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The Effect of Filler Variation and Interfacial Behaviour on the Dielectric Performance of Snail Shell Particulate Polyester Composites.

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Abstract: The present experimental study was aimed at determining the dielectric performance of snail shell particulate polyester composites. The snail shell (SnS) materials were acquired, washed, sun dried, milled into powder and sieved into sieve grade of 300 µm. Composites having 10, 20, 30, 40 and 50 wt% weight fraction of snail shell particles were made using hand layup method. X-ray diffractometer (XRD) analysis carried out revealed that the snail shell particles contain the following elements: C, O, Na, Mg, Al, Si, K and Ca. Surface morphology of the snail shell particulate composites as revealed by the SEM confirmed the particles to be solid in nature. The TGA/DTA analysis reveals the thermal stability of the SnS particulate composites. The properties tested and analyzed are: dielectric strength, dielectric constant, resistivity, moisture content and water absorption capacity. The effect of filler variation on the above mentioned properties was studied and used as criteria for the evaluation of the composites. The maximum dielectric strength, dielectric constant and resistivity were observed with the 10 wt%, 30 wt% and 50 wt.% snail shell particulate polyester composite respectively. It was also observed that the 50 wt% sample has the highest moisture content and water absorption values. It showed a progressive increase from 10-50 wt% for the water absorption capacity and moisture content. The measured properties of the snail shell particulate – polyester composites were comparable with some standard insulators. Thus, they can serve as substitute dielectric to the conventional standard insulators in use.

1. Introduction

The science of dielectrics is one of the oldest branches of physics and has close links to chemistry, materials, and electrical engineering. The term dielectric was first coined by Faraday to suggest that there is something analogous to current flow through a capacitor structure during the charging process when current introduced at one plate (usually a metal) flows through the insulator to charge another plate (usually a metal). The dielectric properties or behaviors are the properties that are

being exhibited by dielectric materials (Kim *et al.*, 2008). A dielectric material is a substance that is a poor conductor of electricity, but an efficient supporter of electrostatic fields. It suggests the absence of conduction and describes materials which are not electrical conductors. The study of dielectric properties concerns the storage and dissipation of electric and magnetic energy in materials. (Debasmita and Alok 2012). An important property of a dielectric is its ability to support an electrostatic field while dissipating minimal energy in the form of heat. The lower the dielectric loss (the proportion of energy lost as heat), the more effective is a dielectric material (Adrian 2013). Over time, low dielectric materials have been researched by ceramic and polymer scientists. However, these materials possess numerous electrical, thermal, chemical, and mechanical properties that are just as crucial as the name that classifies them. Therefore, in many cases, the applications of low dielectric constant materials are dictated by these other properties, and the choice of low dielectric material may have a tremendous effect on a device's performance and lifetime (Seung *et al.*). In the field of microelectronics, many of the early low dielectric materials have satisfactorily covered the required properties; but due to continuous growth in the microelectronics industry, more advanced processes and materials have been in demand.

Subsequently, the field of engineering is changing rapidly as a result of introduction of the composite technology. This is as a result of the combination of properties that is possessed by these materials which cannot be gotten by direct convectional materials such as polymer, metal or ceramic. Recently, materials of unique dielectric responses have been studied and utilized in novel ways. For instance, polymer scientists and technologists expanded their horizons from consumer products to the high technology arena. Particularly notable are inventions in telecommunications, where plastic fibers are used for short optical data links, and polymeric films with large third-order susceptibility X_3 are used for nonlinear optics applications (Kakani, 2005). Ceramic dielectrics tend to have very high dielectric permittivity but relatively low dielectric strength. Increasing their dielectric strength is one of the challenges and goals in modern day dielectrics research. The presence of grain boundaries, porosity, impurities, surface defects, and chemical deterioration causes ceramic dielectrics to fail at relatively low field stresses (< 10 kV/mm) (Daniel and Patricia, 2011). Thin film dielectrics are an important area leveraging either polymer or ceramic materials. The thin films are usually in nanometer to submicron in thickness with very high breakdown strength. But, they are primarily useful for low voltage and small size microelectronic application. Certain ceramic materials can be selected to blend with polymers to provide synergy between the high breakdown strength polymer and high permittivity ceramic materials. A number of research areas are being actively pursued to fully explore the advantages of the functional composites (Lewis, 1994).

Recently, some electrical industries have begun converting to polymer composite materials for insulation applications. Composite insulators are less costly, lighter in weight, and have excellent hydrophobic capability. This combination makes them ideal for service in special areas. However, these materials do not yet have the long-term proven service life of glass and porcelain. Hence, the need for further research on how we can utilize agro-based filler (snail shell) in polyester resin matrix to produce a composite for insulation purpose.

Agricultural wastes of both plants and animals in most African countries are enormous and often constitute environmental menace (Igwe, 2007). Most of these animal wastes (for instance snail shell) could be used as reinforcements in developing composite materials. Finding a material that supports their applications in electrical industries will enhance better recycling of these wastes. Thus, this research fabricated a composite material from snail shell particulate in polyester matrix for application in dielectric (power transmission insulation).

Snail Shell is a naturally occurring outer shell covering of a snail (Turritella communis). It is an external exoskeleton which protects the snail from their predators and mechanical damage. Structurally, snail shell has several layers and is typically made of an organic matrix (conchiolin) which is bonded with calcium carbonate precipitates. This calcium carbonate-filled organic matrix shell are impervious to water and this property makes it possible for snail shells and their derivatives to have very good applications in making composite for dielectric applications (Painter and Hemmer, 1979). Snail shell is a domestic waste obtained after snail meat has been removed. The shell is brownish in colour with dark brown markings. Snail shell is usually very hard and protects the snail from predators, dehydration and physical damage. Locally, snail shell is used in the manufacture of jewels, buttons and collections for arts (Jatto *et al.*, 2013). The shells are known as rich source of calcium and have been used as fillers in the ceramic industry, paint, animal feed, construction and paper industry (Jatto *et al.*, 2013). Snail shell has limited domestic application in our locality, and could be found littering dust bins in our big cities and farm yards in villages. However, effectively recycling and utilizing of this local cheap mineral particulate can bring immense economic prosperity. This work investigated the potency of snail shell particles as reinforcement in polyester matrix. Snail shell particulate, if found to be good reinforcement in polyester matrix for the required application, besides the technical benefits, our environment would be rid of some solid pollutants and our gross domestic product would improve. It is expected that this work will play an increasing role, in conforming to the desired dielectric properties for the intended application.

Earlier studies by some authors revealed that quality fibres and particulates suitable as reinforcement in composites can be produced from maize stems and hulls, cow bones, cassava cortex, rice straw and husk, banana bunch, wheat straw, sorghum stems and leaves, pineapple, coconut shells, nettle, velvet, empty palm fruit bunch, hemp, flax, kenaf, cotton, soy hulls and straw, milk weed stems and bamboo (Gattenholm, *et al.*, 1993, Fuad, *et al.*, 1998). Example include the work of (Inegbenebor and Adeniji, 2007), who investigated the possibility of using agro-waste materials: shells of coconut, mango endocarp, palm kernel, groundnut and bean as well as corncob and rice husk, as electrical insulators. The results showed that the electrical insulation properties of these materials were comparable with the known standard values.

Snail shell powder is important filler in the paper industry because of its calcium carbonate and chitin contents. It is used to increase the mechanical properties of paper. Such properties include: moisture resistance, smoothness, abrasion, machine flowability, brightness, strength and opacity (John *et al.*, 2016). Snail Shells in powdery form has also been used in the ceramic industry in the manufacture of breakable plates, pipes and kitchen utensils (Israel *et al.*, 2016). Despite the above mentioned studies by researchers on industrial and agro – waste materials like snail shell as reinforcing fillers for composites, the novelty of snail shell waste-based polymer composites in terms of dielectric properties, has not been well explored. This implies that snail shell particulate composite dielectric properties have not been well studied. Based on this, this study aimed at producing an insulator from snail shell particles using polyester as the matrix and characterization of the dielectric properties of the produced insulator to ascertain its efficacy for application in electrical and electronics industries.

2. Methodology

2.1 Sourcing and preparation of materials

Snail shells were sourced from Enugu locality and processed into powder, 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 used as purchased from a vendor Chemical shop at Enugu. 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.

The shells were cleaned and sun dried for seven days; after which it was ground and sieved into particle size of 300 μm. The unsaturated polyester resin with 0.1% catalyst and acelerator were vigorously stirred before adding various weight fractions (10wt% - 50wt%) of snail shell reinforcement. For every 100wt% of the composite composition, the ratio of the polyester to fibre was 9:1, 4:1; 7:3; 3:2 and 1:1. The stirring was vigorous to avoid agglomeration of the particles. The prepared mixtures were cast into moulds with release agent applied. The mixture was allowed a curing period of 24hours and the overflow flakes were cut off. The fabricated samples are shown in figure 2; and they were subsequently prepared for various tests. Figures 1 and 2 represents the raw snail shell and the fabricated samples respectively.

Figure 1. Snail shells/crushed snail shell

Figure 2. Fabricated composite samples for dielectric measurements

2.2 *Characterization*

TGA was carried out on the samples to determine the degradation temperature and thermal stability of the material. Sample size is 6.9850mg. All the samples were scanned from 50 °C to 1000 °C at a heating rate of 20 °C/min under N_2/O_2. 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 in studying the micrographs of the polyester composite samples with 10%, 30% and 50% snail shell reinforcement; and the elemental compositions measured using EDS.

2.3 Experiments

Dielectric strength in kV/mm was measured using alternating current dielectric strength tester. Cylindrical specimens of 18 mm diameter and 105 mm length were placed between two 10 mm diameter copper ball electrodes and the electrode system containing the measured sample was covered with glass screen to prevent the surface flashover from splashing out. The test voltage was applied

across two ball-typed electrodes and was increased until the specimen failed at a given voltage. The failure was characterized by smoking.

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 the two parallel plate capacitors with same thickness as the composite sample. The parallel plate capacitors were connected to the battery and the voltage across was measured (V_o). The samples were then used separately to fill the air gap between the capacitor plates that are connected to a battery and the voltage across was also measured differently for each of the composite samples.

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. To test for water absorption capacity, the samples were immersed in water for a period of one week and then allowed to drain by gravity. The weights (M_1) and (M_2) of the samples were measured before and after the absorption and after draining process respectively. For the moisture content, the samples were placed in the oven at a temperature of 105 ˚C and their weights measured at an interval of an hour until a constant mass (M_3) was obtained.

3. Results and Discussion

3.1 *Dielectric strength*

The dielectric strength in kV/mm was estimated after the measurement. Figure 7 shows a plot of dielectric strength (kV/mm) versus weight % of snail shell reinforcement. It is observed that the values of the dielectric strength decreased as the percentage of filler loading increased. The reason is that smaller wt% will disperse evenly in the matrix than higher wt% thereby enhancing better wettability between the reinforcement and the matrix, thus enhanced interfacial bonding between the particulate and the polyester resin. This is in agreement with the work of (Saira 2011). Better enhancement of property (i.e. dielectric strength) was obtained for the composites with lower wt%. Moreso, as the filler content is increased, the number of particles in the composite is more with the inter-particle distance being smaller, hence the volume fraction of loose polymer layer reduces and the particles themselves act as barriers to flow of current between the electrodes. These factors contribute to hindrance in the flow of current in the composite, resulting in higher dielectric strength.

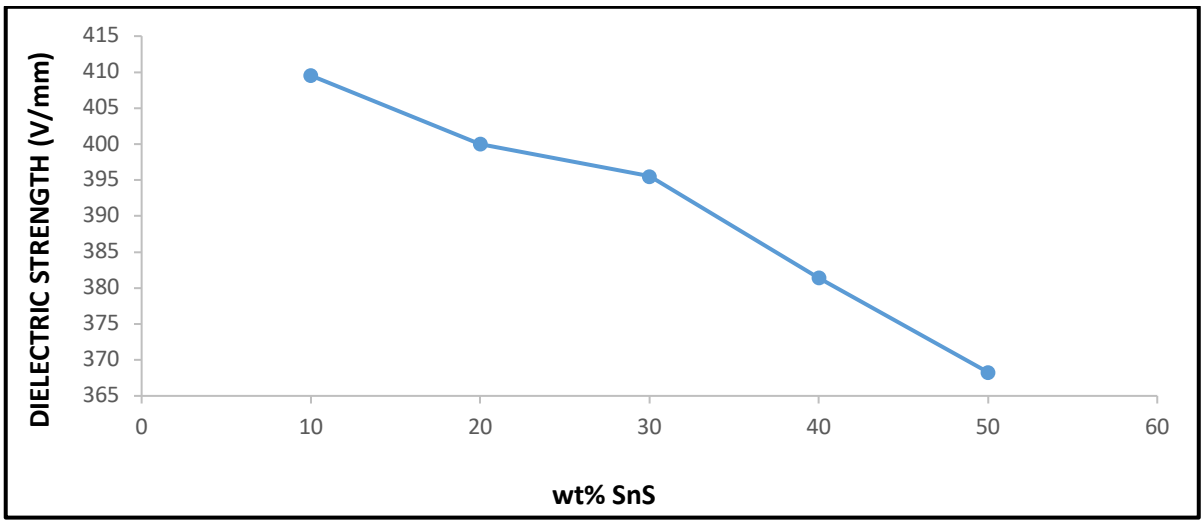

Figure 7. Dielectric strength of SnS polyester composite

3.2 Dielectric constant

Dielectric constant ($\mathcal{E}_r$) relates to the permittivity of the material ($\mathcal{E}$). The permittivity of a material expresses the ability of a material to polarize in response to an applied field. This implies that 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 ($\mathcal{E}_o$). The dielectric constant ($\mathcal{E}_r$) was estimated by considering the voltage across parallel plate capacitor with an air gap (V_o) and the voltage across the parallel plate capacitor with the composite sample in between them (V) using equation 1:

$$\mathcal{E}_r = \frac{V_0}{V} \qquad \textbf{Eqn. 1}$$

Figure 8 is a graph of dielectric constant against wt% SnS. The graph shows an initial increase in dielectric constant as the SnS increased but eventually started decreasing as SnS further increased, with the highest constant corresponding to the 30 wt%. This is as a result of each wt% addition leading to increase in the number of dipoles per unit volume, which means increase in the number of polarized dipoles that orients due to the effect of electric field. However, beyond the 30 wt% composition, the observed decrease in dielectric constants of the composites might have resulted from poor interfacial bonding which as well led to increased pores in the samples; since the presence of free volume in the form of pores resulted in a decrease in dielectric constant as the relative permittivity of air is about one (Simpson and Clair, 1997). This is in agreement with the work done by (Jie *et al*., 2008). The SnS particle size used was 300 µm. The particle size used enhanced the dielectric constant due to unrestricted polarization of the particles. The small grain size (300 µm) used made room for the accommodation of numerous particles in the resin, which means increase in the number of polarized dipoles that orients due to applied electric field.

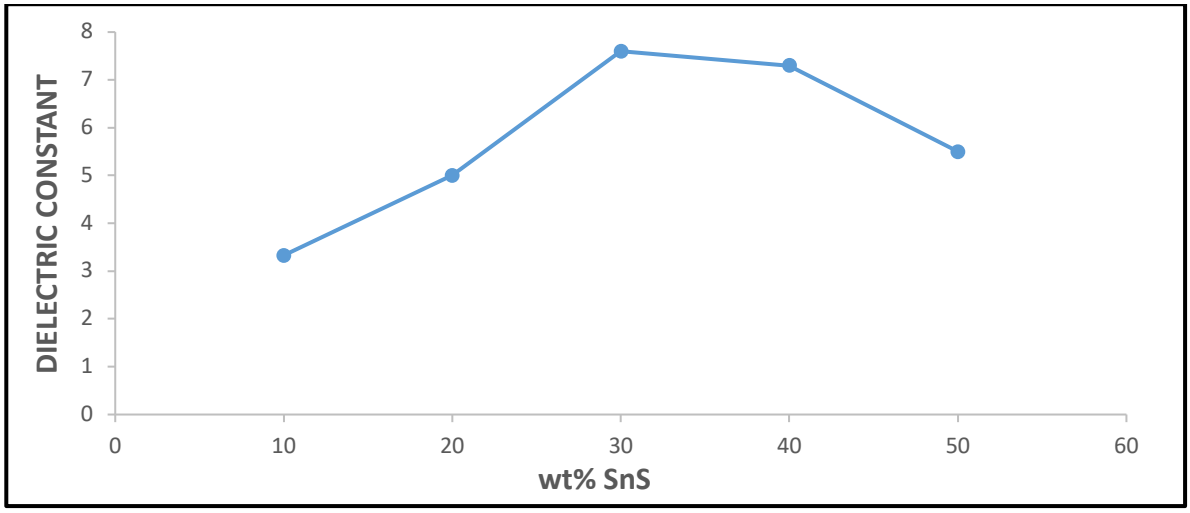

Figure 8. Dielectric constant of SnS polyester composite

3.3 Resistivity

The graph of resistivity against wt% SnS of the composite is represented in figure 9. The figure shows an increase in resistivity with the incorporation of fillers. This behavior indicates that at lower filler loading, the fraction of extended loose polymer layers is high which probably allows the existence of

free electrons and also their unhindered transport through the bulk of the material, thus, increase in the electrical conductivity (low resistivity) through the volume of the material, but at higher filler loading, the compacted particles probably hinder the free movement of electrons thereby enhancing the resistivity of the composite material. However, there is an unusual increase in resistivity value for the 20 wt% sample. This could be due to some human error either from sample preparation or property measurement.

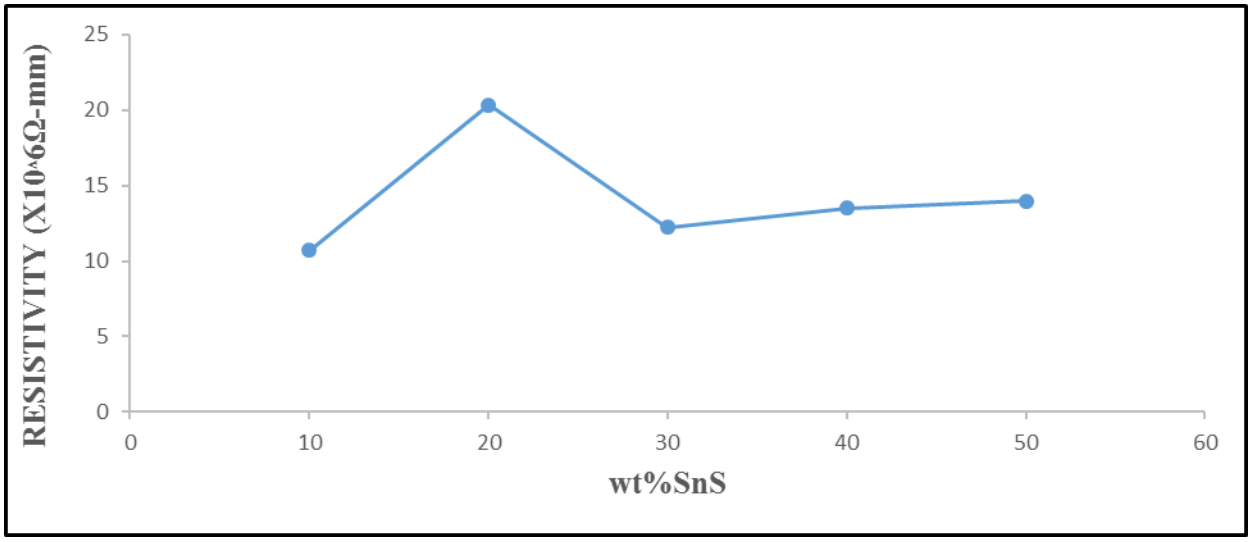

Figure 9. Resistivity of SnS polyester composite

3.4 Determination of water absorption and moisture content

From the measured weights, the water absorbed (W_A) was calculated as percentage weight gain using equation 2 below according (ASTM D570-98, 2018)

$$W_A = \frac{M_2 - M_1}{M_2} \times 100 \qquad \textbf{Eqn. 2}$$

The moisture content (M_C) was calculated as percentage of the dry sample from equation 3:

$$M_C = \frac{M_1 - M_3}{M_1} \times 100 \qquad \textbf{Eqn. 3}$$

Figure 10 shows the water absorption capacity/moisture content with increasing wt% of SnS. It was observed that water absorption increased with increase in percentage of SnS particle fillers. This was because of poor interfacial bonding as more filler wt% is increased which resulted in an increase in the number of micro-voids due to lack of sufficient resin for strong bond formation, thus, causing increased water absorption and poor dielectric properties (Abdullah *et al*., 2011, Omah *et al*., 2017). Similarly, the moisture content also increased with increased particulate filler due to the presence of free water present in the snail shell particles. Thus, 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.

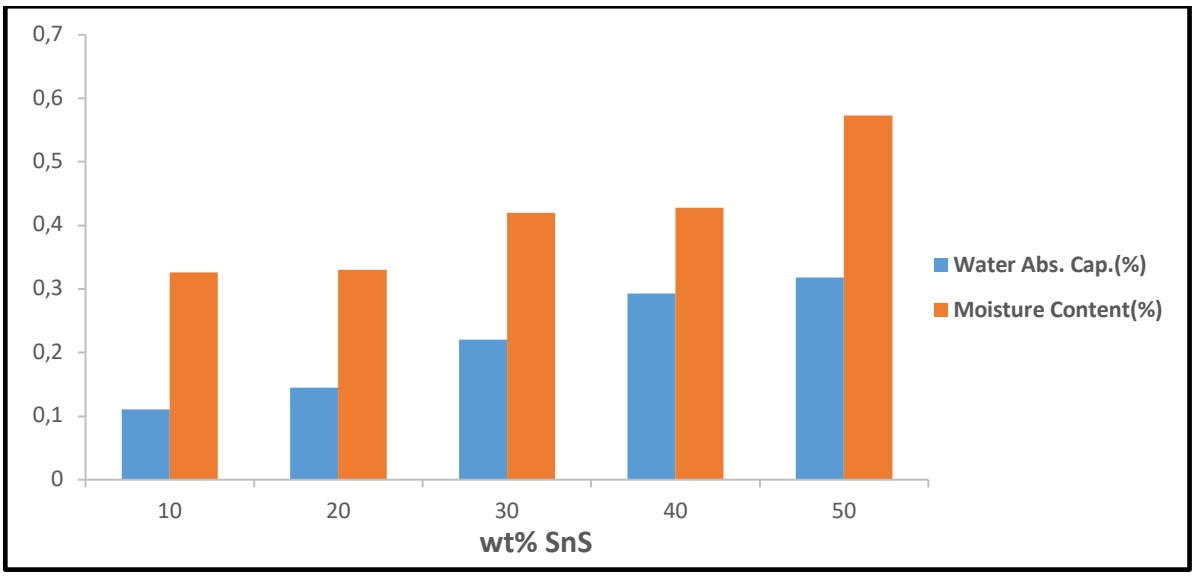

Figure 10. The water absorption capacity and moisture content of SnS polyester composite

3.5 Thermo-gravimetric analysis (TGA)

The thermo-gravimetric analysis data collected on the SnS are shown in Figure 6. Three areas of major weight changes in the TGA graph are observed. The first occurred at around 100 °C resulting from evaporation of water. The major change in weight is observed between 200 °C and 450 °C, this is as a result of decomposition of cellulose and other decomposable organic matters contained in the shell. The thermal stability after this temperature maintained a slow decrease i.e. from 500 °C until final degradation occurred at above 800 °C. At this stage, the graph assumed almost a linear nature until the point of termination of the experiment.

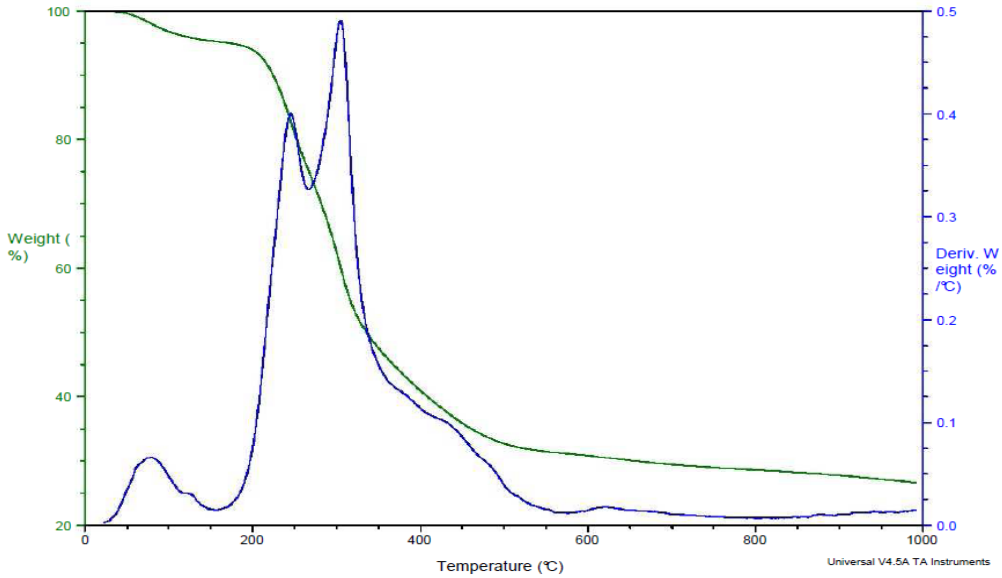

Figure 11. Thermo-gravimetric analysis (TGA) of SnS polyester composite

3.6 XRD

The XRD pattern of the SnS shown in figure 6 revealed that phases present were: silicon oxide (SiO_2), dolomite $CaMg(CaCO_3)_2$, calcite ($CaCO_3$) Diopside, Ca(Mg,Al) alumina, and syn$(Si,Al)_2O_6$.; these phases have the following scores 48, 21, 19, 15 and 16, with scale factors of 0.743, 0.432, 0.236, 0.093 and 0.232. These represent the relative amount of each phase in the XRD pattern displayed in Figure

12 and Table 1. SiO_4 has the highest amount with a score of 48 and a scale factor of 0.743. The major diffraction peaks were observed at 2θ positions: 20.96, 26.71, 29.86, 30.88, 36.65 and 67.73 degrees, while their corresponding inter-planar distances were: 4.24, 3.34, 2.99, 2.90, 2.45 and 1.38 Å respectively (Table 2). The FWHM, d-spacing, intensity of these phases are listed in table 2. The identified phases from the x-ray pattern suggest that the fabricated composite is an agro-based material. Agro-based materials usually contain at least one of these elements: C, O, Na, Mg, Al, Si, K, Ca, and their existence in the fabricated composites is confirmed from the SEM/EDS scan results of figures 13 – 15. Also, from the identified phases, it is clear that snail shell particles belong to the calcium family and does not contain toxic and harmful materials.

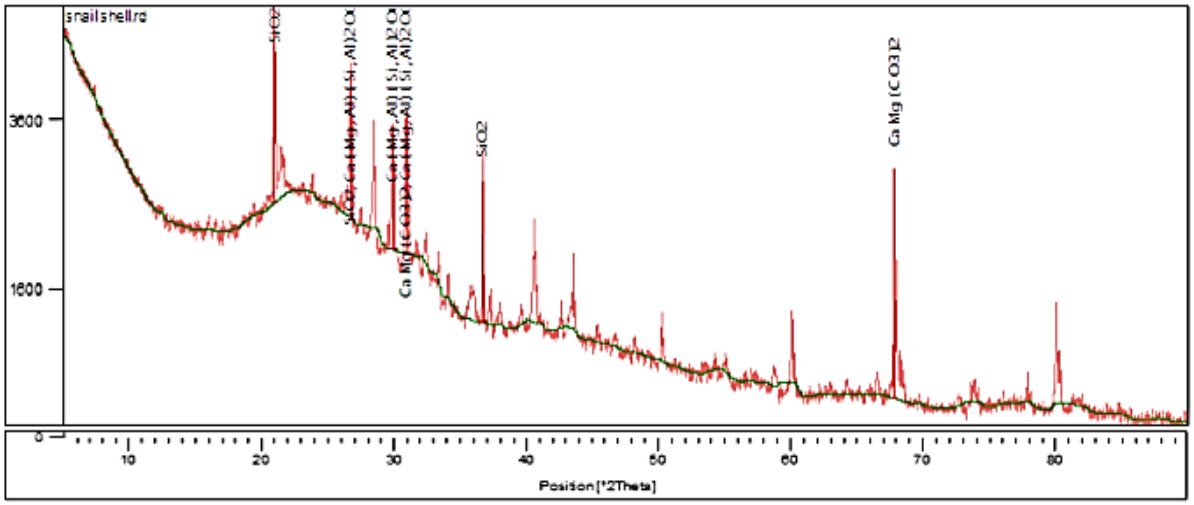

Figure 12. X-ray diffraction pattern of SnS powder.

Table 1. Identified patterns list of the elements present

Visible	Ref. Code	Score	Compound Name	Scale Factor	Chemical Formula
*	85-0794	48	Silicon Oxide	0.743	SiO_2
*	74-1687	21	Dolomite	0.432	$CaMg(CO_3)_2$
*	05-0622	19	Dolomite	0.236	$CaMg(CO_3)_2$
*	72-1652	15	Calcite	0.093	$CaCO_3$
*	25-0154	16	Diopside, aluminian, syn	0.232	$Ca(Mg,Al)(Si,Al)_2O_6$

Table 2. XRD peak list

Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]	Tip width [°2Th.]
20.9561	2933.09	0.1004	4.23921	100.00	0.1020
26.7126	1993.83	0.1338	3.33731	67.98	0.1360
29.8626	1519.83	0.1673	2.99207	51.82	0.1700
30.8778	1771.86	0.1673	2.89596	60.41	0.1700
36.6464	1496.47	0.1338	2.45227	51.02	0.1360
67.7328	2217.16	0.1020	1.38230	75.59	0.0850

3.7 SEM/EDS

The SEM micrographs and the EDS graphs of the 10 wt%, 30 wt% and 50 wt% composite are shown in **figures 13, 14 and 15** respectively. The SEMs show that the reinforcements are solid particles and are randomly distributed; the micrographs also show the particles in white while the polyester resin matrix is dark in colour. It is seen that the white parches increased with increase in SnS particles. It is also evident from the micrograph that the SnS particles agglomerated a little in spite of the vigorous manual stirring. The shapes of the particles vary: the particles with smooth spherical surface had more surface area for interaction with the matrix thus, good particle – matrix interfacial strength for enhanced dielectric properties. The SEM/EDS for 30 wt% and 50 wt% revealed the presence of more particles than that of 10 wt%.

The elemental compositions of the SnS particulates revealed that aluminum is the major constituent present as shown in the EDS results. There are also high traces of other significant elements present like, silicon, iron, manganese, carbon, oxygen, calcium, copper and nickel. The EDS analysis also revealed that the SnS particles did not contain radioactive elements that could be detrimental to the human body.

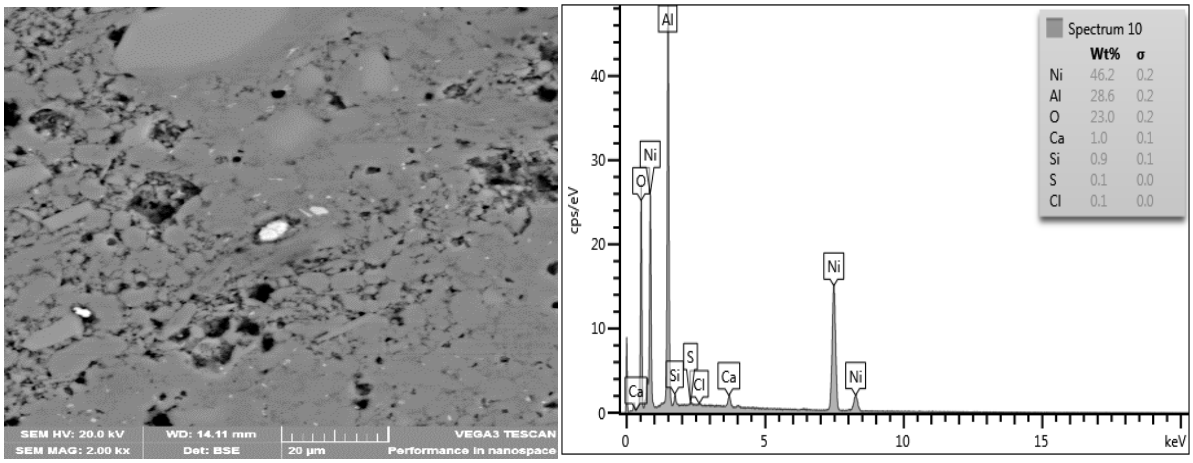

Figure 13. SEM/EDS of 10 wt% SnS polyester composite.

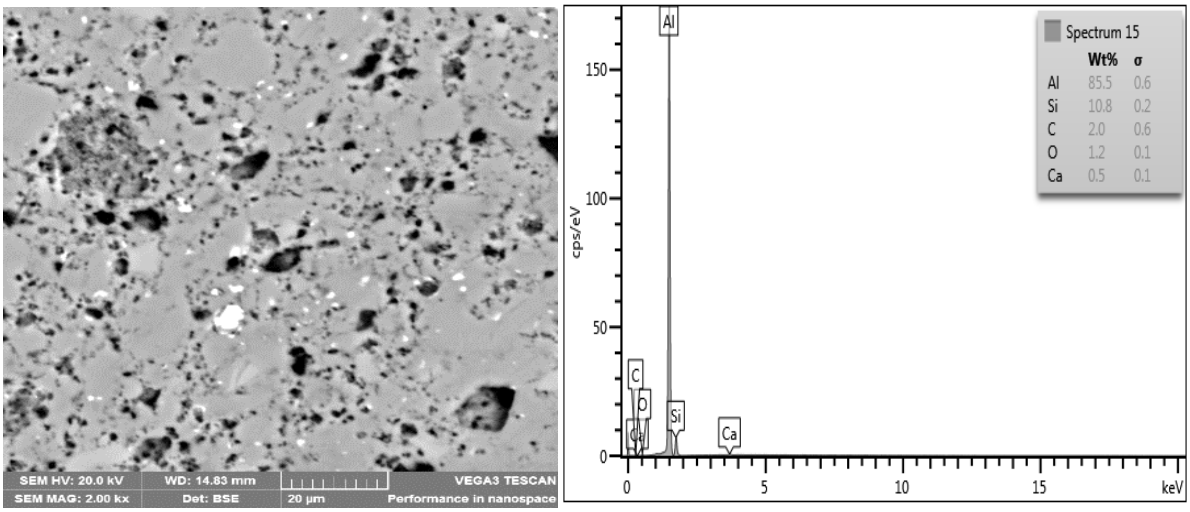

Figure 14. SEM/EDS of 30 wt% SnS polyester composite.

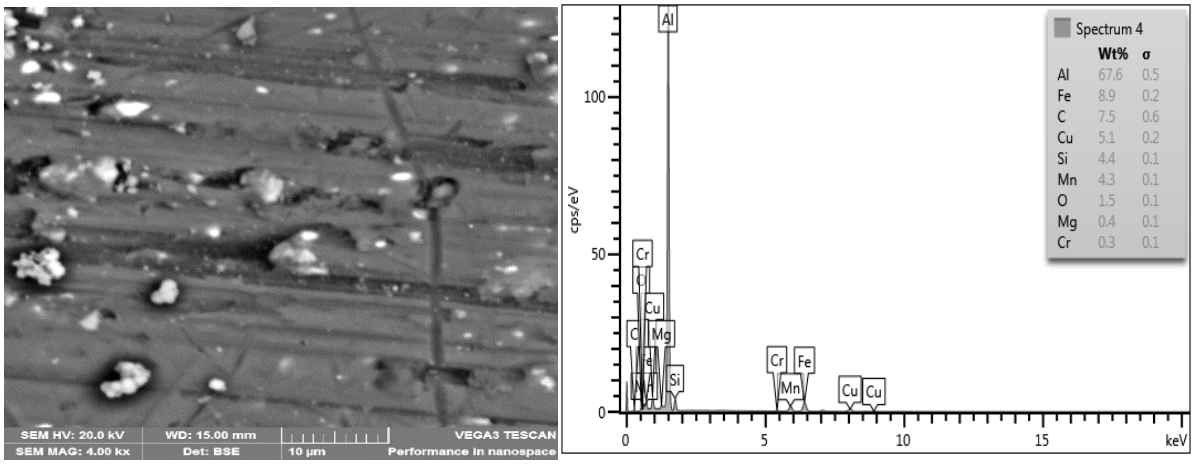

Figure 15. SEM/EDS of 50 wt% SnS polyester composite.

Conclusions

The dielectric properties of snail shell polyester composite were studied. The study revealed that by incorporating snail shell as reinforcement into polyester matrix, it can be used in the production of dielectric materials. The composite insulator material shows good dielectric properties, low moisture content and low water absorption capacity. The dielectric constant values (which lies between 3 and 8) signifies that the composites can be used as insulation materials and as dielectrics in capacitors. Dielectric strength and dielectric constant had their optimal values at 10 wt% and 30 wt% particulate content respectively. Thus, lower filler loadings enhanced stronger interfacial bonding between the resin and particulates for optimal dielectric properties. The samples are also considered to be environmentally friendly insulating materials.

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Disclosure statement: *Conflict of Interest:* The authors declare that there are no conflicts of interest.

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