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STATISTICAL DESIGN ANALYSIS FOR THE PREDICTION OF THE POLARIZABILITY (DIELECTRIC CONSTANT) OF CASSAVA CORTEX PARTICULATE REINFORCED EPOXY COMPOSITES

A.D. Omah[1], E.C. Omah[2], E.O. Oji[1], M.N. Eke[3], I.C. Ezema[1], P.O. Offor[1].
[1]Department of Metallurgical & Materials Engineering, University of Nigeria, Nsukka, Nigeria.
[2]Department of Food Science and Technology, University of Nigeria, Nsukka, Nigeria.
[3]Department of Mechanical Engineering, University of Nigeria, Nsukka, Nigeria.

ABSTRACT

The possibility of reinforcing epoxy resin with cassava cortex (CCtx) for composite production was studied. The produced composites were experimentally investigated and statistically analysed for the development of a mathematical model to predict the composites' dielectric constant. The CCtx was carbonized at 550°C and subsequently milled to 150, 300 and 600µm. Composites were produced using the CCtx particles varying from 40 to 60wt%. The results show that smaller particles and smaller wt% have the best properties. Better enhancement of property was also obtained from the carbonized composites. The Cube and contour graphs show the estimated response surface for the composite property as a function of sample condition, wt.% and particle size. The developed model can be used for optimization of the process parameters of the dielectric constant of the developed composites. The predicted dielectric values were found to be close to the experimentally observed ones.

KEY WORDS: Composites, Cassava Cortex, Carbonization, Epoxy, Mathematical Model.

1.0 INTRODUCTION

A capacitor is a device for storing electric charge when connected to a voltage source. It consists of two metal plates separated by a dielectric as shown in figure 1a. When the capacitor is connected to a voltage source, one of the plates acquires positive charge, and the other negative charge. The property of a capacitor to store electricity (or electric charge) when its plates are at different potentials is known as its capacitance. It is designated by the letter 'C'. The capacitance of a capacitor is therefore defined as the amount of charge required to create a potential difference of one volt between the plates. [1] A capacitor when connected to a voltage source will store charge proportional to the area A of the capacitor plates. When subjected to a voltage, an electric field E between the plates is created. The charge is stored on the capacitor plates. If a dielectric material is inserted between the capacitor plates the capacitance of the capacitor will increase. [1] The value of the capacitance between the plates is given by the equation:

$$C = \varepsilon \times \frac{A}{t} \quad - \quad -- \quad - \qquad (1)$$

where:

A = the area of the plates; *t* = the separation between the plates and **ε** (Greek letter epsilon) is the absolute permittivity of the dielectric, which is a measure of the electrostatic energy stored within it and therefore dependent on the material. The most obvious advantage to using such a dielectric material is that it prevents the conducting plates on which the charges are stored from coming into direct electrical contact. More significant, however, a high permittivity allows a greater charge to be stored at a given voltage and thus greater capacitance. Charge separation in a parallel-plate capacitor causes an internal electric field. A dielectric reduces the field and increases the capacitance.

Dielectric constant (ε_r) is therefore the ratio of absolute permittivity ($\boldsymbol{\varepsilon}$) of a dielectric to the absolute permittivity ($\boldsymbol{\varepsilon_0}$)

of the free space. It is an important property of dielectric because it determines the capacity of a dielectric to develop charges on its surface due to polarization. Hence a good dielectric should have a high value of dielectric constant.

Thus dielectric constant $\varepsilon_r = \frac{\varepsilon}{\varepsilon_0}$ - (2)

Where $\boldsymbol{\varepsilon_0}$ = absolute permittivity of the free space or vacuum, $\boldsymbol{\varepsilon}$ = permittivity of a dielectric.

The dielectric constant of an insulating material is therefore numerically the ratio of the capacitance of a capacitor containing that material to the capacitance of the same electrode system with vacuum replacing the insulation as the dielectric medium.

It is a common observation by researchers and industries like electronic, electrical, construction and automobile industries, that particulate composites are getting attention over fiber composites due to their ease of production [2]. Apart from the cost advantage of the agro-waste material over other reinforcement fillers in use which helps to lower composite’s production cost, cassava cortex (CCtx) is readily available in Nigeria as a plant waste. Integrating CCtx into epoxy will also help to reduce the composite density and improve the mechanical and dielectric properties of the composites. Natural particle-containing composites are more environmentally friendly, and are used in transportation (automobiles, railway coaches, aerospace), military applications, electrical, electronic industries etc. [3].

Cassava peel waste is increasingly generated annually in Nigeria from the production of cassava tuber for human consumption, starch production and industrial uses [4]. Cassava peel (phelloderm) is one of the three separate segments in a cassava root and the peel contains the cortex. These peels are waste, thus they are usually discarded and with time they constitute environmental nuisance and become hazardous to lives in the environment. More so, vegetation and soil around the cassava peel heaps become unproductive due to ecological and chemical reactions that take place between the fermenting peels, soil and the surrounding vegetation. [5].

Cassava cortex was used to reinforce epoxy resin in this research so as to add to earlier efforts by researchers to recover agro-waste materials for useful products. This work therefore presents the developed statistical design model of this composite based on the carbonization of CCtx, particle size and weight percent of the reinforcing particles. This work will give a lift to the commercial and other benefits of CCtx as an agricultural waste and as well control the environmental pollution caused by it. CCtx being a natural filler is expected to have good interfacial contact with the epoxy resin. To achieve this, the cassava cortex materials were carbonized at 550°C.

Carbonization of CCtx involves converting CCtx as an agricultural residue into elemental carbon and other chemical compounds by heating in the absence or limited amount of air to a temperature high enough to dry off free moisture and other volatile substances in the cassava cortex. [6]

However, there is dearth of information on the effect of carbonization on the dielectric constant of agro-waste materials. This work shows the relevance of carbonization on agro-waste materials with respect to the dielectric properties of the materials.

Considering Figure 1b below, two charged plates are separated, with equal and opposite charges on either side. Assuming that there is a vacuum between the plates, there will exist an Electric Field in the figure directing downward (from the positive charge to the negative charge). But if a dielectric material made up of atoms which often form molecules is placed between the plates; these molecules will have some sort of dipole moment. In the absence of an external electric field, these molecules will align randomly. See figure 1c. If this material is placed between the charged plates of Figure 1d, the molecules will align themselves by their dipole moment and the external electric field as shown in Figure 1d: this is called polarization. This is expressed by a number called the dielectric constant. Although the term "insulator" implies low electrical conduction, "dielectric" is typically used to describe materials with a high polarizability. The latter is expressed by a number called the dielectric constant. Thus, the polarization of the dielectric by the applied electric field increases the capacitor's surface charge which implies increased dielectric constant [7].

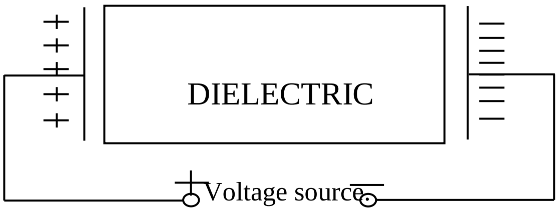


Fig. 1: A capacitor

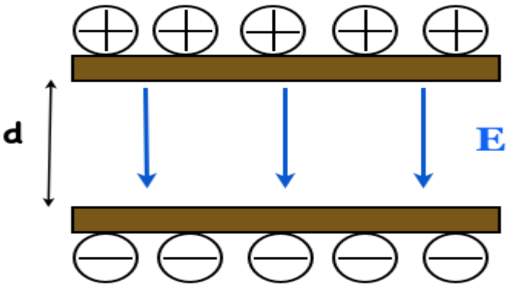


Fig. 1b Two Plates Capacitor with Equal Charge Separated by a Distance d. [8]

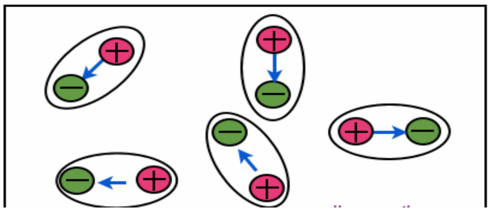


Fig. 1c: A dielectric material showing random orientations of molecules in the absence of an externally applied electric field. [8]

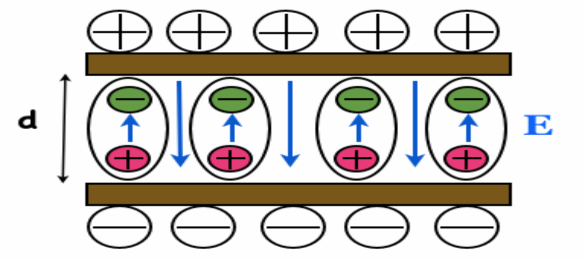


Fig. 1d: A dielectric inside two capacitor plates showing alignment of molecules by their dipole moment and external Electric Field. [8]

2.0 MATERIALS AND METHOD

2.1 Materials

The following materials were used in this work: cassava cortex (carbonized and uncarbonized), DC battery, a digital multimeter, set of sieves (mesh sizes: 150, 300, and 600µm), Epoxy LY556 (matrix), hacksaw, vernier caliper, grinding machine, heat treatment furnace, a pair of tongs and digital weighing balance etc.

2.2 Method

A low cost manufacturing process was developed for this study. The agro-wastes material (CCtx) was sourced from local cassava dealers in Nsukka town of Enugu State. They were washed, sun dried, carbonized at a temperature of 550°C and milled and sieved after carbonization to particle sizes of 150, 300, and 600µm. See figure 3. The composites were produced by varying the CCtx from 40 to 60wt%. The mixture was allowed to cure in the mould and was removed for test analysis.

(a) (b) (c)

Fig. 2. Photographs of the cassava cortex: (a) cassava cortex under sun drying (b) sieved uncarbonized cassava cortex (c) sieved carbonized cassava cortex.

The cast samples are shown in figure 3 below.

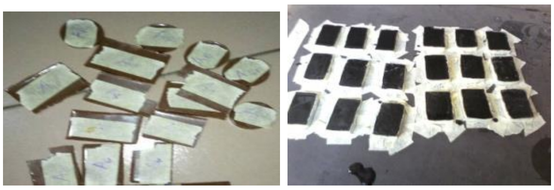


Fig.3: Mould and cast samples for the composite.

Determination of dielectric constant

The test was conducted in accordance with ASTM D 150 (2013). [9] To determine the dielectric constant, the composite samples were moulded into rectangular plates of length 50mm, width 30mm and thickness 2mm. Figure 4 shows the experimental set-up used for the determination of the dielectric constant. It consists 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 (Vo). The composite samples were then used separately to fill the air gap between the capacitor plates that were connected to battery and the voltage (V) across was also measured differently for each composite sample.

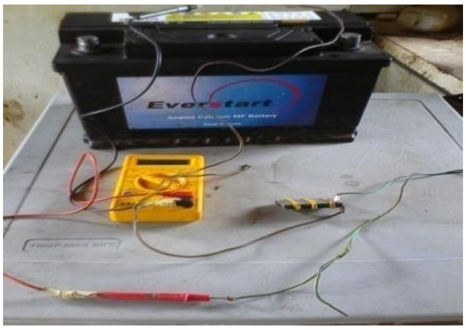


Fig.4: Experimental set up showing the determination of the dielectric constant of the composites.

The relationship between Vo (voltage across the capacitors with an air gap between them) and V (voltage across the capacitors with the composite sample between them) gives the dielectric constant, thus:

$$\varepsilon_r = \frac{V_o}{V} \text{ -------} (3)$$

3.0 STATISTICAL DESIGN ANALYSIS FOR THE DIELECTRIC CONSTANT OF CASSAVA CORTEX COMPOSITES

The process parameters considered in this research are sample condition i.e. carbonization temperature (A), wt% filler loading (B) and particles size (C). The two levels for process parameters with their units and notations are given in Table 1.

Table 1: Low and High level of each factor.

Factors	Low level (−)	High level (+)
Sample Condition (A)	32°C	550°C
Weight % Filler	40wt%	60wt%
Particles Size (C)	150µm	600µm

Full factorial design is a statistical tool to analyse a set of results with minimum number of experiments. [10**]** The methods of designing such experiments are dealt with in literature [11]. Two level full factorial design was used in this study. The eight sets of coded conditions of experiments based on 2^3 full factorial designs are given in Table 2 [11]. 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.

3.1 Development of Mathematical Model

The model for the dielectric constant of the composites was obtained by representing the dielectric constant values by ‘D’ so that the response function can be expressed by the equation (4) below:

$$D = f\,(A, B, C) \quad - \quad (4)$$

Where A = Sample condition (carbonization temperature)

B = weight % filler loading

C = Particle size

The model selected includes the effects of main variables first-order and second-order interactions of all variables. Hence the general model is written as:

$$D = a_o + a_1A + a_2B + a_3C + a_4AB + a_5AC + a_7BC + a_8ABC \quad - - \quad (5)$$

Where a_o is the response variable at the base level and a_1, a_2, a_3, are coefficients associated with each variable A(Sample condition), B(weight percentage) and C(particles size) respectively; and a_4, a_5, a_6, a_7 and a_8 the interaction coefficient between A, B and C within the selected levels of each variable. The methodology for calculating the values for each regression coefficients, using the coded values A, B and C of each variable, is described elsewhere [12] Thus, the number of trial experiments, to be conducted for each material is 8 (i.e. 2^3=8). The eight sets of coded conditions of experiments based on 2^3 full factorial designs are given in Table 2 [11]. The standard order of sequence is shown in Table 2. The sum of squares for main and interaction effects was calculated using Yates algorithm.

4.0 RESULTS AND DISCUSSION

The results of the research are given below.

Table 2: Standard Order of Test Sequence and Actual/Predicted Values Result

Experiment number		weight %(B)	Particle size (C) µm	DIELECTRIC CONSTANT	
				ACTUAL VALUES	**PREDICTED VALUES**
1	Ucarbonized(-1)	10(-1)	150(-1)	10.59	10.70
2	Carbonized(+1)	10(-1)	150(-1)	11.67	11.73
3	Ucarbonized (-1)	60(+1)	150(-1)	7.44	7.62
4	Carbonized (+1)	60(+1)	150(-1)	9.00	8.65
5	Ucarbonized (-1)	10(-1)	600(+1)	8.00	7.40
6	Carbonized (+1)	10(-1)	600(+1)	8.00	8.43
7	Ucarbonized (-1)	60(+1)	600(+1)	4.00	4.32
8	Carbonized (+1)	60(+1)	600(+1)	5.50	5.35

Coded=-1(low level), +1(upper level)

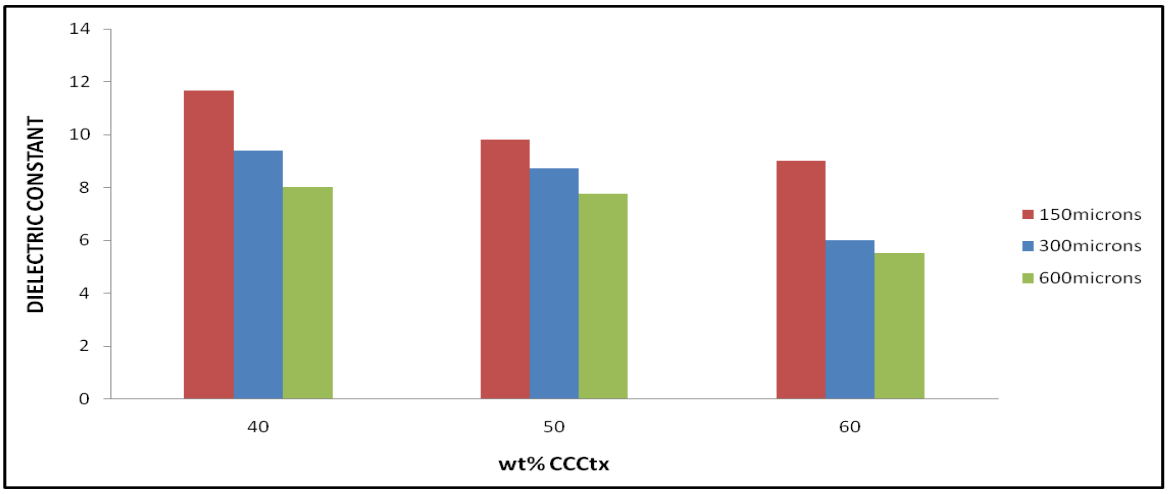


Fig. 5a: Dielectric Constant of CCCtx

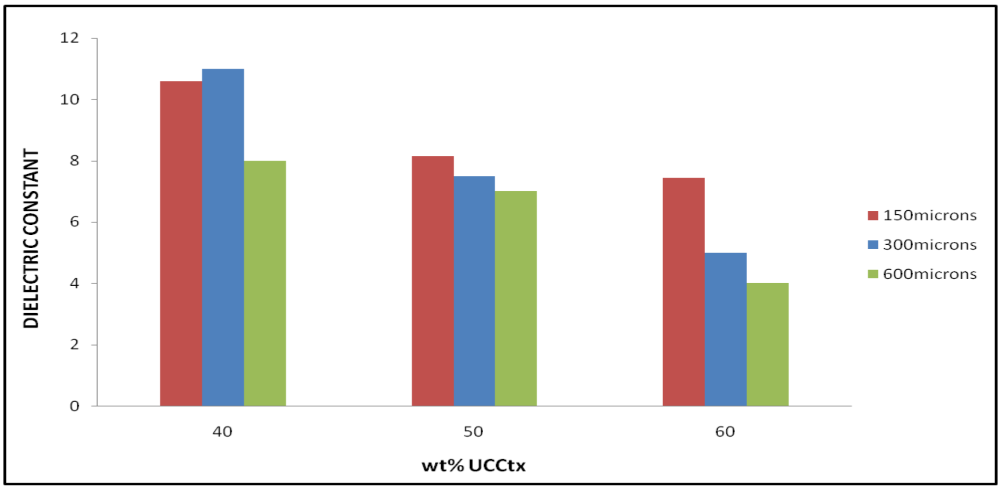


Fig. 5b: Dielectric Constant of UCCtx

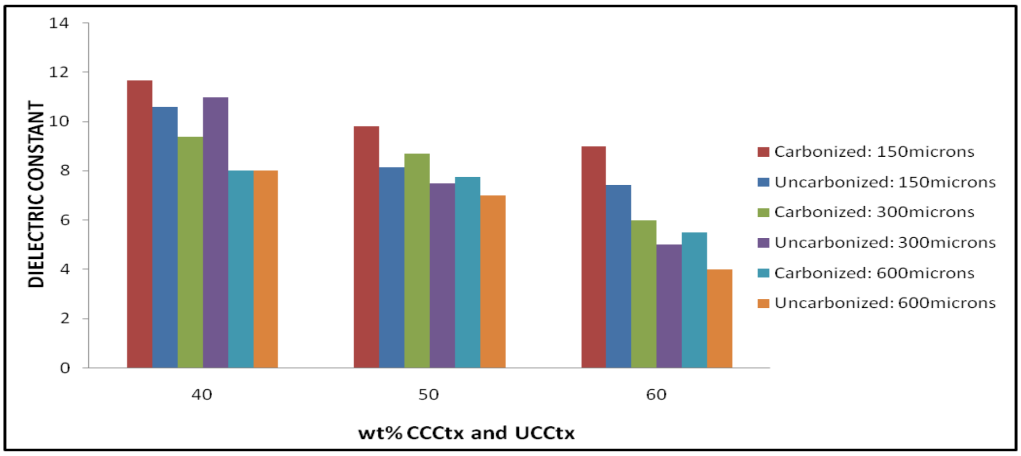


Fig. 5c: Dielectric Strength of CCCtx and UCCtx [2].

MODEL FOR PREDICTION OF THE POLARIZABILITY OF CASSAVA CORTEX COMPOSITES [2].

Table 3: Expanded Plan Matrix for Factorial Design of the Dielectric constant of cassava cortex composites

S/n	**x_0**	**A**	**B**	**C**	**AB**	**AC**	**BC**	**ABC**	**Polarizability (Dielectric Constant)**
1	1	-1	-1	-1	+1	+1	+1	-1	**10.59**
2	1	1	-1	-1	-1	-1	+1	+1	**11.67**
3	1	-1	1	-1	-1	1	-1	+1	**7.44**
4	1	+1	+1	-1	+1	-1	-1	-1	**9.0**
5	1	-1	-1	+1	+1	-1	-1	+1	**8.0**
6	1	+1	-1	+1	-1	+1	-1	-1	**8.0**
7	1	-1	+1	+1	-1	-1	+1	-1	**4.0**
8	1	1	1	1	1	1	1	1	**5.5**
Effects	**8.03**	**1.03**	**-3.08**	**-3.3**	**0.49**	**-0.29**	**-0.17**	**0.25**	

Figures 5a and b show the dielectric constant of CCCtx and that of UCCtx. The dielectric constant of the CCCtx and UCCtx was therefore observed to decrease with increasing particle size. This is also in agreement with the work done by Q.G. Chi et al. [13] Increase in particle size increases the tendencies of porosity in the material. Thus, as the particle size is increased, there exist more pores; the experimental results therefore show that the dielectric constant of the samples reduces evidently with increasing porosity in the sample. This is also in agreement with the work done by Jie XUy et al. [14**]**

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 [15]. The presence of free volume in the form of pores will similarly result in a decrease in dielectric constant as it is 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. [16]

The figure 5c shows that CCCtx has better dielectric constant than the UCCtx. The carbonization process led to an enhancement in the carbon content (as impurities) of the particulate reinforcement thereby increasing the number of dipoles which polarize in the direction of the electric field. This enabled the material to store more charges thus increasing the dielectric constant of the CCCtx. This is in line with the work of N. Jaitanong e-tal [17] who worked on the “Effect of Carbon Addition on the Dielectric Properties of 0 – 3 PZT – Potland Cement Composites,” and discovered that carbon addition slightly increased the Dielectric Constant of PZT – PC Composite at room temperature.

Table 3 shows the result of factorial design of cassava cortex composites. The sample condition, wt% and particle size of cassava cortex are seen to be the most important variables with main effect of (1.03), (-3.08) and (-3.3). This indicates that carbonizing the cassava cortex increased the composite’s polarizability (dielectric constant) by 1.03; increasing the wt% from 40 to 60 decreased it by 3.08 and increasing the particle size of cassava cortex decreased the dielectric constant by 3.3. It can also be observed that the interaction of AB (0.49) and ABC (0.25) has a positive effect on the composites polarizability; while the interaction of AC(-0.29) and BC(-0.17) have negative effect on the composites polarizability; (see the half normal plot, contour graph and cube graph). These three graphs show the estimated response surface for dielectric constant as a function of sample condition, wt% and particle size. It is noticed from these graphs that the dielectric constant was highly influenced more by the wt%(B) and particle size(C) of cassava cortex; this is followed by the sample condition(A) of the cassava cortex. The dielectric constant of the samples was found to slightly increase when changing sample condition from uncarbonized to carbonized, but decreased when reinforcement is changed from 40wt% to 60wt% and particle size from 150microns to 600microns.

The developed model equation for the dielectric constant of the composites can be expressed as: The developed model equation for the dielectric constant of the composites can be expressed as:

$D= +8.03+(0.52*A)-(1.54*B)-(1.65*C)$-(6)

Substituting the coded values of the variables for any experimental condition in Equation 3, the dielectric strength for the composites can be calculated**.**

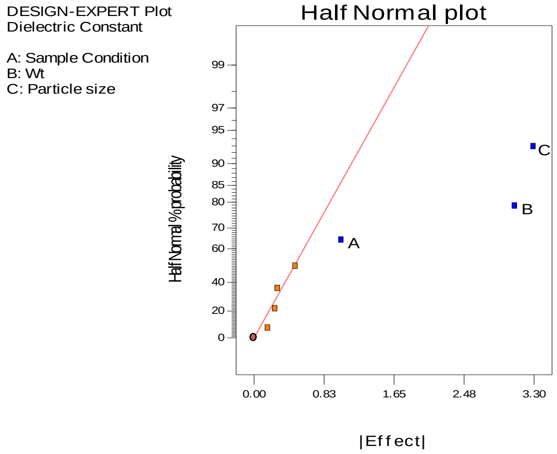


Fig. 6a: Variation of half probability with effects

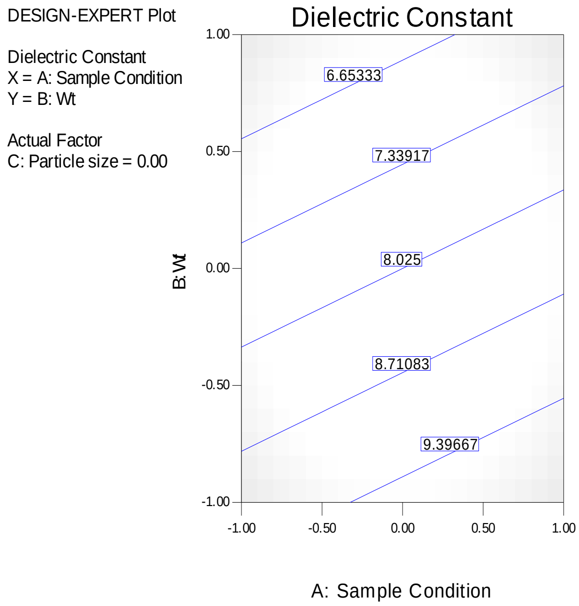


Fig. 6b: Contour surface plot of the effect of parameters (A and B) on the Dielectric Constant of Cassava Cortex – epoxy composite.

Figure 6a: shows the plot of three factors: A, B and C as the significant model terms that significantly influenced the dielectric constant of the c/cortex; while the contour graph shows two factors: sample condition(A) and wt%(B). It is seen from the contour graph that as you navigate on the wt% axis from 0.00 to -1.00 there is increase in the dielectric constant of the c/cortex, but as you navigate from 0.00 to +1.00 there is a decrease in the dielectric constant. Thus, increased wt% reduced the dielectric constant of c/cortex, while decreased wt% enhanced the dielectric constant of the composite. Moving towards +1.00 of **C** – axis, the dielectric constant reduces, while it increases towards -1.00; which implies that smaller particle sizes enhanced the dielectric constant of the c/cortex than the larger sizes. Moving towards +1.00 of A – axis enhances the dielectric constant, while it diminishes towards -1.00; which implies that carbonized samples have more enhanced dielectric constant than the uncarbonized ones.

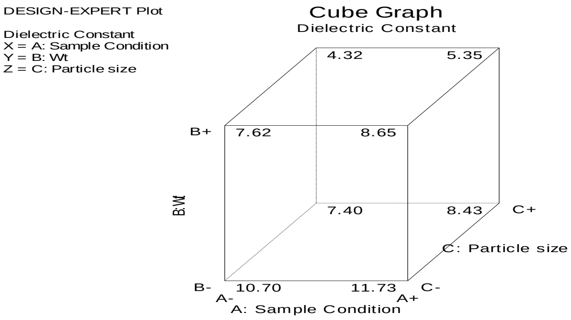
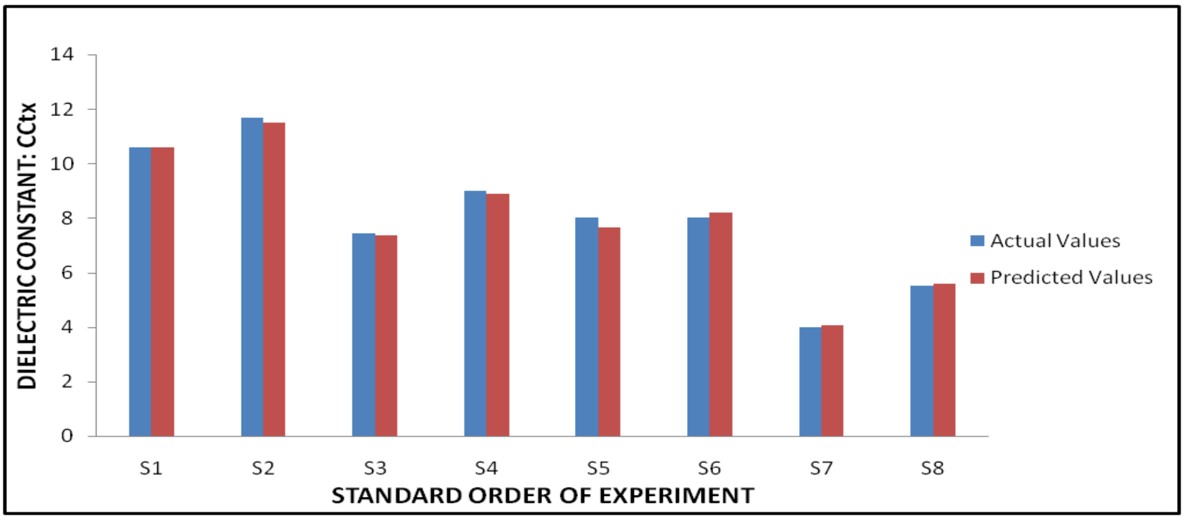


Fig. 6c: Cube plot of the effect of the D/Constant: cassava cortex



(d)

Fig. 6d: Actual & Predicted Values of the D/Constant: cassava cortex

Table 4: ANOVA for Selected Factorial Model of the dielectric constant value

Source	**Sum of Squares**	**DF**	**Mean Square**	**F Value**	**Prob > F**	
Model	42.90	3	14.30	68.06	0.0007	**significant**
A	2.14	1	2.14	10.20	0.0331	**significant**
B	18.97	1	18.97	90.31	0.0007	**significant**
C	21.78	1	21.78	103.67	0.0005	**significant**
Residual	0.84	4	0.21			
Cor Total	43.74	7				

Figure 6c: is the cube plot showing the three factors: Sample Condition (A), wt%(B) and Particle Size(C). The dielectric constant of the c/cortex is enhanced 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 40wt% reinforcement significantly improved the dielectric constant of the composite more than the 60wt% reinforcement. The dielectric constant of the CCtx is also enhanced as you move from the -ve end of C axis to the +ve end of the C axis. Thus, smaller particle size significantly influenced the dielectric constant of the CCtx than lager particle sizes. Moving towards +1.00 of A-axis enhanced the dielectric constant, while it decreased towards -1.00; showing that carbonized samples of the CCTx yielded more enhanced dielectric constants than the uncarbonized ones.

Figure 6d: shows the predicted values along with the actual experimental values in different experimental conditions. It is evident from the figure that the actual experimental values are in close proximity with the predicted values. This can also be seen in Table 2.

ANOVA was used to determine the design parameters significantly influencing the dielectric constant. The ANOVA table revealed that the Model F-value of 68.06 implies the model is significant. There is only a 0.07% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case (sample condition)A, (wt%)B and particle size(C) are the significant model terms (see Table 4) The "Pred R-Squared" of 0.9231 is in reasonable agreement with the "Adj R-Squared" of 0.9664 with a standard devation of 0.46 and mean of 8.03.

Figure 6d: shows the predicted values along with the actual experimental values in different experimental conditions. It is evident from the Figure that the actual experimental values are in close proximity with the predicted values.

Generally, the model results are in good agreement with experimental data. Thus, the predicted results obtained using factorial design model agreed well with the experimental results.

5.0 CONCLUSION

Dielectric tests were carried out on the produced cassava cortex epoxy composites. The following conclusions were made from the experimental analysis, modelling of results and discussions of our investigations:

1. Cassava cortex can be used as reinforcement into epoxy matrix for polymer composites production.

2. Increasing the filler loading of CCtx particles from 40wt% to 60wt% decreased the dielectric constant.
3. The developed composites have better dielectric constant as the particle size additions decreased from 600µmto 150µm.
4. Carbonizing the CCtx increased the dielectric constant of the composites.
5. The main and the interaction effects of model parameters within the range of investigation of CCtx epoxy composite can be studied effectively by factorial design technique.
6. The developed mathematical model can be used to predict the dielectric constant in terms of the model parameters.
7. The results obtained from the statistical analysis are in good agreement with the experimental findings for the model parameters.
8. The developed mathematical model can be employed for optimization of the process parameters of CCtx epoxy composites with respect to mechanical properties values.

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