

# Measurement of dielectric properties of polymer matrix composites developed from cow bone powder

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**Abstract** This research was focused on determining the dielectric properties of composite materials developed from carbonized and uncarbonized agrowaste (cow bone) material. The uncarbonized materials were washed, air dried, and part of the materials were carbonized at a temperature of 750 °C after which they were milled into powder and sieved into sieve grades of 150, 300, and 600 μm. The cow bone particles were varied from 40 to 60 wt%. A mineralogical analysis carried out by the X-ray diffractometer revealed that the carbonized and uncarbonized bone particles contain the following elements: O, Na, Mg, Al, Si, Ca, and C. Surface morphology of the carbonized and uncarbonized particles as revealed by the SEM confirmed the particles to be regular and solid in nature. The TGA/DTA analysis shows that the carbonized particles were more thermally stable than the uncarbonized particles were. The dielectric strength, dielectric constant, and water absorption capacity were determined. The effect of carbonization on the abovementioned properties was studied and used as criteria for the evaluation of the composites. The results shows that smaller particles and weight percentage have the best properties: example at the 40 wt% of 150 μm, the dielectric strength for the CCB and UCB are 217.96 and 510.84 Kv/mm, respectively. The same trend was observed in all the compositions and all the particle sizes. Better enhancement of property was obtained for the composites with lower wt% and smaller particle sizes compared to the higher wt%

and bigger particle-sized composites. The significant factors (main and interaction) were identified by analysis of variance technique. The Cube and 3D graphs show the estimated response surface for the composite properties as a function of sample condition, wt%, and particle size. It is also evident from the results that the actual experimental values are in close proximity with the predicted values.

**Keywords** Dielectric · Carbonization · Composites · Agrowaste · Cow bone

## 1 Introduction

The rapid development of the electronic information industry has led to the demand for better properties for substrate and packaging materials, such as low coefficients of thermal expansion (CTE), low dielectric constant, and high thermal stability [1]. Polymers, such as polyethylene, epoxy, and polyamide, are ordinarily used as these materials due to their high resistivity, low dielectric constant, and excellent processability. However, these polymers suffer from disadvantages such as low thermal conductivity, high CTE, and low stiffness and strength [2]. To offset these deficiencies, adding inorganic particles (such as agrowaste materials) to a polymer is a versatile method. This method synergistically integrates the advantages of polymers and inorganic fillers; and thus, the thermal, electrical, and mechanical properties of the composites can be improved by properly selecting the fillers, their shape, size, and concentration [3, 4]

Every material has a unique set of electrical (and thermal) characteristics that are dependent on its dielectric properties. Accurate measurements of these properties can provide scientists and engineers with valuable information to properly incorporate the material into its intended application for more

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solid design and usage [5, 6]. Furthermore, a dielectric material's measurement can provide critical design parameter information for many electronic applications. For example, the loss of a cable insulator, the impedance of a substrate, or the frequency of a dielectric resonator can be related to its dielectric properties. The information is also useful for improving ferrite, absorber, and packaging designs. More recent applications in the area of industrial microwave processing of food, rubber, plastic, and ceramics have also been found to benefit from knowledge of dielectric properties [7].

A dielectric is an electrical insulator that can be polarized by an applied electric field. When a dielectric is placed in an electric field, electric charges do not flow through the material, as in a conductor, but only slightly shift from their average equilibrium positions causing dielectric polarization. Because of dielectric polarization, positive charges are displaced toward the field and negative charges shift in the opposite direction. This creates an internal electric field which reduces the overall field within the dielectric itself [8, 9]. If a dielectric is composed of weakly bonded molecules, those molecules not only become polarized but also reorient so that their symmetry axis aligns to the field [10]. When dielectrics are placed in an electric field, practically no current flows in them because, unlike metals, they have no loosely bound or free electrons that may drift through the material. Dielectrics are not a narrow class of so-called insulators, but rather are the broad expanse of nonmetals considered from the standpoint of their interaction with electric, magnetic, or electromagnetic fields. Thus we are concerned with gases as well as with liquids and solids, and with the storage of electric and magnetic energy as well as its dissipation [11]. In view of the above statement, dielectric materials can be solids, liquids, or gases. In addition, a high vacuum can also be a useful, lossless dielectric even though its relative dielectric constant is only unity. Solid dielectrics are perhaps the most commonly used dielectrics in electrical (and electronic) engineering, and many solids are very good insulators. Some examples include porcelain, glass, and most plastics. Air, nitrogen, and sulfur hexafluoride are the three most commonly used gaseous dielectrics. Liquid dielectrics include the following: industrial coatings, mineral oil, castor oil, etc. The novelty of this work is to further exploit

the properties of the agrowaste materials: animal bone (cow bone) so as to complement their existing areas of application as well as determine new areas of application such as their use as dielectric materials. From previous works done by researchers on the electrical insulating properties of some agrowaste materials, there is dearth of information on the effect of carbonization on the electrical insulating properties of the materials. Thus, this work will show the relevance of carbonization on agrowaste materials with respect to the dielectric properties of the materials.

## 2 Materials and method

### 2.1 Materials/equipment

Epoxy (matrix), cow bone, stain remover (water soluble), set of sieves (mesh sizes 150, 300, and 600  $\mu\text{m}$ ), hacksaw, universal tensile testing machine, Digital Rockwell hardness machine, Charpy Impact testing machine, measuring cylinder, Vernier caliper, bone crusher, digital weighing balance, scanning electron microscope, Digital Mega Insulation Tester MS360, heat treatment furnace, and Switch and Control Desk for high voltage 5RP0.5/5Trg were used in this work.

### 2.2 Method

#### 2.2.1 Preparation of the cow bone

The cow bone used was sourced locally from Orba market in Udenu Local Government Area, Enugu state, Nigeria. It was collected, washed, and sun dried for 2 weeks (Fig. 1). The bone particles were degreased with a water soluble stain remover (acetone) to remove any trace of marrow, blood, and other substances that will inhibit proper bonding between the epoxy matrix and the reinforcement particles. The cow bone was carbonized (CCB) at a temperature of 750  $^{\circ}\text{C}$  in the absence of oxygen using a heat treatment furnace. The cow bone was then milled after carbonization to the required granules and sieved to particle sizes of 150, 300, and 600  $\mu\text{m}$  in order to obtain different particle sizes for the research.

**Fig. 1** a Cow bone under sun drying. b Sieved carbonized cow bone



a



b

### 2.2.2 Characterization of the bone particles

The characterization of the cow bone particles was investigated through X-ray diffractometer (XRD) and thermogravimetric analysis (TGA/DTA). The XRD, scanning electron microscopy (SEM), and TGA/DTA tests were carried out at the University of Witwatersrand, Johannesburg, South Africa. The X-ray diffractograms were taken using Cu K $\alpha$  radiation at scan speed of 3°/min. The SEM JEOL JSM-6480LV was used to identify the surface morphology of the particles. Thermal decomposition was observed in terms of global mass loss by using a TA Instrument TGA Q50 thermogravimetric analyzer. This apparatus detects the mass loss with a resolution of 0.1 as a function of temperature. The sample was placed in an open sample pan of 6.4 mm diameter and 3.2 mm deep. The temperature change was controlled from room temperature (30  $\pm$  5 °C) to 900 °C at a heating rate of 10 °C/min.

### 2.2.3 Moulding

The material used for preparing the moulds is aluminum bars. The aluminum bars were used for producing rectangular moulds and cylindrical shaped plastic pipes were also used. The mould was coated with a releasing agent for easy removal of specimens from the mould after curing. The samples were cured at room temperature for about 24 h and then they were removed from the mould and finished to the size by filing the edges. For each composition, different specimens were prepared for different test analysis (see Table 1). The low temperature curing epoxy resin (Araldite LY 556) and corresponding hardener (HY951) are mixed in a ratio of 2:1 by weight as recommended [6] and then cow bone particles were added; proper stirring was done to ensure a homogenous mixture after which it was immediately poured into the mould. The mixture was allowed to cure properly in the mould and was removed for various test analysis (see Fig. 2).

**Table 1** The sample compositions

S/N	Designation	Composition
1	150 $\mu$ m cow bone particulate	40 % epoxy + 20 % hardener + 40 % particulate
		33 % epoxy + 17 % hardener + 50 % particulate
		27 % epoxy + 13 % hardener + 60 % particulate
2	300 $\mu$ m cow bone particulate	40 % epoxy + 20 % hardener + 40 % particulate
		33 % epoxy + 17 % hardener + 50 % particulate
		27 % epoxy + 13 % hardener + 60 % particulate
3	600 $\mu$ m cow bone particulate	40 % epoxy + 20 % hardener + 40 % particulate
		33 % epoxy + 17 % Hardener + 50 % particulate
		27 % epoxy + 13 % hardener + 60 % particulate

### 2.2.4 Determination of dielectric strength

The test was carried out at 50-Hz frequency and at room temperature. The cylindrical sample of 25 mm of diameter was placed in a dielectric testing machine, and an impulse voltage was gradually applied to the sample from the control desk until the specimen fails at a given voltage. The failure was characterized by a loud sound. The value at which the material failed was recorded and the dielectric strength was calculated by dividing the breakdown voltage by the thickness of the specimen. The breakdown voltage of all the specimens were recorded via the same procedure and their corresponding dielectric strength calculated as well.

### 2.2.5 Determination of water absorption capacity

The water absorbed by the material is specified as the percentage weight gained by the material. The samples were weighed to the nearest 0.01g ( $M_1$ ) and immersed in water for a period of 2 weeks. It was subsequently removed from the water and allowed to drain by gravity before the sample was weighed again ( $M_2$ ). The water absorbed was calculated as percentage weight gain using the formula below: [10].

$$W_A = \left( \frac{M_2 - M_1}{M_1} \right) \times 100 \quad (1)$$

### 2.2.6 Determination of dielectric constant

To determine the dielectric constant, the composite samples were moulded into rectangular plates of length 50 mm, width 30 mm, and thickness 2 mm. It comprises of two parallel plate capacitors, DC battery, and a digital multimeter for measuring the applied voltage across the samples. An air gap was created between the two parallel plate capacitors which has the same thickness as that of the composite sample. The parallel plate capacitors were connected to the battery and the voltage across was measured ( $V_0$ ). The samples were then used separately to fill the air gap between the capacitor plates that are

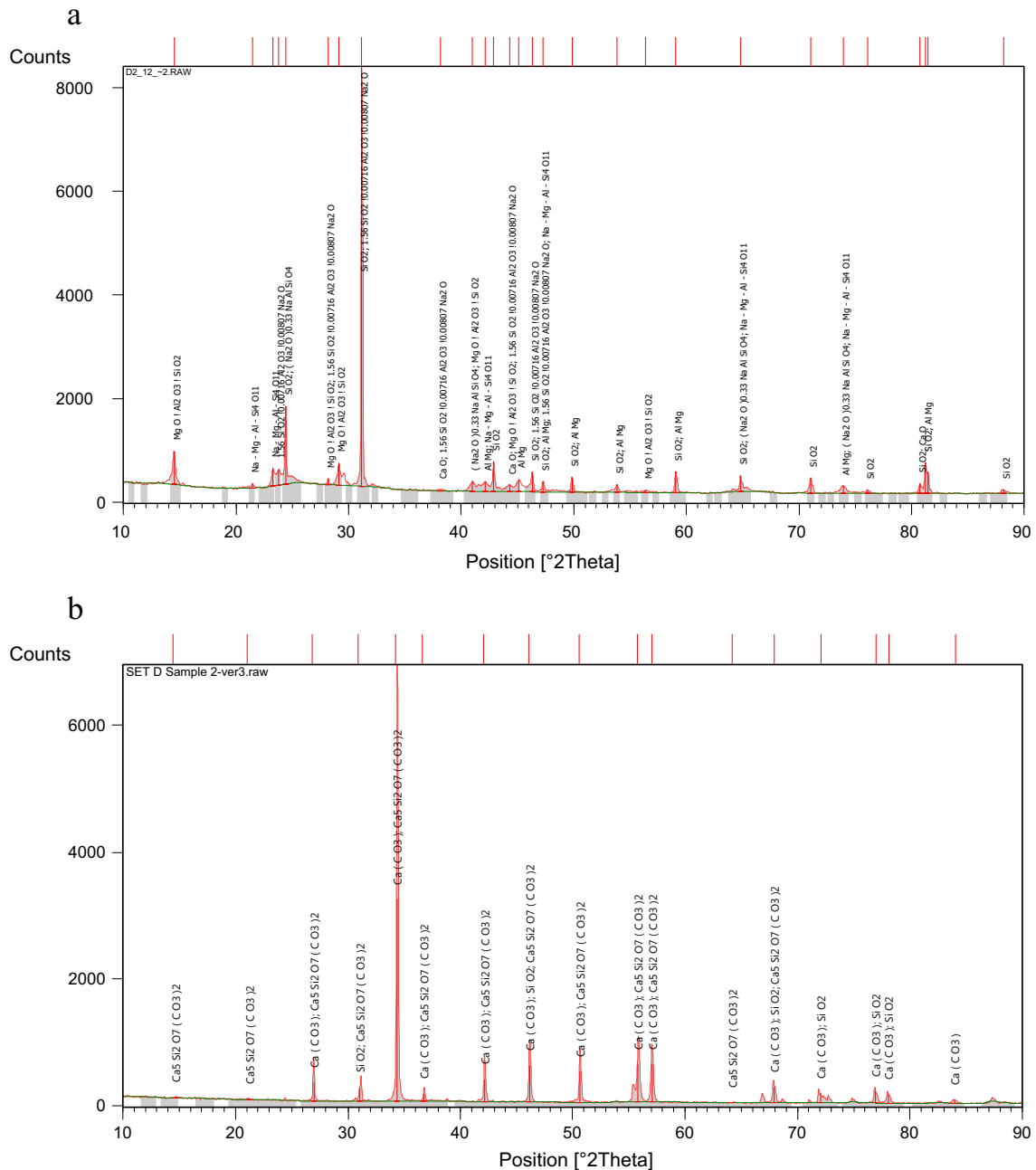
**Fig. 2** Showing the cast samples for the composite



connected to the battery, and the voltage across was also measured differently for each of the composite sample. Given below is the relationship between the  $V_o$  (voltage across the capacitors with an air gap between them) and  $V$  (voltage across the capacitors with the composite sample between

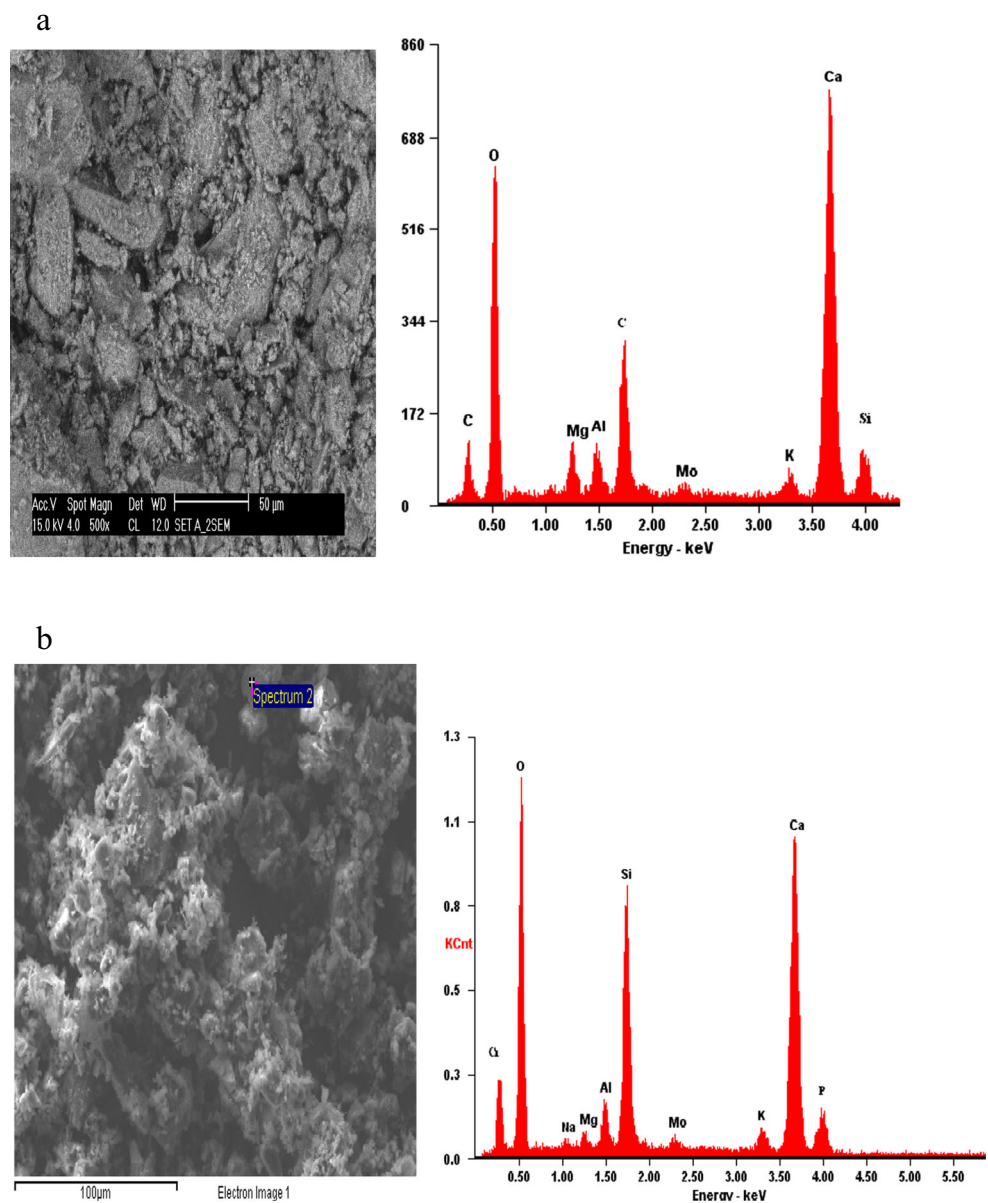
them). This relationship between them gives the dielectric constant, thus

$$\epsilon_r = \frac{V_o}{V} \tag{2}$$



**Fig. 3** **a** XRD spectrum of CCB particles. **b** XRD spectrum of UCB particles

**Fig. 4** **a** SEM/EDS of the CCB particle. **b** SEM/EDS of the UCB particles



$V_0$  is the voltage across parallel plate capacitor with an air gap

$V$  is the voltage across parallel capacitor with the sample in between the two plates

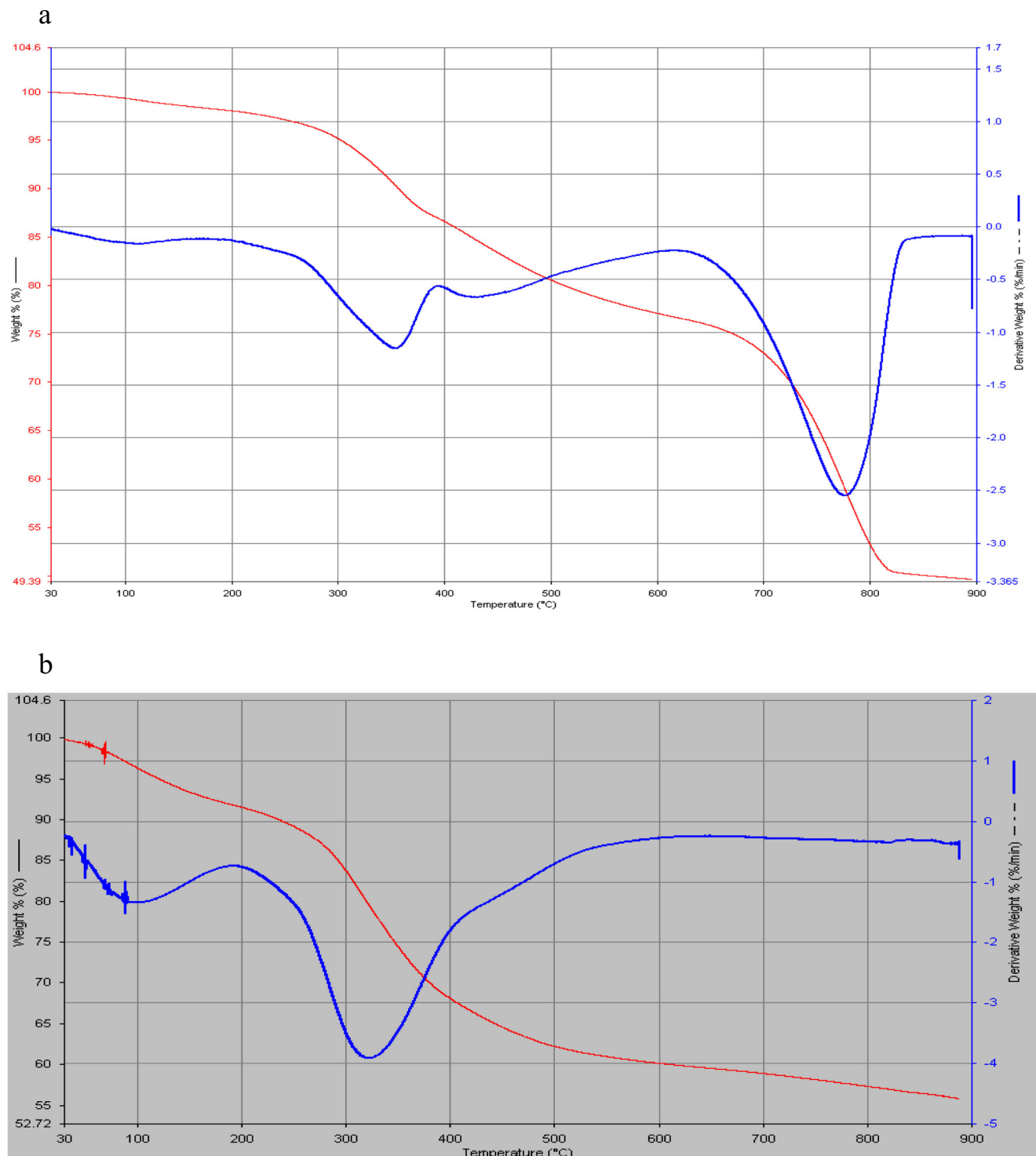
### 3 Results and discussion

#### 3.1 Compositional analysis and microstructure of the cow bone

The XRD pattern of the cow bone (Fig. 3) revealed phases of quartz low ( $\text{SiO}_2$ ), lime ( $\text{CaO}$ ), aluminum, magnesium ( $\text{AlMg}$ ), sodium aluminum silicate ( $\text{NaAlSiO}_4$ ), magnesium

aluminum silicate ( $\text{MgOAl}_2\text{O}_3\text{SiO}_2$ ), sodium aluminum silicate, and montmorillonite ( $\text{NaMgAlSi}_4\text{O}_{11}$ ). From this analysis, it is seen that the bone particles have similar characteristics with other agrowaste particles now used in polymer composites. These results show that the carbonized cow bone (CCB) and uncarbonized cow bone (UCB) particles do not contain harmful radioactive elements that could be cancerous and harmful to the human body. The major difference between the XRD pattern of CCB and UCB is the presence of carbon in the CCB.

Surface morphology of the CCB and UCB particles as revealed by the SEM are seen in Fig. 4a, b. The particles were observed to be solid in nature. From the EDS spectrums, it can be seen clearly that the elemental compositions of the CCB and UCB particulates were revealed. It is obvious that apart

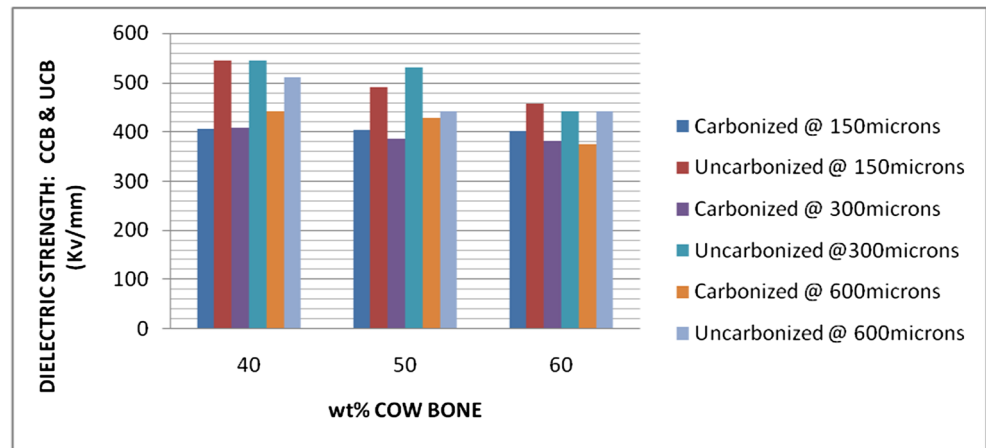


**Fig. 5** **a** DTA/TGA pattern of CCB particles. **b** DTA/TGA pattern of UCB particles

from oxygen and calcium which are the main constituents of bone, there was the presence of carbon in the CCB. This is due to the effect of carbonization.

The EDS of the cow bone particles reveals that the particles contain Ca, Si, O, C, Mg, and P with the presence of C in the carbonized eggshell particles. The

carbon presence is due to the carbonization process (see Fig. 4). These elements confirm that the cow bone particles consists of calcium carbonate in the form of calcite ( $\text{CaCO}_3$ ), the carbonized ES have carbon in graphite form, etc. These analyses are in par with other analyses of reinforcement used by other authors [3, 4].

**Fig. 6** Dielectric strength of CCB and UCB

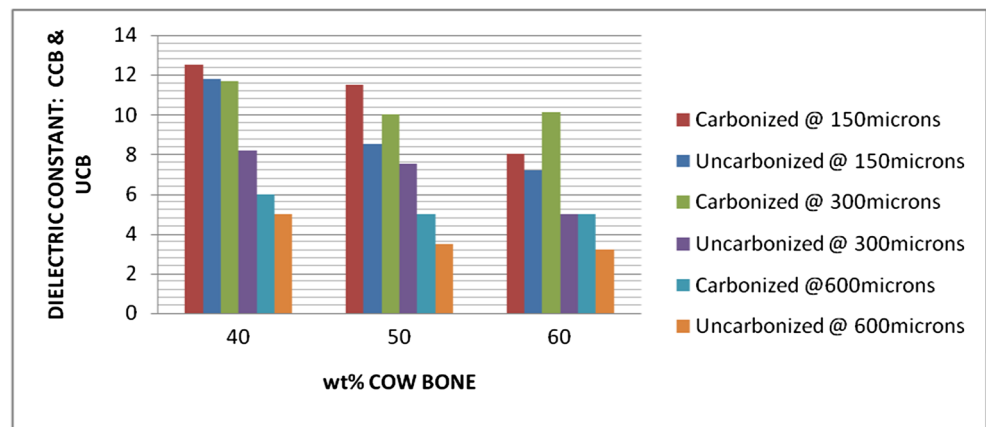
The result of the DTA/TGA scan of the bone particle is shown in Fig. 5. For the TGA curve, the first step of weight loss in the CCB occurred between 30 and 300 °C while for the UCB, the first step of weight loss occurred between 30 and 250 °C. There was an initial weight loss at this stage which was due to the vaporization of water from the bone particles. The degradation of the particle commenced precisely after 300 °C for the CCB and 250 °C for the UCB. There was a gradual decrease in the thermal stability of the CCB particle between 380 and 680 °C. This is the second phase. The reason for this effect is that some of the water of the bone has been burnt off during carbonization process. The thermal stability after this temperature maintained a faster decrease, i.e., from 680 °C until final degradation occurred at precisely 780 °C. This is the third stage. But the UCB particles experienced a faster decrease in the thermal stability of the particle between 270 and 500 °C. The thermal stability after this temperature maintained a gradual decrease, i.e., from 500 °C until final degradation occurred at precisely 890 °C. This is the third stage.

As the CCB sample was heated close to 300 °C, it started to decompose and the decomposed byproducts including silica and carbon were formed. The silica ash gradually delays the degradation process and makes the carbonized cow bone

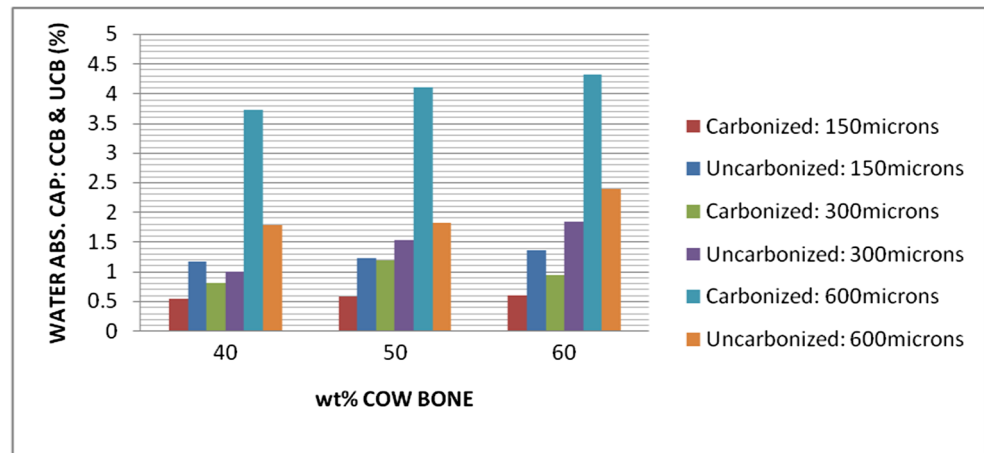
particles more thermally stable. More so, during carbonization, the spongy portion of the bone is being decomposed and densified, resulting in the elimination of volatile matters [12] Thus, the CCB becomes more thermally stable than the UCB is. This shows that carbonized cow bone particles can be used to increase the thermal efficiency of the composite since the temperature of maximum thermal decomposition is around 780 °C which is reasonably high.

### 3.2 Dielectric strength

Figure 6 revealed that smaller particles gave better dielectric strength in each wt% reinforcement. As the particle size increases, there will not be proper bonding between the bone particles and the epoxy. This is because smaller particle sizes have enough resin to bond with than bigger particles do; thus, it will enhance good wettability between the reinforcement and the matrix. It was also observed that 40 wt% gave a better strength of the dielectric than higher 50 wt% did which also gave better strength than 60 wt%. The reason also is that lower wt% will have sufficient resin to bond than higher wt% would, thereby enhancing better wettability between the reinforcement and the matrix. More so, increasing the weight percent of bone particles

**Fig. 7** Dielectric constant of CCB and UCB

**Fig. 8** Water abs. cap of CCB and UCB



will create more pores due to lack of sufficient resin for strong bond formation. Thus, better enhancement of property (strength) was obtained for the composites with lower wt% and smaller particle sizes compared to the higher wt% and bigger particle-sized composites.

The above Fig. 6 depicts that the composite material with optimal strength is the uncarbonized ones. For example, at the 40 wt% of 150  $\mu\text{m}$ , the dielectric strength for the CCB and UCB are 217.96 and 510.84 Kv/mm respectively. The same trend was observed at all the compositions and all the particle sizes. The reason is that activated carbon prepared from high temperature char had a significant amount of micropore volume. Thus, high carbonization temperature causes the formation of micropores in the carbonized material [12]. The high carbonization temperature introduced micropores in the materials which adversely affected the interfacial contact between the particles and the epoxy resin thus low dielectric strength. The lower strength of the CCB therefore was the adverse effect of the high carbonization temperature on the particulates of the samples. Generally, researchers observed that as carbonization temperature increases, both the total micropore volume and the specific surface area also increases [13] thereby affecting the property of the material.

### 3.3 Dielectric constant

The dielectric constant of the CCB and UCB (Fig. 7) was therefore observed to decrease with increasing particle size. This is also in agreement with the work done by [1] wherein

**Table 2** Upper and lower level of each factor along with their coded values

S/No	Variables	Upper level	Lower level
1	Sample condition(A)	Carbonized(+1)	Uncarbonized(-1)
2	Weight (%) (B)	60 (+1)	40 (-1)
3	Particle size ( $\mu\text{m}$ ) (C)	600 (+1)	150(-1)

increase in particle size increases the tendencies of porosity in the material, thus as the particle size is increased, there exist more pores; thus, the experimental results show that the dielectric constant of the samples reduce evidently with increasing porosity in the sample. This is in agreement with the work done by [3]. Once there is large charge storage, it can be readily released on demand. In a rectifier, a capacitor is used to smooth off the pulsating direct current [3]. Free volume is also an important factor in determining the dielectric constant. Free volume is defined as the volume which is not occupied by the polymeric material [4]. The presence of free volume in the form of pores will similarly result in a decrease in dielectric constant as it is being occupied by air whose relative permittivity is about one. A higher fractional free volume means that the density of the material will be lower resulting in a lower polarizable group per unit volume [3].

Figure 7 shows that CCB has better dielectric constant than the UCB does. The carbonization of the bone particles added carbon as impurities. This enabled the material to store more charges thereby increasing the dielectric constant of the CCB. This is in line with the work of [5] who discovered that carbon addition slightly increases the dielectric constant of PZT–PC composite at room temperature.

### 3.4 Water absorption

It can be seen from Fig. 8 that the 600- $\mu\text{m}$  particle size of the composites absorbed more water than did the 300 and 150- $\mu\text{m}$  ones. This is because, the epoxy matrix and the bone reinforcement are not soluble in each another and interfacial bonding between the bone and the matrix phases may not be perfect thereby have given rise to interphase and interparticle spaces otherwise known as porosity [13]. As the wt% of bone increases, the level of porosity also increases hence the amount of water absorption. The uncarbonized cow bone (UCB) composites showed a higher level of porosity by absorbing more amount of water than the carbonized cow bone (CCB)



**Table 3** Standard order of test sequence and result

Experiment number	Sample condition (A)	Weight % (B)	Particle size (C) μm
1	Uncarbonized (-1)	40 (-1)	150 (-1)
2	Carbonized (+1)	40 (-1)	150 (-1)
3	Uncarbonized (-1)	60 (+1)	150 (-1)
4	Carbonized (+1)	60 (+1)	150 (-1)
5	Uncarbonized (-1)	40 (-1)	600 (+1)
6	Carbonized (+1)	40 (-1)	600 (+1)
7	Uncarbonized (-1)	60 (+1)	600 (+1)
8	Carbonized (+1)	60 (+1)	600 (+1)

composites. This is because the spongy portion of the bone is being decomposed and densified during carbonization thereby minimizing the degree of macropores in the bone particles. This result is in agreement with the outcome obtained when polyester was reinforced with iron fillings [1–3].

**3.5 Model for prediction of the dielectric properties of cow bone composites**

A full factorial design of experiments of the type  $P^n$  was used in this study, where  $n$  corresponds to the number of factors and  $P$  represents the number of levels, i.e.,  $n$ , corresponds to the number of factors (sample condition, wt% of reinforcement and particle size) and  $P$  the number of levels ( $P=2$ ) upper and lower levels of each variable (see Table 2). Thus, the number of trial experiments to be conducted for each material is 8 (i.e.,  $2^3=8$ ).

The standard order of sequence is shown in Tables 3 and 4. The sum of squares for main and interaction effects was calculated using Yates algorithm. The significant factors (main and interaction) were identified by analysis of variance (ANOVA) technique.

From the result of the factorial design in Table 4, the sample condition and wt% of cow bone appears to be the most important variable with main effect of -72.12 and -68.12. This implies that carbonizing the cow bone decreases the

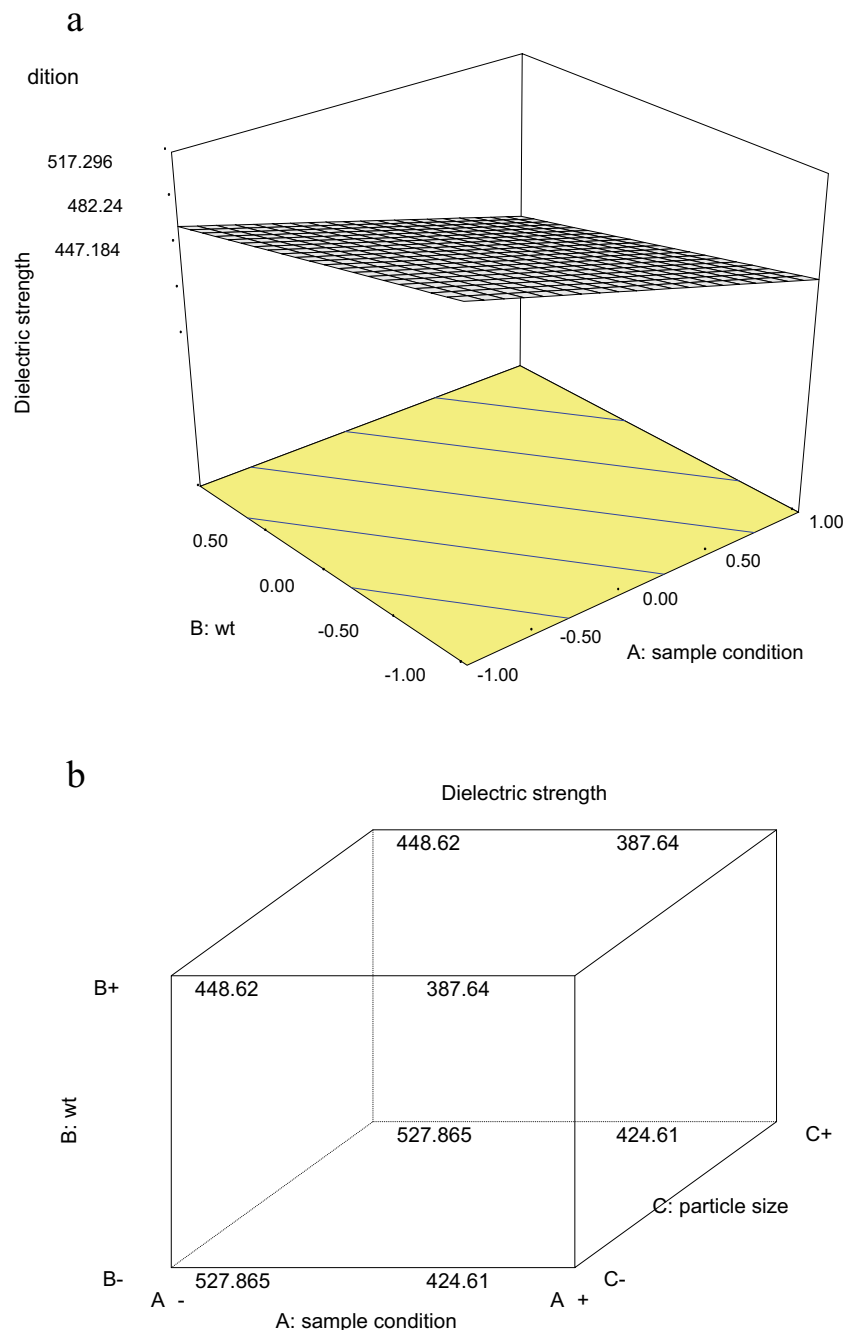
dielectric strength by -71.12 and also increasing the wt% of cow bone decreases the dielectric strength by -68.12. The effect of the particle size is -19.23; thus, increase in particle size from 150 to 600 μm decreased the dielectric strength by -19.23. It can also be seen that the interaction of AB(31.135) and AC(24.92) have positive effects on the dielectric strength of the composites, while the interaction of BC(-20.93) and ABC(-10.21) have negative effects on the dielectric strength of the composites. The Cube graph, half normal plot, and 3D graph show the estimated response surface for dielectric strength as a function of sample condition, wt%, and particle size. It can be seen that the dielectric strength were highly influenced by the sample condition (A) and wt% (B) of cow bone. For the sample condition and wt% of the cow bone particles, the dielectric strength were found to decrease when changing sample condition from uncarbonized to carbonized and wt% from 40 to 60.

Figure 9a shows the plot of two factors A and B and the interaction of the factors AB that significantly influenced the dielectric strength of the cow bone. It is seen from the 3D surface plot that as you navigate on the wt% axis from +1.00 to -1.00, there is increase in the dielectric strength, but as you navigate from -1.00 to +1.00, there is a decrease in the dielectric strength. Thus, increased wt% reduces the dielectric strength. Figure 9b shows the cube plot of the three factors: sample condition (A), wt% (B), and particle size (C).

**Table 4** Expanded plan matrix for factorial design of the dielectric strength of the composites

S/n	$x_0$	A	B	C	AB	AC	BC	ABC	Dielectric strength (Kv/mm)
1	1	-1	-1	-1	+1	+1	+1	-1	544.89
2	1	1	-1	-1	-1	-1	+1	+1	406.5
3	1	-1	1	-1	-1	1	-1	+1	456.35
4	1	+1	+1	-1	+1	-1	-1	-1	400.67
5	1	-1	-1	+1	+1	-1	-1	+1	510.84
6	1	+1	-1	+1	-1	+1	-1	-1	442.72
7	1	-1	+1	+1	-1	-1	+1	-1	400.89
8	1	1	1	1	1	1	1	1	374.61
Effects	442.18	-72.12	-68.12	-19.23	31.135	24.92	-20.93	-10.21	

**Fig. 9** **a** 3D surface plot of the effect of dielectric strength of cow bone. **b** Cube plot of the effect of the dielectric strength



The dielectric strength of the cow is enhanced as you move from the +ve end (i.e., carbonized) to the -ve end (i.e., uncarbonized) of the A axis and as you move from the +ve end (i.e., 60wt %) to the -ve end (i.e., 40wt %) of the B axis. This shows that the uncarbonized particles and the 40 wt% reinforcement significantly improved the dielectric strength of the composite than the carbonized particles and 60 wt% reinforcement. On the +ve end (i.e., 600  $\mu\text{m}$ ) and -ve end (i.e., 150  $\mu\text{m}$ ) of the C axis, there is no change, showing that particle

size is not a significant factor in enhancing the dielectric strength of the cow bone composite.

#### 4 Conclusions

Based on the experimental analysis, the results of the investigations and the discussions, the following conclusions have been made.

1. By incorporating cow bone as reinforcement into epoxy matrix, it can be used in electrical parts.
2. From the result of EDS, it was found that there was high level of carbon in the CCB particles as against the UCB particles and its effect was seen in lower dielectric strength and higher dielectric constant of the composites.
3. The measured properties of the materials are comparable to those of standard insulators. Thus, the developed composite materials can serve as practical and safe dielectrics for low to moderate voltages.
4. The high carbonization temperature introduced micropores in the materials which adversely affected the interfacial contact between the particles and the epoxy resin thus low dielectric strength. The smaller strength of the carbonized samples therefore is the adverse effect of the high carbonization temperature on the particulates of the samples.
5. It has been established that abundant cow bone particles can be used in the production of dielectric materials.

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