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ELECTRICAL AND DIELECTRIC PROPERTIES OF DISCS FABRICATED USING PERIWINKLE SHELL NANOPOWDER AND DRY CASSAVA STARCH

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Abstract. Periwinkle shells and cassava effluent are wastes generated in very large quantities and majorly under-utilized. Due to existence of inefficient waste management systems in developing countries, their disposal is by indiscriminate dumping, a practice that adversely affects the environment and human health. Hence, there is an urgent need to examine their potentials for possible engineering applications in order to further proffer a safe way of managing them. In this work, Periwinkle shell nanopowder and cassava starch were prepared from the wastes and then used to fabricate disc-shaped samples. The samples were developed with various lengths/thicknesses and in triplicates, after which their electrical and dielectric properties were evaluated. For lengths/thicknesses of 2.6 mm to 6.5 mm, the mean electrical resistance and electrical resistivity obtained were (9.89 to 23.00) x 10^6 Ω and (8.231 x 10^5) Ωm respectively. Over the temperature range of 20^{o}C to 50^{o}C, the respective results for thermal sensitivity, activation energy, and temperature coefficient of resistance assessment were (6982 ± 158) K, 0.60 eV, and -8.12 %/K. More so, it was observed that quality factor increased with sample's thickness and frequency of applied signal. With increase in frequency from 0.10 kHz to 100.00 kHz, the dielectric constant of the studied sample decayed exponentially from (20.54 ± 0.04) to (1.67 ± 0.01). Aside meeting the market requirements for NTC thermistors, it was found that the sample could serve as a suitable alternative to conventional dielectrics like mica, paper, plastic film, tantalum oxide, and aluminium oxide for production of capacitors.

Keywords: Activation energy, Dielectric constant, Electrical resistivity, Quality factor, Waste materials

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1. Introduction

All over the world, humans are involved in various activities leading to waste generation. Thus, the exponential growth in global population of humans is, no doubt, an important contributor to the accelerated increase in the amount of wastes generated in recent decades. Generally, improper disposal of wastes can adversely affect the environment and human health. Kaza *et al*. [1] reported that by 2050, the world is expected to generate 3.40 billion tons of waste annually, increasing drastically from today's 2.01 billion tons. In developing countries, wastes disposal has been a serious problem due to existence of ineffective waste management systems [2, 3]. This challenge prompted researchers to come up with recycling as a scientifically-safe way of managing waste materials in order to practically solve the problem.

One typical waste that can be recycled is Periwinkle shell. Periwinkles are marine molluscs with thick spiral shells which act as a strong compact house. In Nigeria, about 12 million tons of waste shells generated are disposed of on land and the seashores yearly out of which Periwinkle shells constitute a considerable amount [4]. Research findings revealed that Periwinkle shells have various useful applications. For example, in concrete production, they can be used as a replacement of various forms of aggregates [5 – 7]. Onuoha et al [8] found in their studies that Periwinkle shells are suitable raw materials for the production of recycled polypropylene composites capable of performing satisfactorily. Aside that, the ash derived from Periwinkle shells (PSA) is a suitable partial substitute for cement for improvement of concrete strength [9, 10]. Also, Periwinkle shell ash can be utilized in stabilization of lateritic soil for road construction [11].

Assorted recyclable wastes are generated from cassava processing industry as well. In this case, the wastewater (otherwise known as cassava effluent) is the most prominent waste material. As of 2019, the Food and Agricultural Organization (FAO) of the United Nations estimated world cassava production to be 278 million tons out of which Africa produced about 170 million tons. According to Achi et al [12], 3 m^3 to 6 m^3 of cassava effluent could be generated during processing of 6 to 8 tons of cassava tubers supplied daily. Apart from using the effluent for production of biogas [13, 14], hydrogen [15], and methane [16, 17], it can be utilized to prepare fertilizer [18]. Moreover, cassava effluent has been found to be a source of starch suitable for preparation of binding agent that can be applied as a coating material [19, 20] and in the development of composite insulation panels [21 – 23].

However, it has been observed that Periwinkle shells and cassava effluent are generated in very large quantities and as such, they still remain under-utilized with attendant disposal problems. For instance, Periwinkle shells are often heaped in open fields and landfills, a practice which causes environmental problems such as pollution and breeding of disease-carrying organisms [24]. Also, in most regions, the cost associated with the handling and disposal of cassava effluent constitutes a large financial burden to the processors, thereby prompting them to indiscriminately dispose the waste into environment. Since these wastes have been identified to be toxic to the environment [25 – 27], there is need to proffer a way to further manage them for sustainable development and beneficial purposes. Thus, this study has been designed to establish additional potentials of the aforementioned wastes for industrial utilization. Specifically, electrical and dielectric properties will be investigated for discs developed using Periwinkle shells nanopowder with starch derived from cassava effluent. It is hoped that findings from the work will be of immense benefit to researchers, materials scientists, engineers, and electrical/electronic industries among others.

2. Experimentation

2.1. Materials and collection

Periwinkle shells (gathered from dumpsites) and cassava effluent (obtained from local cassava processing units) were majorly used in this work. These waste materials were sourced and in large quantities within Mkpat Enin Local Government Area of Akwa Ibom State, Nigeria.

2.2. Preparation and Analysis of the starting materials

Periwinkle shell nanopowder (PSN) and cassava starch (CST) were the starting materials utilized in this work. They were prepared from the as-collected wastes as described below:

The Periwinkle shells were washed thoroughly using clean tap water. This was necessary to ensure that any accompanying impurities were completely removed from them. After that, they were dried in an electric oven (Model N30C, Genlab) until they became moisture-free. The dried shells were crushed by means of Agate mortar and pestle and the quantity of it that passed 250 μm – sieve openings was calcined at 1000^{o}C for 5 hours using Muffle ASCO furnace. When the calcined material cooled to about 35^{o}C, it was pulverized by means of a high-energy ball miller (E_{max}, manufactured by RETSCH, GmbH) operated at 500 rpm for 6 hours. This ball milling machine is capable of reducing the particle size of a material feed from about 5 mm to as fine as less than 80 nm.

Also, as per the method adopted by Robert et al [22], the cassava effluent was left to settle in a plastic bucket for 10 hours after which it was decanted to obtain the residue. The residue was sun-dried until it was in powdery form (starch) and continuously weighed to a constant weight. In accordance with the standard procedure [28], the Carr's index of the PSN and that of the CST were determined.

2.3. Fabrication of the Discs (Test samples)

Prior to development of the samples, a wetting liquid was prepared by mixing 10% solution of the starch with hot water and stirring the mixture until a mildly viscous liquid was obtained. While stirring continued, the mixture was heated by means of an electric hotplate (Model E4102W). The resulting liquid (having viscosity of 9.2×10^{-4} Pas) was applied as the wetting liquid to fabricate the samples. In doing so, the PSN was carefully blended into the liquid in the weight ratio of 6:1 of the nanopowder to the solution. The resulting homogeneous mixture was uniaxially pressed at 700 MPa to a compact of diameter (16.50 ± 0.01) mm as measured by means of digital vernier callipers. The pressed compact was sintered at 1000^{o}C for 1 hour before it was diced into disc-shaped chips of various lengths/thicknesses in triplicates, all of which were tested.

2.4. Properties determination

At a time, a sample was engaged in a holder equipped with a pair of nickel electrodes (each measuring 10 mm x 8 mm x 1 mm), two lead terminals, and a unit for assemblage of a disc-shaped

component. The lead terminals of the holder were then connected to an LCR meter (Model 9183, Lutron) for measurements of electrical resistance, capacitance, and quality factor of each sample. This instrument measures the dielectric parameters at 0.10, 0.12, 1.00, 10.00, and 100.00 kHz. For appraisal of electrical resistance variation with temperature, only samples having length of 4.2 mm were used. In that case, a set-up (consisting of a digital thermometer calibrated and equipped with type –K probe, aluminium block with a central circular cavity, clamp, and electric hotplate) was employed additionally as illustrated in figure 1. Care was taken to ensure that the tip of the thermometer probe made a firm contact with the sample under test. The control dial on the hotplate was set to a reasonable level to allow for steady heating of the sample and the electrical resistance displayed by the LCR meter was noted after each rise in temperature at regular intervals of 5^{o}C from 20^{o}C. In each case, the mean value of the measured parameter was determined with the corresponding standard error.

For determination of electrical resistivity, a graph of mean electrical resistance values against lengths of the sample was plotted and the slope obtained was applied based on the relation [29].

$$R = \rho L/A \tag{1}$$

where ρ = electrical resistivity, R = mean electrical resistance, L = length of the sample, and A = cross-sectional area of the sample.

Also, thermal sensitivity constant, β was deduced graphically from the plot of in R against $1/T$ as defined by the relation [30].

$$R = R_o \exp(\beta/T) \tag{2}$$

where β = thermal sensitivity constant, R_0 = electrical resistance at infinity, and T = temperature (in Kelvin).

The thermal sensitivity constant obtained was then used to compute activation energy, E_a of the sample using the formula [29, 30].

$$\beta = E_{a/k} \tag{3}$$

where k = Boltzmann's constant.

Similarly, temperature coefficient of resistance, α was calculated using the equation [30].

$$\alpha = \pm(\beta/T^2) \cdot 100\% \tag{4}$$

(in which case, the negative sign is considered only if R and T vary inversely)

More so, from the plots of mean capacitance values against the inverse of sample's thicknesses at various frequencies, the dielectric constant values were deduced in accordance with the relation [29, 31, 32].

$$C = \varepsilon_0(\varepsilon_r \cdot A/d) \tag{5}$$

where ε_r = dielectric constant, ε_0 = 8.85 x $10^{(-12)}$ $Fm^{(-1)}$, and d = thickness of the sample.

All measurements were carried out at (24.0 ± 1.0)^{o}C.

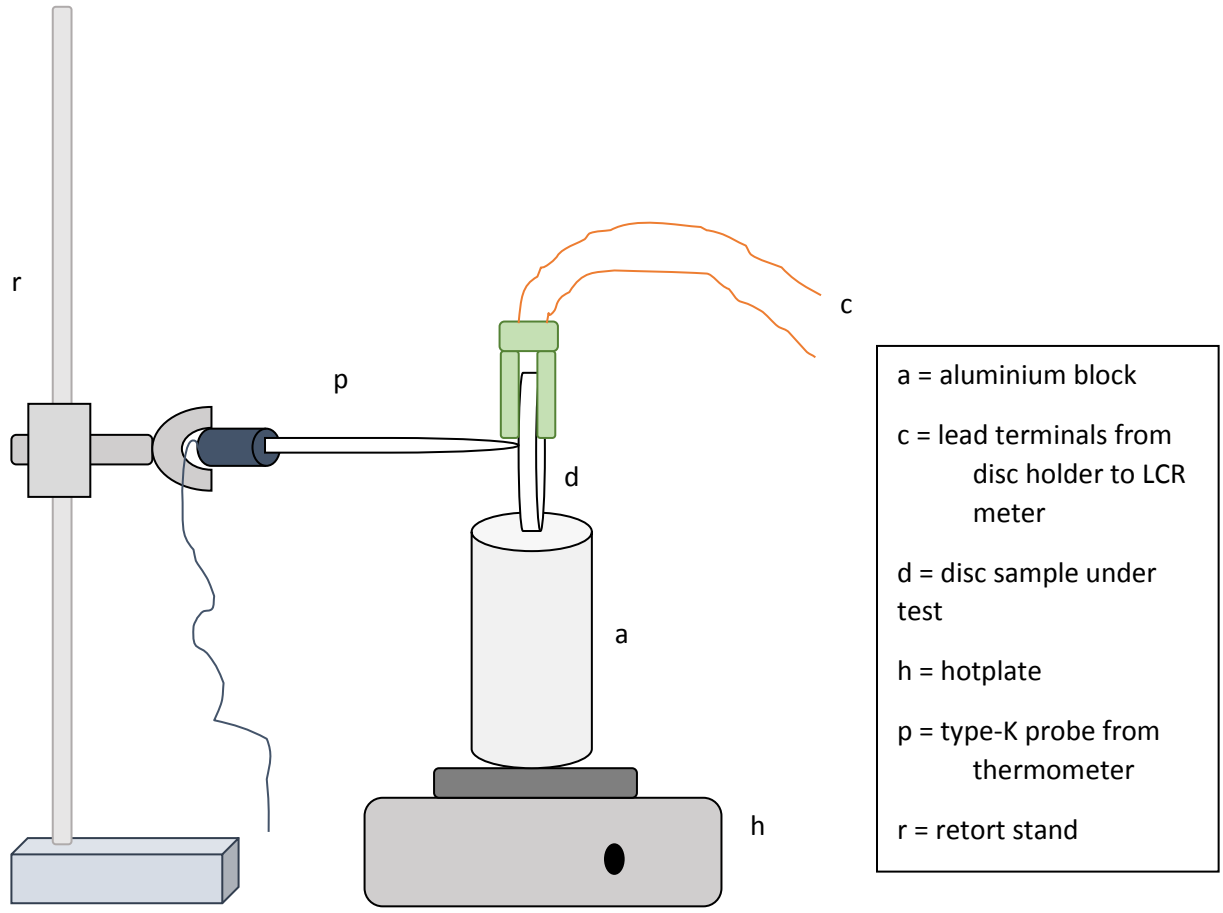


Fig. 1: Schematic diagram of the set-up for resistance – temperature measurements

3. Results and Discussion

Table 1 shows the Carr's index, each of the PSN and CST used as starting materials in this study. The values obtained for the materials are less than 15%, thus signifying high flowability [33, 34]. This implies that the PSN and CST are capable of ensuring good content uniformity in the fabricated samples.

Table 1. Particulars of the starting materials

Starting material			Carr's index (%)
Name	**Code**	**Form**	
Periwinkle shell nanopowder	PSN	Dry/powdery	10.78 ± 0.01
Cassava starch	CST	Dry	11.27 ± 0.01

N/B: The Carr's index value is for three determinations

The electrical resistance values obtained per length and also temperature of the samples are recorded in Table 2. It can be observed that the resistance increases as the length of the sample increases but decreases with increase in temperature. Over the range of temperature under consideration (20^{o}C to 50^{o}C), about 90.0% decrement in the resistance is revealed. Observably, while the resistance is in a positive linear relationship with the sample's length (figure 2), it exponentially decays with temperature (figure 3), thus indicating that the electrical resistivity is the same and the sample has negative temperature coefficient of resistance.

Table 2. Results of the Electrical resistance measurements

Sample's length, L (10^{-3} m)	Electrical resistance, R (10^6 Ω)	Temperature, T (^{o}C)	Electrical resistance, R (10^6 Ω)
2.6	9.89 ± 0.01	20.0	25.01 ± 0.02
3.0	10.96 ± 0.02	25.0	16.02 ± 0.03
4.0	15.40 ± 0.02	30.0	11.40 ± 0.02
4.4	17.73 ± 0.02	35.0	7.82 ± 0.01
5.2	19.87± 0.01	40.0	5.51± 0.02
6.0	23.00 ± 0.02	45.0	4.01 ± 0.02
6.5	24.98 ± 0.08	50.0	2.50 ± 0.01

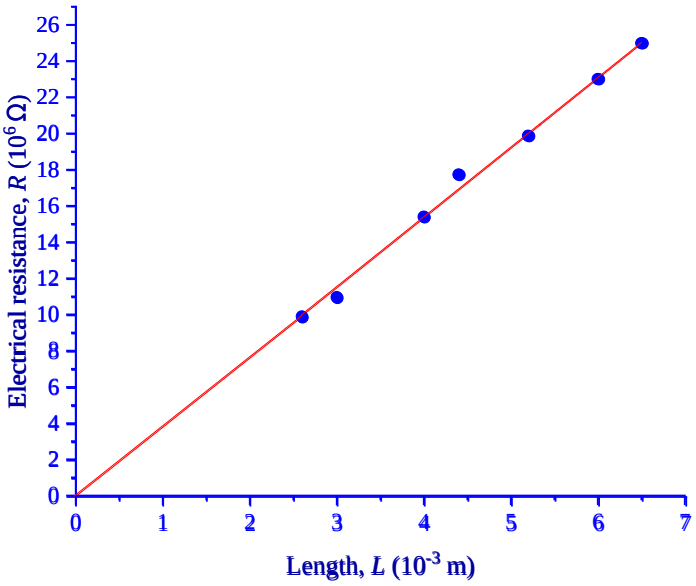


Fig. 2: Variation of Electrical resistance with sample's length

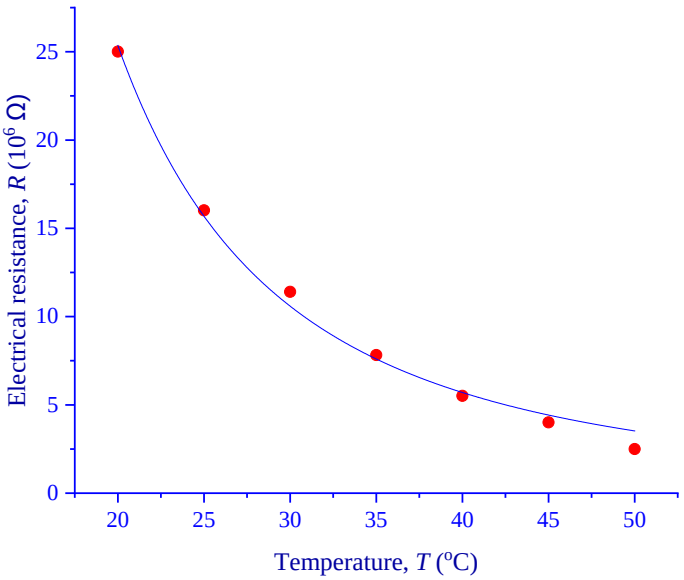


Fig.3: Variation of Electrical resistance with sample's temperature

Properties values deduced from the electrical resistance measurements are presented in Table 3. Resistivity is an intrinsic property of a material. The mean value of electrical resistivity obtained for the sample (8.231 x 10^5 Ωm) is lower than the electrical resistivity at 20°C reported by Sedha [29] for semiconductors and insulators like paper (10^{10} Ωm), mica (5 x 10^{11} Ωm), and glass (10^{12} Ωm). This may be attributed to the fact that Periwinkle shell contains about 93.9% of $CaCO_3$ [35], in which case calcium is a good conductor of electricity. However, it portrays the sample to be insulating.

As for the thermal sensitivity constant, the value obtained for the sample, that is (6983 ± 158) K, shows that it could vary from 6825 K to 7141 K over the considered temperature range. This parameter relates directly with activation energy. Interestingly, the activation energy of the sample (0.60 eV) is 30.4% greater than the maximum value (0.46 eV) Robert et al [30] reported for disc-shaped compacts fabricated using calcined eggshell nanopowder (CEN) for engineering applications. This means that the energy required for formation, movement, and hopping of charge carriers in the studied sample is greater compared to the CEN – based disc.

For negative temperature coefficient (NTC) thermistors, thermal sensitivity constant and activation energy are required to be such that $\beta \geq 2000$ K and $0.1 \text{ eV} \leq E_a \leq 1.5$ eV respectively with $R \geq 10\Omega$ [36]. On the strength of such considerations, it is obvious that the studied sample meets the specified market requirements and thus, could be adjudged to have the potentials of comparing very well with an NTC thermistor when correctly applied to perform certain tasks.

Table 3. Data deduced from the Electrical resistance measurements

Property of the sample	Symbol	Value obtained	Unit
Electrical resistivity	ρ	$(8.231 \pm 0.065)\ 10^5$	Ωm
Thermal sensitivity constant	β	6982 ± 158	K
Activation energy	E_a	0.60	eV
Temperature coefficient of resistance	α	-8.12	%/K

Furthermore, the results recorded in Table 4 show that the capacitance of the sample decreases with increase in thickness. This indicates that if the sample with smaller thickness is used as a dielectric in capacitor, a greater field force will be enhanced and that will bring about a greater field flux for a given voltage applied across the plates compared to the use of the sample with a larger thickness. It can also be observed that irrespective of the sample's thickness, the capacitance decreases with increase in frequency of the applied signal. For thicknesses of 3.0 mm and 4.0 mm, about the same capacitance values are obtained at 1.00 kHz and 0.12 kHz respectively. A similar possibility is observed for thicknesses of 4.0 mm and 5.2 mm at those frequencies respectively.

Stating simply, quality factor is the reciprocal of dissipation factor. In this context, dissipation factor is an indicator of how susceptible a dielectric material is for absorption of some of the energies arising from application of an alternating current signal. From the results presented, the trend in capacitance shows that for a particular sample thickness, more charges could be stored at lower frequencies in the event that the sample is placed in an electric field. In terms of quality factor, contrariety is noticed with respect to thickness variations. As frequency increases from 0.10 kHz to 100.00 kHz, the quality factor differs positively by about 1.557, 1.764, 2.359, 2.626, 3.176, 3.691, and 3.830 for thicknesses of 2.6, 3.0, 4.0, 4.4, 5.2, 6.0, and 6.5 (all in mm) respectively. The quality factor also increases as thickness increases. It therefore implies that the tendency of the sample to absorb energy (when an alternating current signal is applied) decreases as either the frequency of the signal or thickness of the sample increases. Pronounced increment is possible at higher frequencies as revealed in figure 4.

Decrease in dielectric constant of the sample with increasing frequencies shows that more field flux could be allowed at low frequencies. This, in turn, implies that a greater collected charge is possible at lower frequencies for a given amount of field force (applied voltage). As a matter of fact, dielectric constant simply indicates the ability of an insulating material to concentrate electric flux. Its numerical value is specified as the ratio of flux in the insulator compared with the flux in air or vacuum. As such, it can be posited that at 0.10, 0.12, 1.00, 10.00, and 100.00 kHz, the sample is capable of providing a density of electric flux that is approximately 21, 8, 6, 3, and 2 times as great as that of air for the same applied voltage and equal size.

By considering the dielectric constants of some conventional solid materials like ceramics (80 – 100), mica (3 – 8), paper (2 – 6), plastic film (2 – 3), tantalum oxide (25), and aluminium oxide (7), as reported in the literature [37], it can be seen that the sample's dielectric constant values compare well depending on the frequency. Though smaller, the dielectric constant value obtained for the sample at 0.10 kHz is close to that of tantalum oxide but far greater if compared with the value for the aluminium oxide. Being that the two oxides are suitable dielectrics for production of electrolytic capacitors, the sample could be regarded as an alternative dielectric to them. In other cases, the dielectric constant values of the sample at 1.00 kHz and 10.00 kHz lie within the range reported for mica. For similar reason, there is no doubt that the sample could function like either paper or plastic film at 10.00 kHz or 100.00 kHz respectively. Apart from cases involving the conventional dielectrics, the dielectric constants obtained at 10.00 kHz and 100.00 kHz for the sample compare favorably with the values of 3.537 and 1.657 which Etuk et al [31] reported for membranes of hen eggshells and quail eggshells respectively at 10.0 kHz. Figure 5 illustrates that the dielectric constant of the studied sample decays as the frequency increases. This phenomenal trend signifies that re-orientation of dipole gradually becomes impossible at higher frequencies, a situation which also reduces the degree of polarization.

Table 4. Capacitance, Quality factor, and Dielectric constant

Thickness, d $(10^{-3}$ m)	$1/d$ $(10^3 m^{-1})$	Mean with standard error values of capacitance and quality factor per frequency, f									
		f = 0.10 kHz		f = 0.12 kHz		f = 1.00 kHz		f = 10.00 kHz		f = 100.00 kHz	
		C $(10^{-12}$F)	Q	C $(10^{-12}$F)	Q	C $(10^{-12}$F)	Q	C $(10^{-12}$F)	Q	C $(10^{-12}$F)	Q
2.6	0.385	14.93 ± 0.01	0.138 ± 0.001	6.08 ± 0.01	0.315 ± 0.001	4.56 ± 0.02	0.327 ± 0.001	2.56 ± 0.01	0.934 ± 0.001	1.22 ± 0.02	1.695 ± 0.002
3.0	0.333	15.97 ± 0.01	0.158 ± 0.001	5.26 ± 0.01	0.361 ± 0.002	3.99 ± 0.01	0.376 ± 0.002	2.20 ± 0.02	1.085 ± 0.001	1.05 ± 0.01	1.922 ± 0.002
4.0	0.250	9.73 ± 0.01	0.207 ± 0.002	3.96 ± 0.02	0.481 ± 0.001	3.00 ± 0.02	0.501 ± 0.001	1.67 ± 0.02	1.446 ± 0.002	0.80 ± 0.02	2.566 ± 0.001
4.4	0.227	8.86 ± 0.02	0.231 ± 0.001	3.58 ± 0.02	0.529 ± 0.002	2.69 ± 0.02	0.550 ± 0.002	1.51 ± 0.01	1.587 ± 0.002	0.73 ± 0.02	2.857 ± 0.001
5.2	0.192	7.50 ± 0.02	0.270 ± 0.002	3.04 ± 0.01	0.629 ± 0.001	2.31 ± 0.01	0.653 ± 0.001	1.27 ± 0.01	1.889 ± 0.001	0.63 ± 0.01	3.446 ± 0.001
6.0	0.167	6.47 ± 0.01	0.312 ± 0.001	2.62 ± 0.01	0.723 ± 0.001	1.96 ± 0.01	0.751 ± 0.002	1.06 ± 0.01	2.174 ± 0.001	0.53 ± 0.02	4.003 ± 0.001
6.5	0.154	5.99 ± 0.01	0.339 ± 0.001	2.43 ± 0.01	0.790 ± 0.002	1.80 ± 0.02	0.820 ± 0.002	1.01 ± 0.01	2.324 ± 0.001	0.46 ± 0.01	4.169 ± 0.001
Dielectric constant, ε_r		20.54 ± 0.04		8.34 ± 0.12		6.23 ± 0.02		3.48 ± 0.02		1.67 ± 0.01	

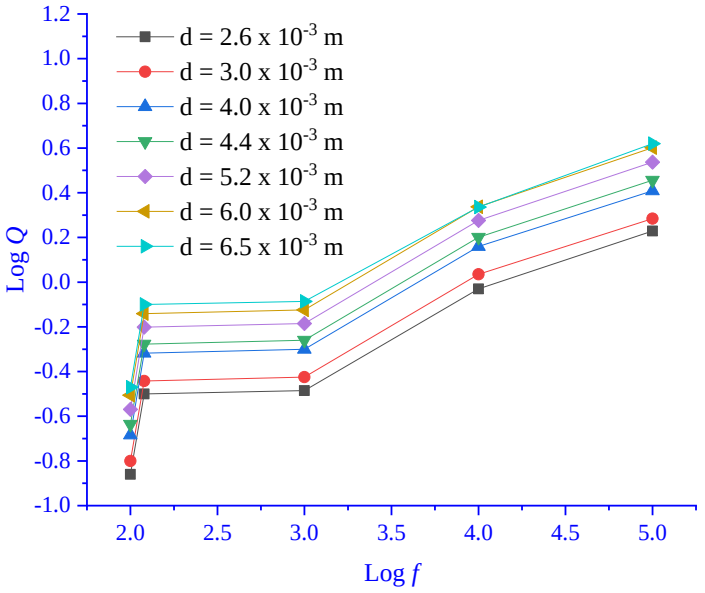


Fig.4: Log plot of Sample's Quality factor against frequency

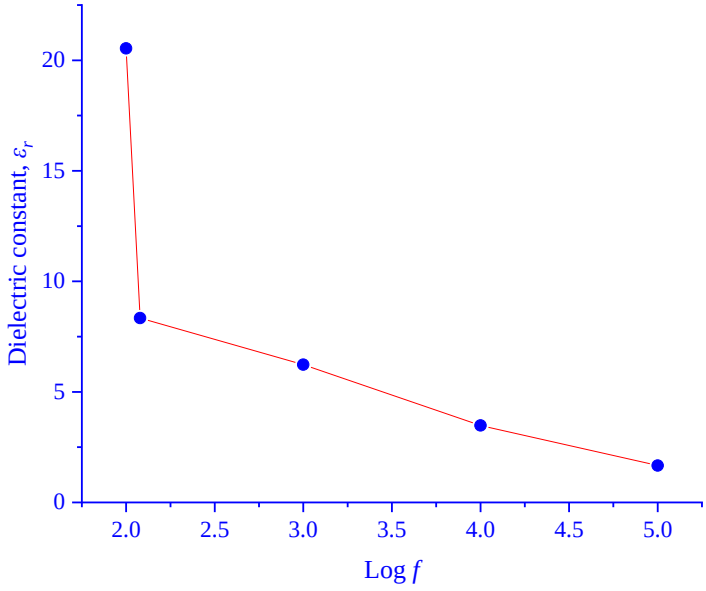


Fig.5: Sample's Dielectric constant versus log of frequency

4. Conclusion

The experimental investigations conducted in this study revealed that disc samples fabricated using Periwinkle shell nanopowder and cassava starch could exhibit high electrical resistance and resistivity with negative temperature coefficient of resistance. Values obtained for electrical resistance, thermal sensitivity constant, and activation energy of the developed samples were found to meet the market requirements for NTC thermistors. At the frequency of 0.10, 0.12, 1.00, 10.00,

and 100.00 kHz, it was noticed that the dielectric constant of the sample was 20.54, 8.34, 6.23, 3.84, and 1.67 respectively. Comparatively, it was observed that the sample could serve as a suitable alternative to conventional dielectrics like tantalum oxide, aluminium oxide, mica, paper, and plastic film which are well-known materials for capacitor making. Hence, Periwinkle shells and cassava effluent could be considered to be promising raw materials for utilization in electrical/electronic industry for production of capacitors or thermistors. This, if implemented, would further help to solve the disposal problems associated with the wastes.

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