Effect of clay/nepheline tailing ratio on the dielectric relaxation and conduction mechanism of the conventional ceramic

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Abstract
The dielectric relaxation and conduction mechanism of 40 wt% nepheline tailing and 60 wt% clay-based ceramic, i.e., the 40/60 ceramic, have been investigated over wide ranges of frequency and temperature, using a broadband dielectric spectrometer (BDS) and then compared with the data recently reported for the 50/50 ceramic. Both 40/60 and 50/50 ceramics were fabricated by grinding the raw materials to be very fine, wet homogeneously mixing, drying and finally firing at 1200 °C. Their crystalline phases identified by X-ray diffraction were quartz, hematite, cristobalite, and albite. The 40/60 ceramic of lower glassy phase (nepheline tailing) content displays lower crystallinity than the 50/50 ceramic. Its conduction activation energies (Eac) show values between 0.12 and 0.32 eV, corresponding to the activation energy of oxygen vacancies (Vo++ ~ 0.22 eV). As oxygen vacancies migrate at relatively low operating voltages, the fabricated ceramics would be promising in manufacturing the random access memory (RAM), taking into consideration that the 50/50 ceramic is more useful than the 40/60 ceramic. As a result, nepheline tailing or the glassy phase component may generate more oxygen vacancies and thus enhance the ceramic electrical properties. Finally, the conduction mechanism of both ceramics is described via the correlated barrier hopping (CBH) model.

Keywords Nepheline tailing · Clay · Permittivity · Activation energy

1 Introduction
The excellent thermal and electrical insulating properties of ceramics make them useful in many applications; micro-electronic devices, ceramic heaters, heating elements, semiconducting material, optical, magnetic, and high-voltage applications, as well as in power transmission lines [1–5]. The technical ceramics like aluminum oxide, zirconium oxide, silicon carbide, barium titanate, lead titanate, lead zirconate titanate, etc., have been widely used for electrical, electronic, optical, and magnetic applications [6–15]. Most of their products are given in powdered form, where chemical reactions are needed to assure sufficient purity. Therefore, the raw materials and processing of most technical ceramics are very costly. On the contrary, the main raw materials required for fabricating the conventional ceramics are available in nature and inexpensive, such as ball clay, china clay, feldspar, silica, dolomite, talc, calcite, and nepheline syenite. Each contributes a certain property such as dry strength, plasticity, shrinkage, etc. to the ceramic body. By careful selecting of these materials, desired properties are acquired for the final product. Due to the complex structure of the conventional ceramics, little attention has been paid to studying their electrical or dielectric properties, where many anomalous results are difficult to interpret. Although we reported some enhancements in the dielectric properties of these ceramics, upon sintering at 1100 °C and adding LiF, B2O3, ZnO, or MnO [16–21], our physical understanding of the relaxation phenomenon is still poor. Therefore, in our recent published work, we tried to understand the relaxation phenomenon on the basis of grains and grain boundaries capacitive contributions, which are related to some defects such as oxygen vacancies and valence fluctuation of ions [21]. To achieve that, Abu Khrug nepheline tailing (Eastern Desert, Egypt) and Wadi Abu Suberia clays (near Aswan, Egypt) were fined and wet homogeneously mixed at the same ratios to produce the 50/50 ceramic. Then, dielectric spectroscopy as a powerful technique has been applied to analyze...
the molecular relaxation processes associated with the bulk grains (G), grain boundaries (GBs), which are related to some defects such as oxygen vacancies and valence fluctuation of ions. Further, it analyzes the dielectric losses which originate from the conduction losses, dipole losses, and/or vibrational losses [22]. The experimental results revealed the existence of two relaxation processes associated with the grain boundaries (GBs) and grains in the low- and high-temperature regions, respectively. Further, the relaxation process associated with grain is predominate more over GBs relaxation within the temperature range studied. Interestingly, we reported an anomaly in dielectric behavior of the 50/50 ceramic at −40 °C, at which all dielectric properties changed abruptly. For importance, the current paper is a complementary dielectric study to evaluate the effect of 40 wt% nepheline tailing on the electrical properties of the 40/60 ceramic and then compared it to that recently reported for the 50/50 ceramic.

2 Materials and methods

2.1 The raw materials and ceramic processing

The clay/nepheline tailing (C/NT)-based ceramics were fabricated from the inexpensive and available Egyptian raw materials; Wadi Abu Suberia clay (near Aswan, Egypt) and nepheline tailing; product of the beneficiation (Eastern Desert, Egypt). Nepheline syenite ore is an essential constituent in ceramics and glass raw material meals, as a flux and as a source of alumina. However, it contains some environmentally hazardous elements such as uranium, thorium, and radon, which are usually eliminated or reduced to the allowable limits by beneficiation. The beneficiation of an Egyptian nepheline syenite rock (Abu Khrqu locality, Eastern Desert, Egypt) has been carried out by magnetic separation and flotation techniques, separately or in combination. Under optimum operating conditions, magnetic separation followed by reverse anionic flotation gave a concentrate assaying 0.2% Fe₂O₃ and about 24% Al₂O₃ at alumina recovery of about 80%. The radioactive elements were reduced in the concentrate to lower levels than their levels in the original rock. Basically, three types of nepheline syenite (NS) can be given as:

1. Concentrate NS
   It contains the highest alumina content and the lowest iron content (0.2% Fe₂O₃).
2. Medium NS
   It contains a relatively high iron content (Fe₂O₃), low alumina content and high amount of silica.
3. Nepheline tailing

   The nepheline tailing (NT) is the final product beneficiation process of nepheline syenite (NS) rock. It is characterized by having a high amount of alkali in the form of feldspathoid, feldspar, and pyroxene. In addition, higher content of iron (Fe₂O₃ > 19) and silica as compared with 1 & 2 types.

The obtained nepheline tailing as well as clay were fired and then their chemical composition given elsewhere [17]. The raw materials were separately wet mixed at two different ratios (40/60, 50/50) in a porcelain ball mill for 1 h to ensure the proper homogeneity, then dried at 110 °C. After that, both ceramic compositions were processed by a semi-dry pressing under a pressure of 30 KN, dried for 24 h at room temperature, then over night at 110 °C. Finally, they were fired at 1200 °C with a temperature interval of 25 °C and 1-h soaking time. Two disc-shaped samples prepared by the slip casting process were obtained namely the 40/60 and 50/50 ceramics. For the dielectric measurements, the two surfaces of each ceramic were coated with a silver paste to ensure good contact with the electrodes of the cell capacitor.

2.2 Dielectric measurements

The dielectric measurements were carried out over a wide frequency range (10⁻¹–10⁷ Hz) and temperature range (~100 to 200 °C) utilizing a high-resolution alpha analyzer with an active sample head (Novocontrol GmbH), BAM institute, Berlin, Germany. The measurements were performed isothermally, where the temperature was controlled by a Quatro Novocontrol cryo-system with a stability of 0.1 K. The device is provided with Windata software program to calculate the sample electrical properties from the output parameters, i.e., the electrical capacitance (C), and the loss tangent (tanδ) as follows:

\[
\varepsilon'(\omega) = \frac{Cd}{\varepsilon_0 A} \tag{1}
\]

\[
\varepsilon'' = \varepsilon' \tan \delta \tag{2}
\]

\[
\sigma_{ac} = 2\pi f \varepsilon_0 \varepsilon'' \tag{3}
\]

\[
M^*(\omega) = \frac{1}{\varepsilon''(\omega)} = M' + iM'' = \frac{\varepsilon'}{\varepsilon'^2 + \varepsilon''^2} + i\frac{\varepsilon''}{\varepsilon'^2 + \varepsilon''^2} \tag{4}
\]

where \((\varepsilon', M')\) and \((\varepsilon'', M'')\) are the real and imaginary parts of the complex permittivity \((\varepsilon^*)\) and electric modulus \((M^*)\). \(f\) is the applied field frequency in Hertz, \(\varepsilon_0 (= 8.85 \times 10^{-12} \text{ F/m})\) is the vacuum permittivity, and \(\sigma_{ac}\) is the AC conductivity. \(d\) and \(A\) are the thickness and surface area of each ceramic compositions.
3 Results and discussion

Note that microstructure and phase identification studies for 40/60 and 50/50 ceramics were investigated in our previous work using scanning electron microscope (SEM) and X-ray diffraction (XRD) techniques [17]. The XRD study indicated that the main crystalline phases developed in both ceramics were quartz, hematite, cristobalite, and albite. These phases show lower crystallinity in 40/60 ceramic than in 50/50 as listed in the Table 1. Further, SEM study for the 40/60 ceramic shows a higher proportion of glassy phase with crystalline phases outcropping with different sizes. This results from the nepheline tailing dissolving the primary mullite present in the clay, forming a glassy phase rich in silica. Based on this, the clay strongly affects the overall ceramic crystallinity.

To get a deep understanding of the ceramic composition dependent on dielectric relaxation and conduction mechanism, the dielectric properties as well as AC conductivity of the 40/60 ceramic are investigated for some detail, then briefly compared to those reported previously for the 50/50 ceramic.

3.1 Permittivity and dielectric loss

Figures 1 and 2 show the frequency and temperature dependencies of the permittivity ($\varepsilon'$) and dielectric loss ($\varepsilon''$) of the 40/60 ceramic. Both properties are affected by temperature and frequency; they show a significant decrease with increasing frequency and an increase with increasing temperature similar to that previously reported for the 50/50 ceramic. The strong dispersion in $\varepsilon'$ at high temperature and low frequency is an indicative of the presence of thermal activated charges, i.e., space charges, charged defects, and defect complexes. Recently, it has been reported that space charge polarization is mainly responsible for the dielectric dispersion at low frequencies [23]. As frequency increases, space charge polarization becomes less effective because the recombination would be faster. This explains why $\varepsilon'$ and $\varepsilon''$ show a decrease as frequency increases. Further, the frequency-dependent properties follow the Kramers–Kronig relation at $T < 60 \, ^\circ C$; $\varepsilon'$ shows a dispersion while $\varepsilon''$ shows a relaxation peak [24]. Accordingly, these peaks are associated with the dynamic relaxation process of a part of a molecule (functional groups etc.) or a molecule as a whole. Single and two relaxation peaks are displayed at $T = -100 \, ^\circ C$ and in the temperature range ($-80 \, ^\circ C$ to $60 \, ^\circ C$), where their positions are shifted to higher frequency with increasing temperature and then they disappear at $T > 60 \, ^\circ C$. Further, both $\varepsilon'$ and $\varepsilon''$ exhibit high values even at low temperatures. The question now, what are possible reasons for such high values and origin of the observed relaxation peaks. The answer is

| Table 1 | The crystalline phases developed in the 40/60 and 50/50 ceramics are characterized by XRD |
|---------|--------------------------------------------------------------------------------------------------|
| Ceramic | Quartz | Cristobalite | Albite | Hematite | Crystallinity (%) | Amorphous phase (%) |
| 40/60   | 18.8   | 6            | 2.9    | 6.9      | 34.6            | 65.4               |
| 50/50   | 28.6   | 11.9         | 9.3    | 22.5     | 72.3            | 27.7               |

![Fig. 1](image1.png)  
Fig. 1 Frequency dependence of permittivity ($\varepsilon'$): a at $-100 \, ^\circ C$, b at temperature range ($-80$ to $60 \, ^\circ C$) and c at temperature range (80 to $200 \, ^\circ C$) for the 40/60 ceramic

![Fig. 2](image2.png)  
Fig. 2 Frequency dependence of dielectric loss ($\varepsilon''$): a at $-100 \, ^\circ C$, b at temperature range ($-80$ to $60 \, ^\circ C$) and c at temperature range (80 to $200 \, ^\circ C$) for the 40/60 ceramic
not easy as there have not been any previous work on clay/nepheline tailing-based ceramic supporting this behavior. Hence, data interpretation will be completely dependent on our experience in studying the dielectric properties for different ceramic materials. Different reasons for high values in $\varepsilon'$ and $\varepsilon''$ have been suggested. One is due to the high resistive GBs generally cover the conducting G, forming a core–shell-like microstructure or an internal barrier layer capacitor (IBLC) that exhibits large capacitance [25]. A second could be due to an increase in the crystallographic dimensions (CD) of the ceramic lattice which results in a large electric dipole, giving rise to an increase of $\varepsilon'$ and $\varepsilon''$ at high temperatures. The relaxation peak in $\varepsilon''$ spectra at $-100 \, ^\circ C$ may reveal a comparable resistance value of G and GBs or it may reveal a large resistance difference between them. Further, the peak shows an intensity increase and shift to higher frequency with increasing temperature, indicating a resistance decrease of G or/and GBs. Thus, the overall polarization increases, causing an increase in both $\varepsilon'$ and $\varepsilon''$. It is interesting to note that $\varepsilon''$ has extremely high values in high-temperature regions due to the space charge polarization and/or the conductivity of insulating ceramics increases with increasing temperature. Usually, the origin of different relaxation peaks in polycrystalline systems can be due to bulk grain (G), grain boundary (GB), sub-grain boundary, electrode effect, and/or interface functional groups. Taking into consideration, the electrode/interface effect (electrode polarization) occurs at lower frequency, GB at intermediate frequency, whereas G at higher frequency. Recently, for the 50/50 ceramic, we attributed the relaxation processes to the capacitive contributions of G and GB [21]. Similarly, the frequency and temperature dependence of $\varepsilon'$ and $\varepsilon''$ can be explained by considering the 40/60 ceramic as an inhomogeneous medium made of two Maxwell Wagner-type layers consisting of semiconducting grain (G) separated by insulating grain boundaries (GBs) [26, 27].

### 3.2 The complex electric modulus

For properly understanding the possible contribution of G and GBs to the dielectric data of 40/60 ceramic, usually the complex electric modulus plot ($M''$ vs. $M'$) is suggested to solve the corresponding relaxation processes, because it highlights the smallest capacitance and suppresses the electrode polarization effects [28, 29]. Furthermore, it provides more deeply information about the intrinsic contribution inside the bulk and precisely separates the G and GBs relaxation processes. In the study, the complex electric modulus or Nyquist plot doesn’t display semicircles when it is represented in linear or on the log–log scale (Fig. 3). This happens in the case of non-uniform distribution of the current at the electrode surface or minimal distribution of the charge storage and Ohmic contact of the electrode/ceramic interfaces [30, 31]. Absence of the semicircle doesn’t mean an absence of a charge transfer resistance. Generally, Fig. 3 shows a half semicircle, which indicates the presence of charge transfer resistance.

### 3.3 Electrical conduction mechanism

It is generally pointed out that the frequency-dependent electrical conductivity in solids arises from the current flow by hopping of the localized charge carriers like electrons and/or ions. The relationship between the relaxation and conduction behavior of ceramics can be investigated by studying conductivity as a function of frequency at various temperatures. Therefore, AC conductivity ($\sigma_{ac}$) of the 40/60 ceramic has been investigated as a function of frequency and temperature as illustrated in Fig. 4. In the low-frequency region and high temperatures, $\sigma_{ac}$ shows a frequency independent nature, i.e., a plateau-like behavior, which gives rise to DC-conductivity ($\sigma_{dc}$) arising from the random diffusion of the free charge carriers via activated hopping process [32]. In this case, charge carriers hope randomly in all directions and thus no net current is detected. This is in consistency with the high values of dielectric loss ($\varepsilon''$) (Fig. 2). Values of $\sigma_{dc}$ are obtained by extrapolating $\sigma_{ac}$ to zero frequency using the Jonscher equation, $\sigma_{ac}=\sigma_{dc}+A\omega^{s}$ [33], where $A$ is the temperature-dependent parameter that determines the strength of polarizability, $\omega$ ($2\pi f$) is the angular frequency, and $s$ ($0<s<1$) is the frequency exponent that determines the conduction mechanism nature [34]. The apparent frequency-dependent conductivity indicates the dominance of $\sigma_{ac}$ at high values of frequency and temperature. The high frequency increases the electronic jumps between the localized states [35], whereas the high temperature earns the free
ions (Zn$^{2+}$, Al$^{3+}$, Mn$^{3+}$, Fe$^{3+}$, and Ca$^{2+}$) enough thermal energy to overcome the barrier to lattice diffusion and thus move easily upon applying the electric field. The inset of Fig. 4 shows the log $\sigma_{dc}$ vs $1/T$ plot. The activation energy of DC conduction ($E_{dc}$) was calculated to be 0.63 eV, using the Arrhenius relation:

$$\sigma = \sigma_o e^{-E_a/k_BT} \quad (5)$$

where $\sigma_o$ is the conductivity at infinite temperature, $E_a$ is the electronic activation energy, and $k_B$ is the Boltzmann constant (8.61733 x 10$^{-5}$ eV K$^{-1}$). The activation energy may arise from the existence of charge carriers inside the grain and/or some extrinsic charge carriers (at elevated temperature) due to using a silver electrode.

The temperature dependence of AC conductivity ($\sigma_{ac}$) of the lower glassy phase content ceramic (40/60 ceramic) at $10^2$ and $10^3$ Hz is shown in Fig. 5. In the figure, $\sigma_{ac}$ increases steadily in the higher temperature region (II) while it increases sharply in the lower temperature region (I), indicating two conduction mechanisms having two activation energies ($E_{ac}$). $E_{ac}$ that ranged from 0.14 and 0.19 eV (II) corresponds to the activation energy of the intrinsic oxygen vacancy defects (Vo$^{++}$ ~ 0.22 eV). This is in agreement with the data reported previously for the 50/50 ceramic [21] and the perovskite oxides treated at high firing temperatures [36–38]. While, $E_{ac}$ that displays higher values between 0.40 and 0.52 eV (I), possibly be correspond to the activation energy of the valence fluctuation ions. Accordingly, the electrical conductivity of the high (50/50) and the low (40/60) glassy phase ceramic are dominated by the cooperative motion of oxygen vacancies and free ions. The existence of oxygen vacancies in such ceramics is the key principle of resistance change in oxide-based resistive memory (OxRAM). The practical usefulness of these oxygen vacancy migrations take place at relatively low operating voltages. In view of this, the fabricated ceramics could be useful in manufacturing random access memory (RAM).

To investigate the conduction mechanism nature of the 40/60 ceramic, its $s$ values were plotted as a function of temperature as shown in Fig. 6. As clear in the figure, $s$ decreases with increasing temperature. This behavior is commonly interpreted by the correlated barrier hopping (CBH) model [39]. According to this model, if hopping takes place between the localized states with a random distribution, $\sigma_{ac}$
is directly proportional to \(\omega^s\), where \(0.5 < s < 1\). Further, the low values of \(s\) indicate a multi-hopping process while the higher values indicate a single hopping process \([34]\). This leads one to conclude that the conduction mechanism in the 40/60 ceramic is a single hopping.

### 3.4 Comparison study

The ceramic composition effect has been investigated by studying the dielectric properties of the 50/50 and 40/60 ceramics together in one plot as a function of temperature at constant frequency (0.1 Hz), see Figs. 7 and 8. Both the ceramics show a permittivity and conductivity increase with the temperature increase, which could be possibly due to broking of the intermolecular forces between the ceramic components, causing enhancements of thermal agitation. Further, as the temperature increases, the polar group contained in the ceramic compositions will be freer and able to follow the electric field changes, i.e., overall polarization increases and thus \(\varepsilon'\) increases. The temperature also lowers the viscosity of the glassy phase, which in turn allows the dipole and ions to respond easily to the external electric field, i.e., polarization increases \([40]\). The glassy phase content and crystallinity may also play a significant role in the electrical behavior of these ceramics. For being nepheline tailing rock a source of the glassy phase that supplies different ions (Fe\(^{3+}\) and Ca\(^{2+}\)), it may enhance the ceramic dielectric properties. For the 50/50 ceramic of higher glassy phase content, it shows higher permittivity and conductivity values than the 40/60 ceramic. From the figures, it can be noticed that the glassy phase seems to dominate the ceramic dielectric properties at \(T > -40 \,^\circ\text{C}\), while at lower temperatures, the crystallinity is dominated. So, the 40/60 ceramic of higher crystallinity (67.4%, Table 1) displays higher permittivity and conductivity values than a 50/50 ceramic.

### 4 Conclusion

The nepheline syenite rock under study has been concentrated with the aim of producing a high-quality ceramic having a high amount of alkali in the form of feldspathoid, feldspar and pyroxene alumina content, i.e., nepheline tailing. The effects of frequency, temperature, nepheline tailing, the crystallinity, and the oxygen vacancy defect on the ceramic dielectric properties can be summarized as follows:

i. The permittivity decreases while the conductivity increases with increasing frequency.

ii. Both permittivity and conductivity show an increase with increasing temperature. Their behaviors are highly affected by the crystallinity and the glassy phase content below and above \(-40 \,^\circ\text{C}\), respectively.

iii. Ceramic with higher glassy phase content, i.e., the 50/50 ceramic, shows higher dielectric properties than the 40/60 ceramic.

iv. Oxygen vacancy defect (Vo\(^{++}\) ~ 0.22 eV) dominates the conduction activation energy \((E_a)\) of the 50/50 ceramic within the whole temperature range \((-100\) to \(200 \,^\circ\text{C}\)). It dominates \(E_a\) of the 40/60 ceramic within a temperature range \((120\) to \(200 \,^\circ\text{C}\)) while fluctuations of ions dominate \(E_a\) at lower temperatures.

As oxygen vacancies migrate at relatively low operating voltages, the fabricated ceramics, particular the 50/50 ceramic, would be promising in manufacturing the
random access memory (RAM). The interesting features of these ceramics make them technologically important and competitive to other alternative materials due to their cost-effectiveness.

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Declarations

Conflict of interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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