

Study of Dielectric Relaxation of Insulating Oil from *Lagenaria siceraria* Seeds

Michael Koumbou Piembe¹, Jean Ndoumbe^{2*}, Charles Hubert Kom¹

¹Laboratory of Electronics, Electrotechnics, Automation and Telecommunications, National Higher Polytechnic School of Douala, University of Douala, Douala, Cameroon

²Laboratory of Computer Engineering, Data Science and Artificial Intelligence, National Higher Polytechnic School of Douala, University of Douala, Douala, Cameroon

Email: *jndoumbe02@gmail.com

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Abstract

The present work concerns the study of the dielectric relaxation of dielectric oil based on *Lagenaria siceraria* (calabash) seeds. Dielectric spectroscopy was used to measure the loss angle, the dielectric constant and the electrical modulus. Three relaxation processes in calabash oil were identified. It was also found that the relative permittivity decreases with increasing temperature and frequency. A study of the imaginary part of the electrical modulus was done and revealed a relaxation process at low frequencies. At higher frequencies, the dielectric relaxation is thermally activated. The increase in temperature leads to a decrease in the relaxation rate. The result obtained indicates that relaxation type is not of the Debye type in the high-frequency region. The Cole-Cole model of the imaginary part of the permittivity as a function of its real part in calabash oil for different temperatures was drawn and analyzed. It shows the existence of a negative temperature coefficient of resistance in the fluid and helps identifying a relaxation process in the conductivity of the sample studied. It highlights the presence of Debye relaxation which characterizes the presence of an abnormal dispersion of the dielectric constant over a frequency range. Calabash seed oil exhibits better dielectric constant (relative permittivity) compared to other oils.

Keywords

Dielectric Relaxation, Electric Modulus, Calabash Seed Oil, Dielectric Spectroscopy, Cole-Cole Model, Debye Model

1. Introduction

The power industry in general, and the production of electrical energy in

particular, is at the heart of a nation's economic development, because without electricity there is no possibility of any kind of growth for a country. One of the essential elements in the electrical energy production chain is the power transformer [1], also known as the high-voltage transformer. As demand for transformers increases with population growth, new technologies are working to improve their design, production, processing, maintenance and operation [2]-[4]. Ideally, transformers should never fail before their service life, as they represent almost 60% of the capital investment in a transformer station [5]. To achieve this, they need to be kept under close surveillance. According to [6] [7], the insulation system of a power transformer essentially determines its lifespan; indeed, it is the fundamental component of this system and must therefore operate as efficiently as possible [8] [9]. Controlling the transformer to ensure that it operates without failure throughout its lifetime (as long as possible) will therefore involve monitoring its insulation system [10]. Operating stresses (electrical, thermal, mechanical and environmental) reduce the lifetime of the transformer, which increases the likelihood of failure.

These transformers are made up of active parts at different potentials, which must be insulated from each other and cooled. This dual requirement (insulation and cooling) has been met since 1891 by the use of the first paper and cardboard impregnated with mineral oil [11]. The insulating medium most commonly used in transformers is, therefore, mineral oil, because of its better physico-chemical and dielectric properties and its low cost. Their disadvantages are their biodegradability and their environmental footprint, which is currently very high [12] [13]. Because of these drawbacks, researchers around the world are currently working to find a more environmentally friendly alternative to mineral oils, with natural or synthetic esters as suitable replacements [14].

This research is being carried out by studying the properties of these vegetable oils in order to make a comparison with those of mineral oils. The properties most commonly characterized by researchers are physico-chemical properties such as viscosity, water content, flash point, pour point, etc. However, the factors that control the viscosity of vegetable oils are not known. However, the most reliable quality control factors are the dielectric permittivity values and the relaxation phenomena of vegetable oils, which are indicative of the oil's fatty acid composition. This is why studies are being carried out to determine these parameters. The authors of [15] present a study of the response to dielectric relaxation of electrically insulating liquids under different types of thermal stress. They show that a slower increase in temperature leads to an increase in polarization losses and a reduction in the distribution of relaxation times. They also found that the logarithm of the relaxation frequency decreases linearly with temperature according to Arrhenius law and that mineral oil has the highest activation energy value. The publication [16] works on the qualitative characterization of nine pure vegetable oil samples (peanut, soybean, linseed, castor, safflower, sunflower, walnut and sesame) using dielectric spectroscopy, which is a very useful technique. They measured the

relaxation time of each of these oils and found that the vegetable oil samples with the highest relaxation times in pico seconds (ps) were soybean oil (398.5), peanut oil (412, 5), linseed oil (318.4) and castor oil (305.3) and the oil samples with lower relaxation times were safflower oil (37.91), sunflower oil (30.6), walnut oil (22.4) and sesame oil (38.4). Frequency-domain dielectric spectroscopy is a useful method for general diagnostic insulation testing. It has many advantages over standard 50 and 60 Hz dielectric power loss tests. One of the main advantages is the testing of the material over a wide range of frequencies, which allows the properties of the insulation system to be selectively distinguished [17] [18]. Dielectric relaxation spectroscopy (DRS) is also a powerful tool for analyzing the magnitude and time dependence of dielectric polarization processes [19]. They exploit the Cole-Cole diagram and show that it highlights the tendency for dipoles to realign as a function of an applied field. Complex permittivity spectra indicate the decreasing nature of the molecular alignment, including a slow decline towards coincident mean values as a function of the molecular binding pattern of the vegetable oil samples. The authors of [20] compared mineral oil and hydrocarbon oil (liquefied gas) in terms of conductivity and relaxation mechanisms in the complex plane of the Cole-Cole diagram and dielectric losses using frequency-domain dielectric relaxation spectroscopy at different time-varying electric field strengths. They demonstrate that with increasing time-varying electric field strength, a better approximation of the Debye behavior is observed in all the polarization processes captured from the oils studied. By comparing the distribution of relaxation times, the mineral oil shows characteristics closer to Debye relaxation.

With the aim of making accurate diagnoses of the condition of power transformer insulation, [21] determined the reference characteristics of the complex permittivity of the solid component of the insulation of these transformers. It was found that, as the temperature increases, the frequency dependencies of the permittivity and the imaginary component of the permittivity shift towards the high-frequency region, this being related to the fact that the relaxation time changes with increasing temperature. The values of the activation energies of the relaxation time of the permittivity 0.827 ± 0.0094 eV and of the imaginary component of the permittivity 0.883 eV were determined. These authors found that the Cole-Cole diagrams for the first stage (for low frequencies) are asymmetric and similar to those described by Dawidson-Cole relaxation. For the second stage (high frequencies), the diagrams are arc-shaped, which corresponds to Cole-Cole relaxation.

The present study consists of evaluating the dielectric properties of *Lagenaria siceraria* seed oil, several studies of which have shown the possibility of using it as a replacement for mineral oil [22] [23], but have not placed any particular emphasis on dielectric relaxation, which is a fundamental parameter for the quality of insulating oils. In fact, the relaxation behavior associated with molecular movement in vegetable oil makes it possible to understand the change in dielectric parameters in the fluid. Measurements of loss angle and the imaginary part of the

electrical modulus will be highlighted, with the emphasis on dielectric relaxation. These properties are the most reliable control parameters for transformer oil. Cole-Cole plot of the imaginary part of the permittivity as a function of its real part in calabash oil for different temperatures will be drawn and analyzed.

2. Experiment

2.1. Oil Sample

The oil used for the experiments is a pure, unrefined extract of calabash seeds. It is extracted by a cold process using an oil extractor in order to preserve the natural properties and antioxidant nature of the oils. *Lagenaria siceraria*, commonly known as calabash, is collected in a natural environment like in the West region of Cameroon.

2.2. Properties Measured

In particular, the relaxation phenomenon in calabash oil will be studied. To do this, we will measure dielectric properties (relative permittivity, loss factor or loss angle or dissipation factor, electrical modulus), discussing dielectric relaxation in each case. The Cole-Cole plot will be used to identify other relaxation processes in the oil sample.

2.2.1. Polarization Phenomenon

Polarization refers to the alignment or orientation of dipoles within a dielectric material when an external electric field is applied. The different types of polarization—electronic, ionic, dipolar, and interfacial—contribute to the overall dielectric behavior of a material at different frequencies and temperatures. In practice, the total polarization of a sample is measured as a function of field frequency, but is expressed in terms of complex electrical permittivity:

$$\varepsilon = \varepsilon' - j\varepsilon'' \quad (1)$$

where ε' is the relative permittivity of the sample and ε'' is the dielectric loss corresponding to the energy dissipated in the sample due to the coupling of the applied field with the dipolar fluctuations.

2.2.2. Dielectric Relaxation Theory

Dielectric relaxation refers to the delay in the response of a dielectric material's polarization to changes in an applied electric field. There are several analytical models to illustrate the frequency variation of the permittivity and the loss angle and consequently to study the polarization mechanisms:

- Debye Model

The variation of the complex dielectric permittivity as a function of frequency follows the Debye model for ideal liquid dielectric media (devoid of charges $\sigma = 0$) [24]:

$$\varepsilon^*(\omega) = \varepsilon_\infty + \frac{\varepsilon_s - \varepsilon_\infty}{1 + j\omega\tau} \quad (2)$$

with:

- ε_∞ is the high frequency dielectric permittivity ($\lim_{f \rightarrow \infty} \varepsilon'(f)$),
- ε_s is the static permittivity ($\lim_{f \rightarrow 0} \varepsilon'(f)$),

ω is the pulsation $\omega = 2\pi f$ and τ the relaxation time of the material.

We can separate the real and imaginary parts, and we then obtain $\varepsilon'(\omega)$ and $\varepsilon''(\omega)$:

$$\varepsilon^*(\omega) = \varepsilon_\infty + \frac{\varepsilon_s - \varepsilon_\infty}{1 + j\omega\tau} = \varepsilon'(\omega) + j\varepsilon''(\omega) \quad (3)$$

In Equations (3) $\varepsilon'(\omega)$ and $\varepsilon''(\omega)$ are the static and instantaneous (dielectric loss) relative permittivity (high frequency), respectively, and τ is called the Debye dielectric relaxation time [25]. The relaxation time is determined by:

$$\tau = \tau_0 e^{\frac{E_a}{kT}} \quad (4)$$

with:

- E_a is the activation energy (depends on the material),
- T is the temperature in degrees Celsius,
- k is Boltzmann's constant, $k = 1.38064852 \times 10^{-23} \text{ m}^2 \cdot \text{kg} \cdot \text{s}^{-2} \cdot \text{K}^{-1}$.

τ_0 is a constant that corresponds to a characteristic time.

Determining the activation energy experimentally requires finding the rate of reaction as a function of temperature.

From measurements at two temperatures, the activation energy is given by the relationship:

$$E_a = \frac{R * T_1 * T_2}{T_2 - T_1} \ln \left(\frac{V_2}{V_1} \right) \quad (5)$$

The dielectric loss or dielectric dissipation factor is equal to the quotient $\frac{\varepsilon''(\omega)}{\varepsilon'(\omega)}$; this quotient is also called tangent of the loss angle or $\tan \delta$, δ being the complementary angle of the phase shift between the voltage applied to the dielectric and the resulting current ($\tan \delta = \frac{\varepsilon''(\omega)}{\varepsilon'(\omega)}$).

- Cole-Cole model

According to Cole-Cole, a relaxation time distribution is needed to interpret experimental data. This Cole-Cole model is therefore a modification of Debye's theory by introducing a relaxation time distribution characterized by a factor α ($0 \leq \alpha \leq 1$)

$$\varepsilon^*(\omega) = \varepsilon_\infty + \frac{\varepsilon_s - \varepsilon_\infty}{1 + (j\omega\tau)^{1-\alpha}} \quad (6)$$

2.2.3. Dielectric Relaxation Using the Complex Electric Modulus (Interfacial Relaxation)

During the treatment of the dielectric sample to be used, several free charges

(impurities for example) can contaminate it. This gives rise to another type of relaxation called interfacial relaxation or Maxwell-Wagner-Sillars (MWS) relaxation [26]. To study this type of relaxation, the concept of electrical modulus is exploited. The complex electrical modulus is defined as the inverse of the complex permittivity. From Equation (3), we can write the following relation:

$$M^* = \frac{1}{\varepsilon^*} = \frac{1}{\varepsilon'(\omega) + j\varepsilon''(\omega)} \quad (7)$$

By separating the real and imaginary parts, we obtain:

$$M^* = M' + M'' \quad (8)$$

with:

M^* : The complex electric modulus;

M' : Real part of the electrical modulus;

M'' : Imaginary part of the electrical modulus.

When the dielectric constant obeys the Debye model, the IPC (inverse of the complex permittivity) itself obeys the Debye model and is written as follows [27]:

$$M^*(\omega) = M_\infty + \frac{M_s - M_\infty}{1 + j\omega\tau_M} \quad (9)$$

such as:

$$M_\infty = \frac{1}{\varepsilon_\infty}; \quad M_s = \frac{1}{\varepsilon_s} \quad \text{and} \quad \omega = 2\pi f \quad \text{the pulse.}$$

τ_M : The electrical relaxation time of the material expressed as a function of the Debye relaxation time.

Similarly, if dielectric permittivity obeys the Cole-Cole model, the following equation applies:

$$M^*(\omega) = M_\infty + \frac{M_s - M_\infty}{1 + (j\omega\tau_M)^{1-\alpha}} \quad (10)$$

The complex electrical modulus $M^*(\omega)$, is a valuable tool for highlighting the details of the relaxation information recorded in insulating materials in the low-frequency band [28] [29].

2.3. Measurement Techniques

The measurements are based on the principle of relaxation dielectric spectroscopy. Dielectric spectroscopy is widely used to study the response of a sample to an applied electric field of fixed or variable frequency [30]. It provides information about molecular dynamics as well as important material parameters such as static dielectric permittivity (ε) and continuous electrical conductivity (σ) [31].

2.4. Testing Device

In this work, the dielectric properties of the oil were studied over a wide range of frequencies (10^{-2} - 10^6 Hz) as a function of temperature (-35°C to 60°C). The device used for the measurement is an impedance meter (frequency response

analyzer Analyzer-Alpha A (Novocontrol). The sample holder is the Novocontrol BDS20. The available frequency range of the device extends from 3 μHz to 10 MHz with a maximum applied voltage of 1 V. The measurement limit in $\tan(\delta)$ is approximately 3×10^{-5} for measurements in the range between 10 Hz - 100 kHz and for capacitance between 50 pF - 2 nF.

3. Results and Discussion

3.1. Study of Dielectric Losses with Emphasis on Relaxation

Figure 1 shows the frequency dependence of dielectric losses in calabash oil, for different temperatures. The frequency dependence of dielectric losses in calabash seed oil at different temperatures reveals critical insights into the molecular dynamics, relaxation processes, and overall dielectric behavior of the oil. Dielectric losses, indicate the energy dissipated as heat when the material is subjected to an alternating electric field. The frequency dependence of dielectric losses in calabash oil is divided into three distinct regions:

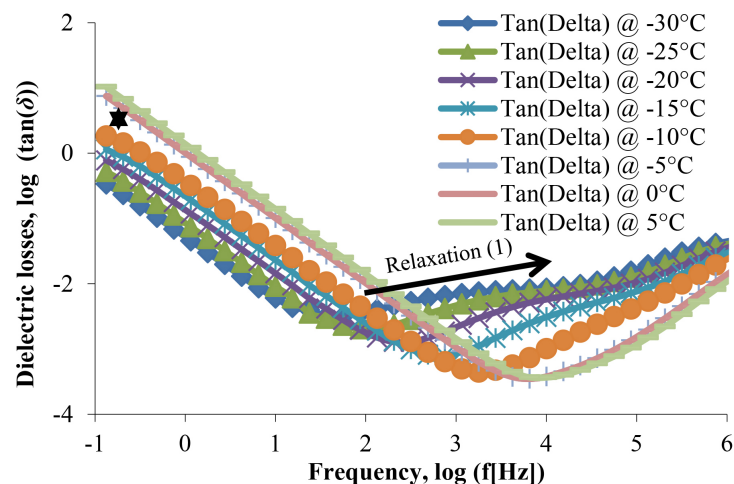


Figure 1. Dielectric losses in calabash seed oil as a function of frequency at different temperatures, identification of the relaxation process.

High dielectric losses at low frequencies are primarily due to the efficient alignment of dipolar molecules with the applied electric field, enhanced ionic and space charge effects, and significant interfacial polarization. These factors can lead to greater energy dissipation at low frequencies as the material undergoes substantial polarization and relaxation processes.

Mid-Frequency Region (Relaxation Peak): As the frequency increases, the dipoles begin to lag behind the rapidly oscillating electric field, leading to a peak in dielectric losses known as the relaxation peak. This peak occurs at a characteristic frequency corresponding to the natural relaxation time of the dipoles. In fact, from a frequency above 10^4 Hz, we observe an increase in losses towards high frequencies. This represents losses due to dipolar relaxation. Indeed, an increase in temperature for a given frequency should lead to a decrease in losses, which is

not the case for conduction losses. This analysis is borne out by the results shown in **Figure 1**.

A dissipation peak is also identified, shown in **Figure 1** by the arrow and called relaxation (1).

In the high-frequency region, dielectric losses in calabash seed oil increase slowly due to the limitations of dipole reorientation, the dominance of electronic polarization, and potential residual absorption mechanisms. The dielectric response becomes less influenced by dipolar processes and more by rapid electronic interactions and any minor high-frequency absorption phenomena. The overall effect is a gradual rise in dielectric losses as frequency increases, reflecting the complex interplay of various polarization and absorption mechanisms at high frequencies.

3.2. Molecular Mobility Localized in the Sample

Figure 2 displays the evolution of the imaginary part of permittivity as a function of temperature for frequencies between 0.01 and 106 Hz. The study of dielectric losses in calabash oil, particularly their frequency dependence at different temperatures, is essential for understanding how this natural oil behaves as an insulating material or in other dielectric applications. The complex dielectric permittivity was measured. In dielectric materials like calabash oil, dielectric losses are generally characterized by the imaginary part of the dielectric constant, $\epsilon''(\omega)$. This component indicates the energy dissipated as heat when an alternating electric field is applied. The frequency dependence of dielectric losses in calabash oil is likely to exhibit the following behavior:

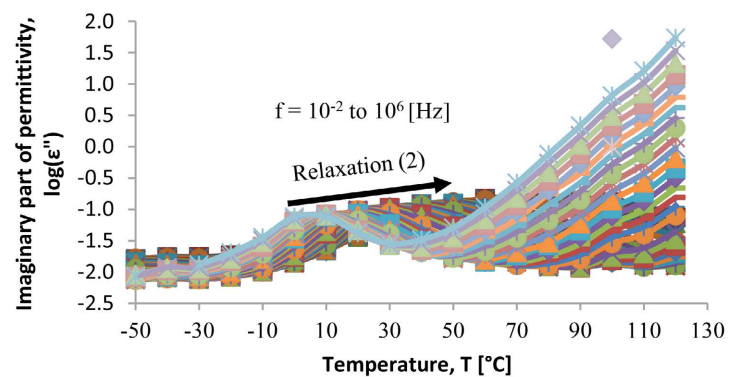


Figure 2. Evolution of the imaginary part of permittivity as a function of temperature for frequencies between 0.01 and 10⁶ Hz.

At lower frequencies, the dipolar molecules in the calabash oil can easily follow the slowly varying electric field. Consequently, the dielectric losses are relatively low because the alignment of dipoles with the field requires less energy dissipation.

As the frequency increases, there comes a point where the dipoles cannot fully realign with each cycle of the electric field. This results in a peak in dielectric

losses, known as the relaxation peak. The frequency at which this peak occurs is related to the relaxation time of the dipolar molecules in the oil. One can observe a relaxation mode (relaxation 2) that broadens and shifts towards higher temperatures as the frequency increases. This broadening indicates a wider distribution of relaxation times, which could be due to the presence of different types of molecules or interactions within the oil that respond differently to temperature changes.

At frequencies much higher than the relaxation frequency, the dipoles are unable to follow the rapidly changing field, leading to a decrease in dielectric losses. The energy dissipation stabilizes at a lower level, often dominated by other mechanisms like conduction losses or electronic polarization.

Temperature plays a crucial role in the behavior of dielectric losses in calabash oil. As the temperature increases, the thermal energy provided to the dipolar molecules increases, which enhances their mobility. This typically results in a shift of the relaxation peak to higher frequencies. In other words, the frequency at which maximum dielectric loss occurs increases with temperature because the dipoles can realign more quickly with the applied field. The magnitude of the dielectric loss peak may also vary with temperature. Higher temperatures generally increase the magnitude of the peak because the increased molecular activity leads to greater energy dissipation. However, this effect can depend on the specific structure and composition of calabash oil.

The Arrhenius diagram can be used to analyze this relaxation mode. It can be seen in **Figure 3**. The obtained result is similar to that obtained in the work of [32].

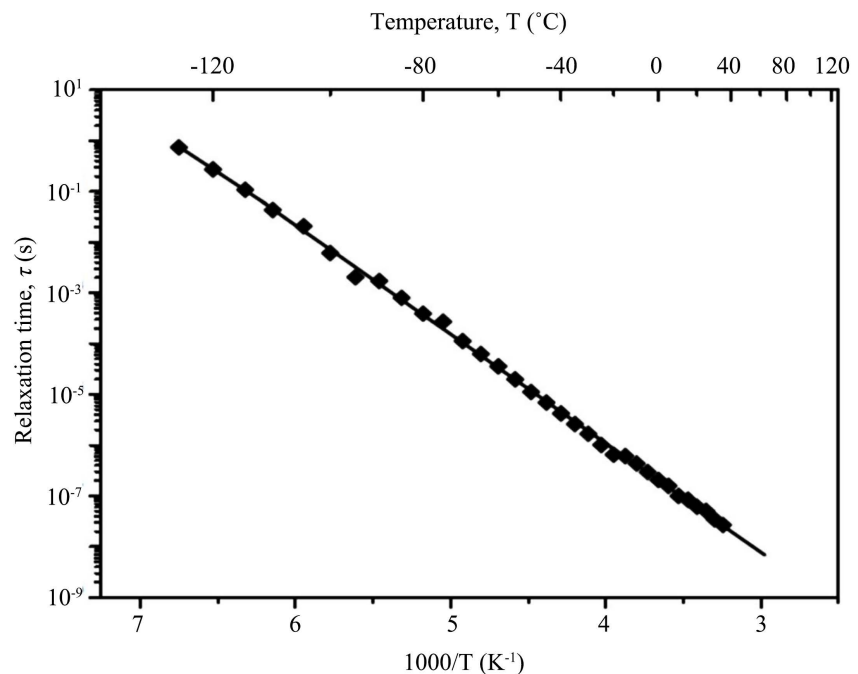


Figure 3. Arrhenius diagram of the sample's low-temperature relaxation mode.

This diagram shows the evolution of the relaxation time as a function of the inverse of the temperature. **Figure 3** shows that $\ln\tau$ decreases linearly with $1/T$. This linearity confirms that the relaxation time follows the Arrhenius law, which is indicative of a thermally activated process. The slope of the Arrhenius plot ($-E_a/k_B$ provides the activation energy for the relaxation process. A steeper slope indicates a higher activation energy, meaning that a larger amount of energy is required to facilitate the relaxation process. Conversely, a shallower slope suggests lower activation energy. Equation (8) allows us to express the activation energy $E_a = 918.79 \text{ J}\cdot\text{mol}^{-1}$. Since this energy is positive for our vegetable oil sample, it indicates an endothermic reaction in the fluid. Therefore, to obtain rotation of the unsaturated molecules in the oil sample with short relaxation times, a high energy is essential.

3.3. Study of the Imaginary Part of the Electrical Module

It has been shown that in order to minimize the effect of conduction on dissipative processes that have not yet been detected, it is necessary to study the electrical modulus; in fact, conduction has a strong impact on dissipation.

Figure 4 shows the frequency dependence of the imaginary part of the electric modulus (M'') of the calabash oil sample for different temperatures. This illustration shows that the imaginary part of the electric modulus (M'') of this oil exhibits a single relaxation peak materialised by the arrow named relaxation (3) at low frequencies. It increases with increasing frequency until it reaches its maximum value (M''_{\max}) at a frequency beyond which it begins to decrease. This confirms the previous analysis of the usefulness of measuring the electrical modulus.

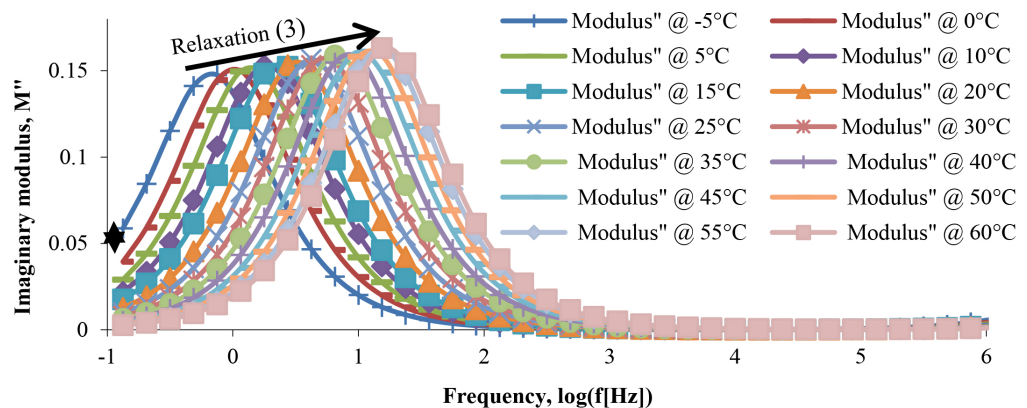


Figure 4. Imaginary modulus M'' as a function of frequency in a temperature range T from -5 [$^{\circ}\text{C}$] to 60 [$^{\circ}\text{C}$], identification of a new relaxation process numbered (3).

It is also observed that the relaxation peak also shifts to higher frequencies as the sample temperature increases; this would suggest a decrease in the relaxation rate of the process and would also indicate that dielectric relaxation is thermally activated. We can then state that the relaxation is not Debye-like in the high-frequency region. Analysis of the complex electrical modulus provides a better

separation between dipolar relaxations and losses with a significant ionic distribution than analysis of the complex permittivity. This is because, at low frequencies, the complex permittivity and the dielectric dissipation factor are very much affected by the effect of the polarization and conductivity of the electrode.

3.4. Cole-Cole Layout

From the Cole-Cole relations, the graphical representation of the variations of the real part (ϵ') of the complex permittivity of the calabash oil sample as a function of the imaginary part (ϵ'') defining the dielectric losses in the oil is given in **Figure 5**. The plot is made for three temperature levels.

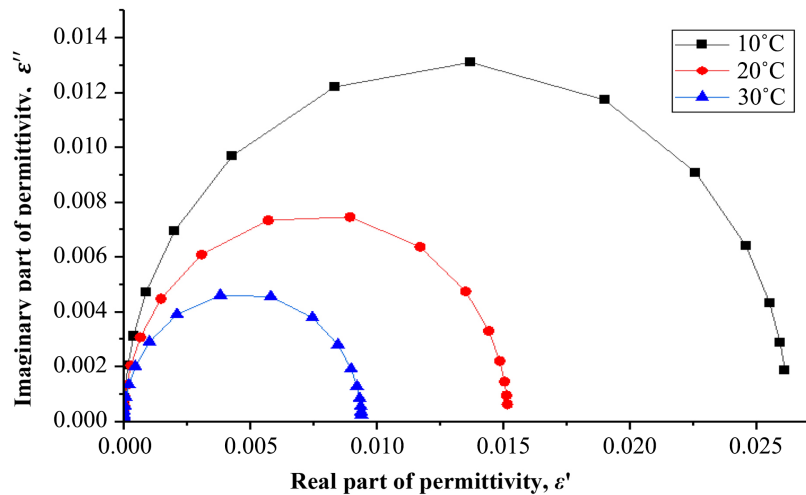


Figure 5. Cole-Cole plot of the imaginary part of the permittivity as a function of its real part in calabash oil for different temperatures.

There is an increase in the area of the semicircles and a movement of the intercept of a semicircle towards the highest value of ϵ' ; this would represent an increase in capacitance, thus justifying the existence of a negative temperature coefficient of resistance in the fluid. Furthermore, the fact that the Cole-Cole plot exhibits semicircles is indicative of the presence of Debye relaxation.

The independence of the dynamic process with respect to temperature is demonstrated by the overlap of all the curves in **Figure 5**; there is therefore a relaxation process in the conductivity of the sample studied.

3.5. Dielectric Constant

Table 1 below shows the dielectric constant of different oils. It presents a comparison between calabash seed oil and other oils. It appears that calabash oil could be better insulating compared to other oils, since it has higher relative permittivity. Indeed, it has been proven that a higher relative permittivity value for insulating oil indicates that it is less exposed to electric field stresses and that this condition is an advantage for insulating quality. It means that the higher the dielectric constant, of insulating liquid is better for a more uniform electric field [33].

Table 1. Dielectric constant of different insulating oils.

Insulating oil	Dielectric constant at 25°C	references
Mineral (transformer) oil	2.2	[34]
Sunflower oil	2.87	
Soybean oil	2.00	[16]
Coconut oil	2.65	
Groundnut oil	2.22	
Calabash seed oil	3.19	Studied sample

4. Conclusion

The study of the dielectric properties of calabash oil with an emphasis on dielectric relaxation enabled us to identify two relaxation processes. A relaxation peak was identified during the measurement of the loss angle. The losses were found to increase with frequency, which would represent losses due to dipolar relaxation. Furthermore, it was observed that the relative permittivity decreases with increasing temperature and frequency; this decrease with frequency being in phase with the relaxation process initially identified. A study of the imaginary part of the electrical modulus identified a single relaxation peak (a second relaxation process) at low frequencies. The shift of this relaxation peak towards higher frequencies with increasing temperature leads to a decrease in the relaxation rate of the process and leads to the conclusion that dielectric relaxation is thermally activated, so this relaxation is not of the Debye type in the high-frequency region. The Cole-Cole plot, showing semicircles, reveals the presence of Debye relaxation. At the end, the curves overlap, indicating the presence of a relaxation process in the conductivity of the sample studied. The results reveal that calabash oil has the highest dielectric constant at temperature 25°C. However, as both the frequency and temperature increases the dielectric constant decreases continuously except for values beyond 10^4 Hz. At frequencies greater than 10^4 the dielectric constant value increased as the temperature increased. This suggests that the dielectric constant of calabash seed oil is easily polarizable at lower frequency and temperature. These results suggest that *Lagenaria siceraria* seed oil is a promising candidate for use as an insulating oil in transformers, combining effective performance with environmental and economic advantages.

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Conflicts of Interest

The authors declare no conflict of interest.

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