisture content were cut to dimensions and weighed to obtain the weight (M_1); the samples were then placed in an oven at 105 °C and reweighed at one hour intervals until a constant mass (M_3) was obtained. The moisture content (M_C) was calculated as percentage of the dry sample from equation 2.

$$M_C = \frac{M_1 - M_3}{M_1} \times 100 \qquad \textbf{Eqn. 2}$$

The breakdown voltage of the samples was determined using a Foster dielectric testing machine. Each cylindrical specimen of 18 mm diameter and 105 mm length were placed in between the machine's electrodes on either side and the voltage steadily increased till there was a breakdown. The failure was characterized by a large sound. The dielectric strength was subsequently determined [13, 14].

Dielectric constant ($\mathcal{E}_r$) relates to the permittivity of the material ($\mathcal{E}$) and a material's permittivity expresses the ability of a material to polarize in response to an applied field. Thus, the greater the polarization by a material in an applied field of given strength, the greater the dielectric constant. It is the ratio of the permittivity of the dielectric ($\mathcal{E}$) to the permittivity of a vacuum ($\mathcal{E}_o$). To determine the dielectric constant, the composite samples were moulded into rectangular plates of length 50 mm, width 30 mm and thickness 2 mm. An air gap was created between two parallel plate capacitors which has same thickness as the sample. This was connected to the battery and the voltage V_o across was measured. Then the samples were each, put in between the capacitors and each sample's voltage, V taken. The dielectric constant ($\mathcal{E}_r$) was estimated using equation 3 which gives the relationship between V_o (voltage across the capacitors with an air gap between them) and V (voltage across the capacitors with the composite sample between them). The dielectric constant $\mathcal{E}_r$, is therefore expressed as follows:

$$\varepsilon_r = \frac{V_0}{V} \qquad \textbf{Eqn. 3}$$

Resistivity test yielded the resistance of the specimen to the flow of electric current. It is measured as the insulation resistance. The samples were moulded into cylindrical shapes of diameter 18 mm and length 105 mm with a copper wire of diameter 2.5 mm and length 5 mm placed at the ends of each sample. The measured values were used to determine the resistivity of each of the samples using equation 4 below.

$$\rho = \frac{R}{A} \times L \qquad \textbf{Eqn. 4}$$

Where: R = Resistance in ohm
A = Area of the sample in mm^2
L = Length of the sample in mm

2.3 Product characterisation

Thermo-gravimetric analysis, (TGA SDT Q600 V20.9) was carried out on the samples to determine the degradation temperature and thermal stability of the material. Samples of size 2.9410mg, were scanned from 50 °C to 1000 °C at a heating rate of 20 °C/min under N_2/O_2. X-ray diffractometer (XRD) measurements were carried out on the samples between 0 to 90 degrees 2θ. XRD was used to carry out a mineralogical analysis to reveal the elements present in the material. The Scanning electron microscope (SEM JEOL JSM-6480LV) was used to study the composites micrographs with 10%, 30% and 50% orange peel particulate reinforcement; and the elemental compositions measured using EDS.

3. Results and Discussion

3.1 Dielectric strength

Figure 3 shows a plot of dielectric strength (kV/mm) versus weight % of orange peels particulate reinforcement. The result showed that the values of the dielectric strength increased with increase in the weight percent of the reinforcement particles. The composition with best dielectric strength was that of 40 wt% particulates. From 10 – 40 wt% filler loading, there was good particle – matrix interface bond, which aided the increase in dielectric strength. Higher loading than 40 wt% may have resulted to poor particle – matrix interaction due to poor wettability arising from insufficient resin for strong bond formation, thus, decreased dielectric strength.

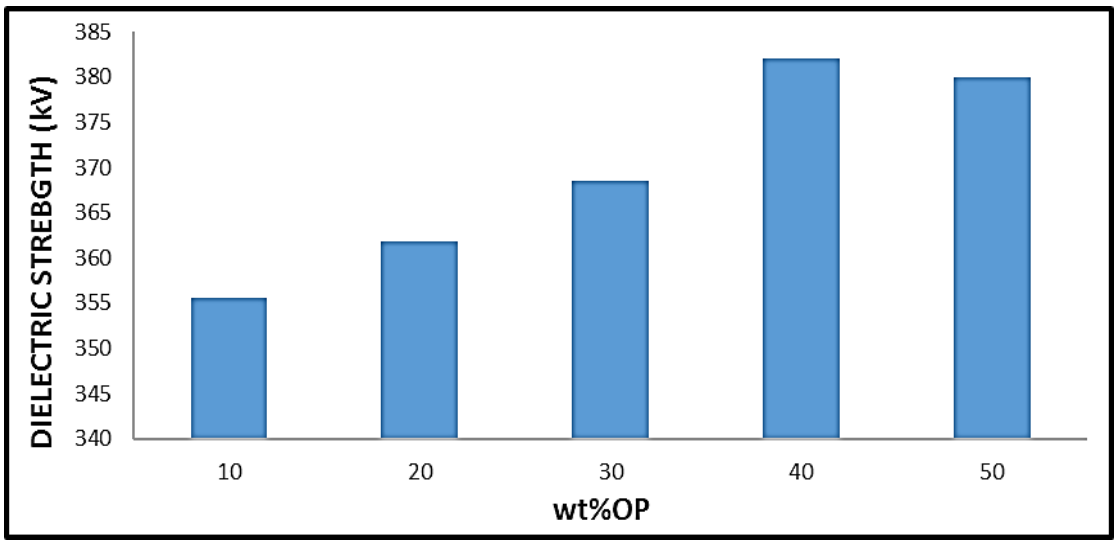

Figure 3. Dielectric strength of OP Polyester Composite

3.2 Dielectric constant

Figure 4 is a graph of dielectric constant against wt% OP particulate. The dielectric constant of the samples initially increased from 10 wt% composition to 20 wt% composition i.e. it increased with the proportion of particles added. This was because the extra wt% addition led to increase in number of dipoles per unit volume, which means increase in the number of polarized dipoles that orients due to the effect of the electric field. However, the dielectric constants of the composites were observed to decrease after 20 wt% composition. Beyond this composition, there was poor interfacial bonding which as well led to increased number of voids in the samples. Thus, the result showed that the dielectric constant of the samples reduced evidently with increased porosity in the sample as a result of poorly bonded particulates. This is in agreement with the work done by Jie *et al*. [15] and Chi *et al* [16]. Moreso, there was high filler – matrix homogeneity in samples with 10 to 20 wt% than in the samples with 30 wt% and above. Samples with higher filler loading had poor dispersal of reinforcements, thus heterogeneity of reinforcements in the matrix which led to particle agglomeration and increased inter – particle pores. Thus, dielectric constant of the samples decreased with increasing porosity in the sample as the relative permittivity of air is about one [17]

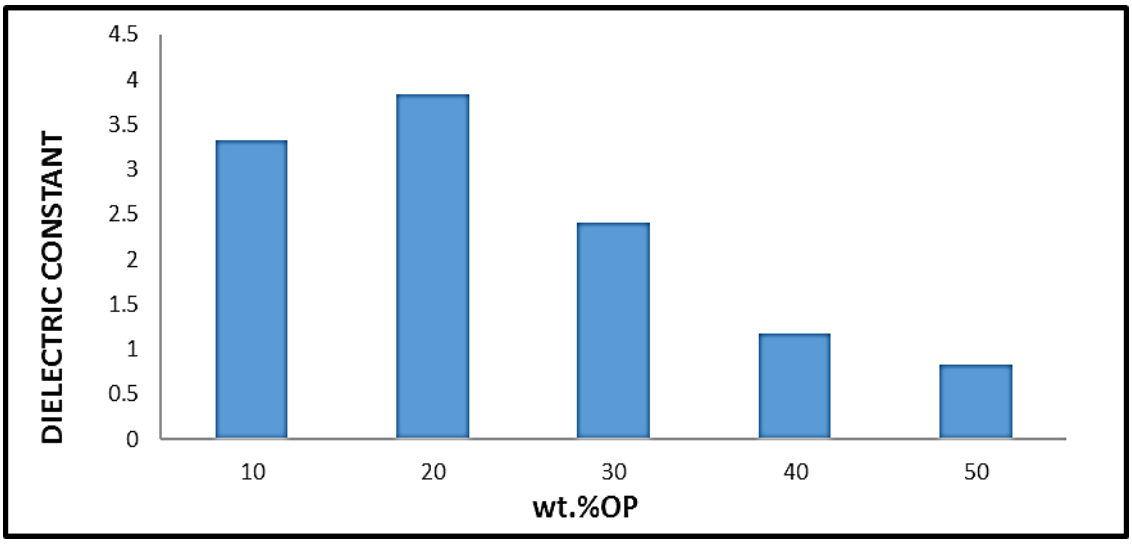

Figure 4. Dielectric constant of OP polyester composite

3.3 Resistivity

The graph of resistivity (ρ), against wt% OP of the composite is shown in Figure 5. The electrical resistivity of the composite significantly decreased with increased filler loading. The electrical conductivity of the composites was reasonably high as a result of the polarization of more organic functional groups in the higher wt% composites, thus lower resistivity in these higher filler loading composites; but at lower filler content, there was polarization of fewer organic functional groups, thus, lower conductivity and high resistivity.

3.4 *Water absorption and moisture content*

Figure 6 shows the effect of filler loading on the water absorption behaviour and moisture content of the fabricated composites. It was observed that the composite with higher filler content absorbed more water molecules. This shows that the filler particles are hydrophilic, hence the addition of hydrophilic particles to the matrix increased the water absorption capacity of the materials. Thus, composites with higher volume fraction possess higher percentage water absorption.

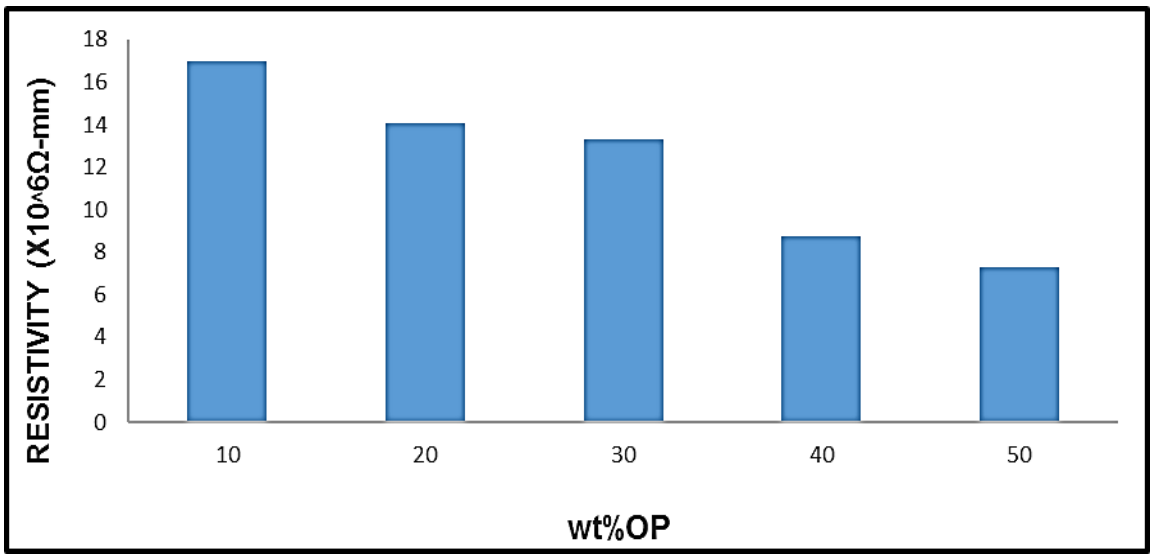

Figure 5. Resistivity of OP Polyester Composite

With increased filler loading, there existed poor interfacial bonding between the filler and the matrix which resulted in an increase in the number of micro-voids, causing increased water absorption. [18, 19]. Better enhancement of property was obtained for the composites with lower wt% compared to those with higher wt%. The small grain size of 300 µm used helped to keep the water absorbed to a minimal due to good particle – matrix interfacial bonding that minimized the presence of inter – particle pores that would have been a good site for water absorption.

It was also seen that the sample with highest wt% (i.e. 50 wt%) has more moisture than other samples; i.e. there was a progressive increase of moisture content from 10 – 50 wt% of the orange peel particulates. This was because more particulates of the orange peels contained more internally and chemically bound water.

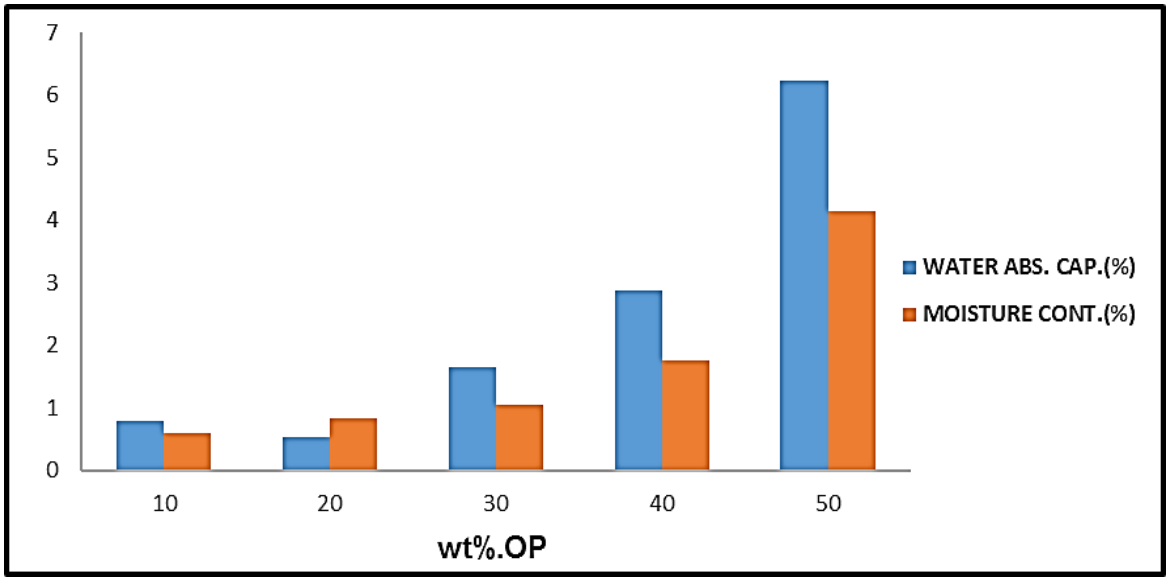

Figure 6. The Water Absorption Capacity and Moisture Content of OP Polyester Composite

3.5 *Thermo-gravimetric analysis (TGA)*

Thermo-gravimetric analysis (TGA) was carried out on the fabricated OP particulate composite using a TA Instruments Q50 TGA tests and the result is as shown in Figure 7. The degradation

temperature/thermal stability of the material and the corresponding percentage weight were quantified and are reported in Figure 7. The increase in degradation temperature of the composite sample is practically useful for application in high temperature wire insulation.

The TGA of the OP composite was studied as a function of weight % loss with increase in temperature. The TGA curve shows three weight loss steps, and two stages of thermal decomposition. The temperature of destruction (T_{des}) of the OP particle was also determined.

The TGA curve reveals the initial weight loss due to moisture dehydration from the OP particles between 25 °C and 100 °C. Shortly after this temperature, the OP particle degradation started. The second phase of weight loss occurred between 100 °C and 325 °C where more moisture and some cellulose content of the OP got burnt off (i.e. hemicellulose decomposition). This is seen in the graph as a very gradual drop in the thermal stability of the OP particle between the temperature range of 100 – 325 °C. The third stage reveals a sharp drop in the thermal stability from 340 °C till final degradation precisely at 425 °C. Thus, the temperature range of maximum decomposition of the OP residual particle from the TGA is 340 °C – 425 °C.

Two temperature peaks were observed at 100 °C and 375 °C but maximum thermal decomposition occurred at the second peak, 375 °C. Thus, the overall effects of the temperature peaks on the OP are seen in the following sequence: dehydrogenation, loss of some volatile matter and the thermal decomposition of the material. The general outcome of the TGA analysis is seen in the weight loss i.e. decreased mass of the sample between 25 °C and 425 °C.

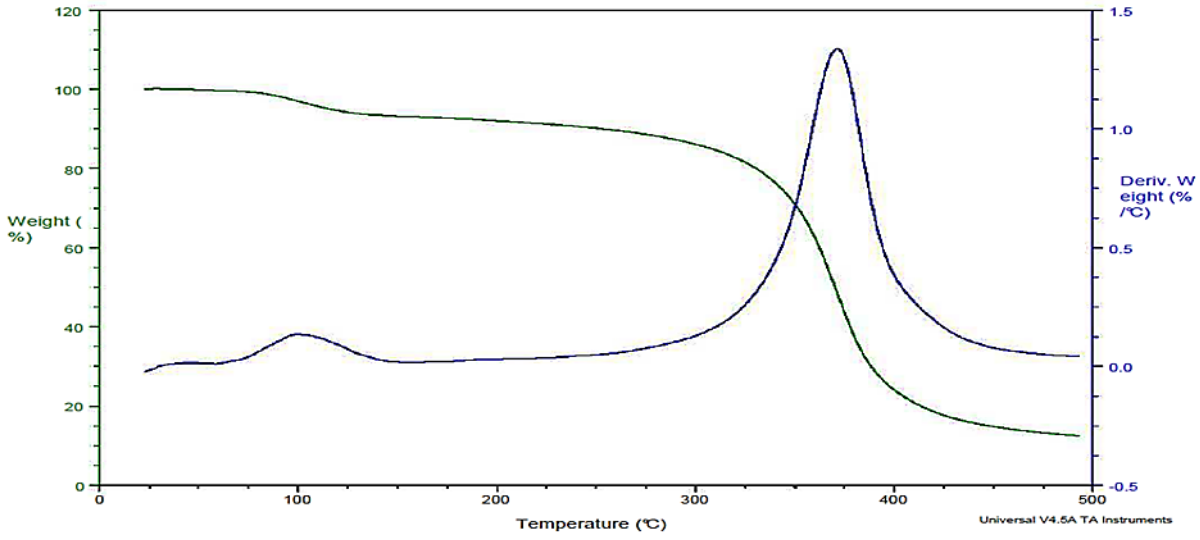

Figure 7. Thermo-gravimetric analysis (TGA) of OP polyester composite

3.6 *X – Ray Diffraction Analysis*

The XRD pattern of the OP shown in Figure 8 revealed the phases at the peaks: quartz (SiO_2), Potassium Aluminum Oxide Hydrate ($K_2Al_2O_4!3H_2O$) and Quinizarin, $C_{14}H_8O_4$, with: 67, 34 and 21 as their respective scores, and scale factors of 0.722, 0.080 and 0.074. SiO_2 has the highest amount with a score of 67 and a scale factor of 0.722. These characterize the comparative volume of each phase in the XRD pattern as shown in Figure 8 and Table 1. The major diffraction peaks as detected at 2θ positions were shown to be: 3.56, 12.56, 100, 13.99, 29.97, 13.23, 13.37, 7.83 and 4.45, with their corresponding inter-planar distances as: 6.23258, 4.25118, 3.34126, 3.14924, 3.02889, 2.94891, 2.28091, 2.50910, and 1.97943Å (See Table 2).

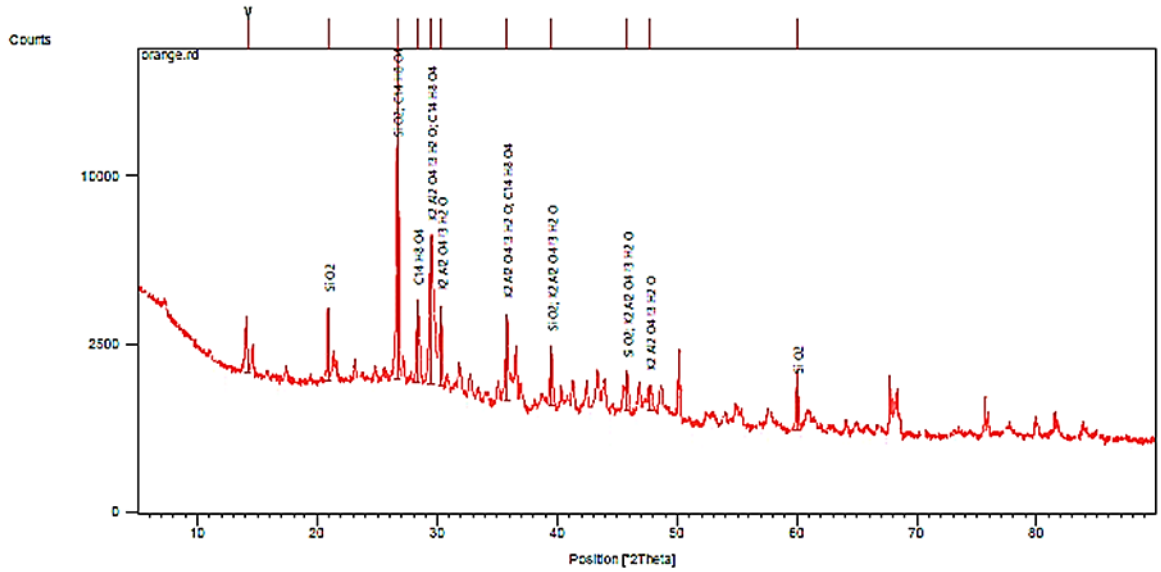

Figure 8. The XRD pattern of OP powder.

Table 1. Identified Patterns List of the elements present

Visible	Ref. Code	Score	Compound Name	Scale Factor	Chemical Formula
*	85-0795	67	Quartz	0.722	SiO_2
*	19-0927	34	Potassium Aluminum Oxide Hydrate	0.080	$K_2Al_2O_4$!$3H_2O$
*	25-1920	21	Quinizarin	0.074	$C_{14}H_gO_4$

Table 2. XRD Peak List

Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]	Tip width [°2Th.]	Matched by
14.2108	622.95	0.8029	6.23258	3.56	0.8160	
20.8964	2199.48	0.1004	4.25118	12.56	0.1020	85-0795
26.6804	17517.99	0.0669	3.34126	100.00	0.0680	85-0795; 25-1920
28.3401	2451.55	0.1338	3.14924	13.99	0.1360	25-1920
29.4912	5249.64	0.1506	3.02889	29.97	0.1530	19-0927; 25-1920
30.3100	2317.54	0.1338	2.94891	13.23	0.1360	19-0927
35.7880	2342.52	0.1338	2.50910	13.37	0.1360	19-0927; 25-1920
39.5095	1371.75	0.2007	2.28091	7.83	0.2040	85-0795; 19-0927
45.8435	779.83	0.2676	1.97943	4.45	0.2720	85-0795; 19-0927
47.7327	503.01	0.3346	1.90541	2.87	0.3400	19-0927
60.0030	725.46	0.4896	1.54053	4.14	0.4080	85-0795

The phases present at the XRD peaks suggests that the developed composite is an agro-based material that could contain at least one of these elements: "H, C, O, Na, Mg, Al, Si, K, Ca". The results of SEM/EDS scan of Figure 9 – 11 confirmed the existence of these elements with carbon and oxygen as the main constituents present and other traces of elements significantly present like silicon, sodium, potassium, magnesium, calcium, aluminum etc. Thus, the OP particles did not contain lethal and destructive materials.

3.7 SEM/EDS

Scanning Electron Microscopy (SEM)/EDS

The dispersion characteristics of the polyester composite have been studied using scanning electron microscope. The reaction products at 10 wt%, 30 wt% and 50 wt% of the samples were characterized and presented as shown in **Figure 9, 10** and **11** respectively. The micrographs revealed the reinforcements to be randomly distributed solid particles; The SEM/EDS for 30 wt% and 50 wt% revealed the presence of more particles than that of 10 wt%.

The micrographs show the particles in white while the polyester resin in dark colour. The white parches increased with increase in OP particles. The rough surface nature of the OP particles enhanced more surface area for interaction with the matrix thus, good particle – matrix interfacial strength for enhanced dielectric properties.

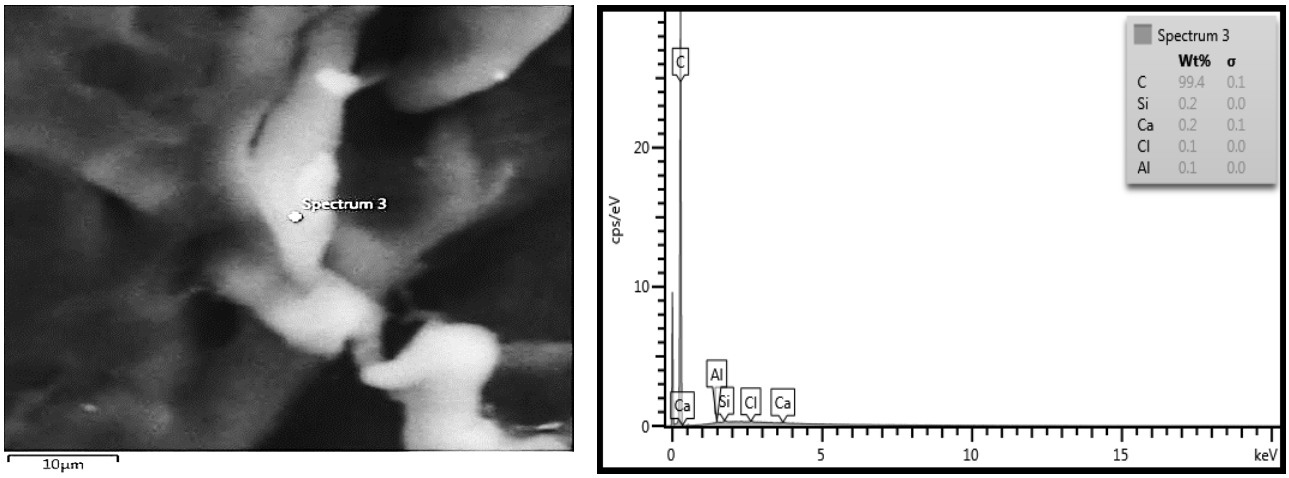

Figure 9. Photomicrograph of 10 wt% OP Particulate Composite as Revealed by SEM/EDS

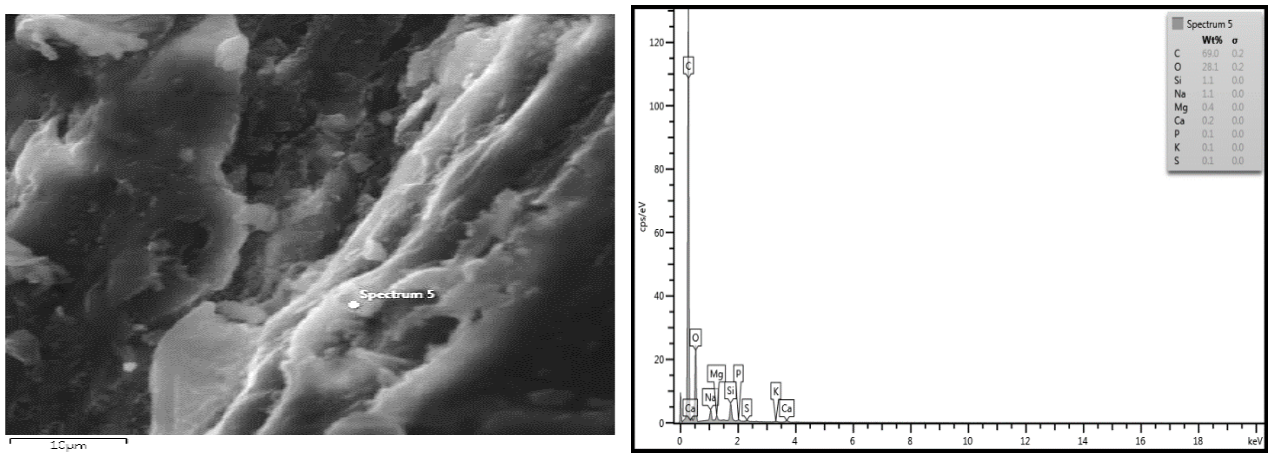

Figure 10. Photomicrograph of 30 wt% OP Particulate Composite as Revealed by SEM/EDS

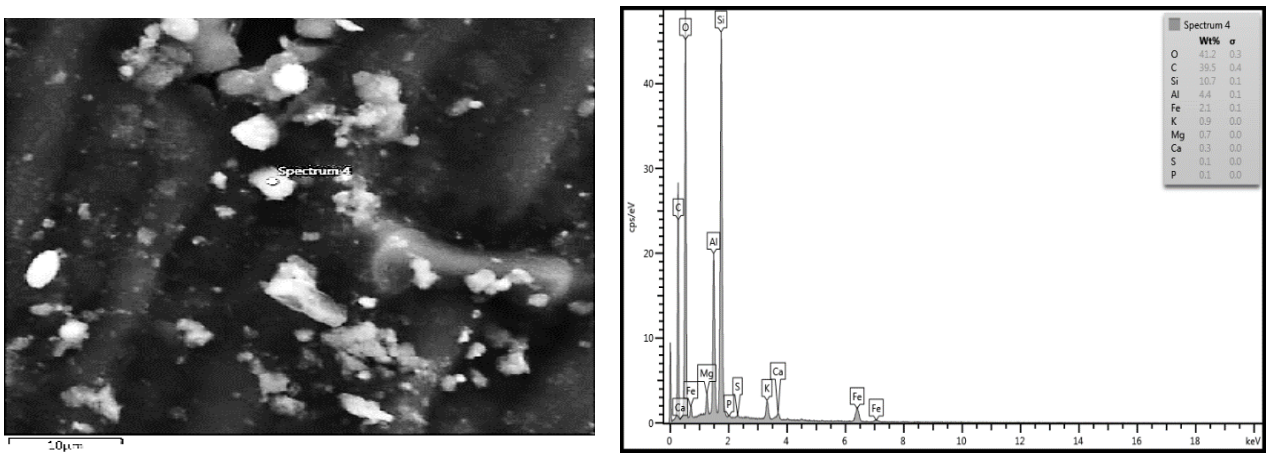

Figure 11. Photomicrograph of 50 wt% OP Particulate Composite as Revealed by SEM/EDS

The elemental compositions of the OP particulates revealed that carbon and oxygen are the main constituents present with traces of other significant elements present like sodium, potassium and iron as shown in the EDS scan of **Figure 9 – 11**. The EDS analysis as was observed also revealed that the OP particles are void of harmful radioactive elements that could endanger human life as was confirmed by the XRD result.

Conclusions

The dielectric properties of OP particulate reinforced polyester composite have been successfully studied. The composite insulator materials show minimal moisture content and minimal water absorption. It also shows appreciable dielectric constant for the 10 wt% = 3.33 and 20 wt% = 3.83, but lower dielectric constant as the filler content increased as compared to standard insulators (like Mica = 4). The values gotten as the dielectric constant (which are less than ten) suggests that the composites can find useful application as insulation materials. Resistivity and dielectric strength had their optimal values at 10 wt% and 40 wt% filler content respectively. Thus, lower filler loadings enhanced stronger particle – matrix interfacial bonding for optimal dielectric properties. The developed OP particulates composite can serve as a candidate material for dielectric purposes under various voltage applications since the properties obtained are in par with that of other insulators.

Acknowledgement-The authors hereby appreciate and acknowledge the Africa Centre of Excellence for Sustainable Power and Energy Development, ACE-SPED, University of Nigeria, Nsukka for their support.

Disclosure statement: *Conflict of Interest:* The authors declare that there are no conflicts of interest. *Compliance with Ethical Standards:* This article does not contain any studies involving human or animal subjects.

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