ISSN: 0067-2904
GIF: 0.851

Preparation and Characterization of BeO-Supported Feldspar Porcelain

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Abstract

Due to the specific characteristic of porcelain, the insertion of Beryllium oxide has been studied. The basic materials and quantities were selected carefully. In this work, porcelain containing 32 wt% feldspar, 24 wt% quartz and 44 wt% clay was synthesized and beryllium oxide (BeO) (1 wt.%, 3 wt.%, 5 wt% and 7wt%) were add. The basic and new composition porcelain powders were uniaxially compacted into standard samples dimensions and fired at various sintering temperatures, 1100°C, 1300°C, and 1450°C then held for 2 hour in a furnace.

The effects of sintering temperatures and beryllium oxide content on mechanical, electrical and structural properties were studied. The increasing of sintering temperature on the basic porcelain leads to an enhancement in bulk density and compressive strength. Basic porcelain which suffering lower sintering temperature (1300°C) introduces higher dielectric constant values in contrast with that suffering higher temperature (1450°C). The addition of BeO to the basic porcelain leads to decrease the bulk density and compressive strength. Porcelain with lower sintering temperature introduces higher έ values in contrast with higher temperature. The FTIR measurement show the existence of absorption at 800 cm^{-1} which attributable to Si-O-Si symmetrical stretching vibration band in all prepared samples. Band near 780 cm^{-1} is attributed to the vibration of a beryllium atom moving in a tetrahedron of fixed oxygen atoms, while the absorption bands in the region 400-1500 cm^{-1} are due to the Be-O stretching and bending vibrations.

Keywords: BeO; Porcelain; Mechanical properties, Dielectric, FTIR

خصائص ومواصفات خزف الفلدسبار المدعم باوكسيد البريليوم

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الخلاصة

نظرا للاهمية الخاصة للخزف، تم دراسة تأثير اضافة اوكسيد البريليوم اليه. وقد تم اختيار المواد الأساسية والكميات بدقة. تم في هذا العمل تصنيع الخزف الذي يحتوي على 32٪ فلدسبار، 24٪ كوارتز و 44٪ من الطين وتم اضافة (1٪ ، 3٪ ، 5٪ و7٪) من أوكسيد البريليوم (BeO). وقد تم كبس مساحيق الخزف الأساسي و ذي المكونات الجديدة بطريقة الضغط الاحادي المحور للحصول على عينات قياسية الابعاد وتم حرق العينات بدرجات تلبيد مختلفة 1100م° ، 1300 م°، و1450 م° درجة مئوية لمدة ساعتين في الفرن.

تمت دراسة تأثير درجات الحرارة ومحتوى أوكسيد البريليوم على الخواص الميكانيكية والكهربائية والهيكلية للخزف المصنع. لقد وجد ان زيادة درجة حرارة التلبيد للخزف الأساسي يؤدي إلى تحسين في الكثافة وقوة الانضغاط. الخزف الأساسي الذي عانى من درجة حرارة تلبيد أقل (1300 درجة مئوية) اعطى قيم ثابت عزل أعلى بالمقارنة مع تلك التي عانت من درجة حرارة تلبيد اعلى (1450 درجة مئوية). ان إضافة اوكسيد البريليوم إلى الخزف الأساسي أدى إلى تقليل الكثافة الظاهرية وقوة الانضغاط. الخزف الملبد بدرجة حرارة اقل

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أظهر قيم ثابت عزل ε اعلى بالمقارنة مع ارتفاع درجات الحرارة. أظهرت قياسات تحويلات فورير للاشعة تحت الحمراء في كل العينات المحضرة والملبدة حزمة امتصاص في 800 سم $^{-1}$ والتي ترجع إلى حزمة اهتزاز الشد المتناظرة Si–O– Si. وتعزى الحزمة قرب 780 سم$^{-1}$ إلى اهتزاز ذرة البريليوم المتحركة في رباعي الوجوه ذو ذرات الأوكسجين الثابتة الموقع، في حين أن حزم الامتصاص في المنطقة 400–1500 سم $^{-1}$ تعود لاهتزاز الانحناء والشد لآصرة الـ Be –O .

1. Introduction

Porcelain has been used as an electrical insulating material due to its characteristic properties high mechanical and dielectric strength, and good corrosion resistance. Two types of porcelain insulators are mostly used: silica and alumina porcelains [1]. Since long time, Porcelain refers to a wide range of ceramic products baked at high temperatures to achieve vitreous, or glassy, qualities such as translucence and low porosity [2]. Porcelain was first made by the Chinese and the two natural substances used were kaolin and feldspar. The kaolin is also known as china clay, which is white, free of impurities and melts at very high temperatures, while feldspar is mineral forming glassy cement and binding the vessel permanently [3].

The properties of porcelain are greatly varying from a source to another; however, many variations are not critical in resin composites. Silicate materials, such as feldspar and kaolin, have alternating structure of the oxygen and silicon atoms in covalent bonding forming tetrahedral units. Several processes, such as the destruction of dehydroxylation, ceramic lattice, destruction of the network, partial melting to form new phases, occur on silicate heating layer [4].

Amongst known oxides, beryllium oxide (BeO) combines good electrical insulator with high thermal conductivity. It is one of the most chemically stable oxides, resisting both carbon reduction and molten metal attack at high temperatures. It also has high electrical resistivity and low density [5-7]. Also, it has low dielectric constant of 6.7 and low loss index of 0.0012 at 1 MHz, which mean the increases in losses index with increasing of applied frequency. Furthermore, BeO is inherently stable, as an oxide ceramic, in oxygen and moisture-containing environments [8-10]. The most common and economically important system is the $BeO-Al_2O-SiO_2-H_2O$ (BASH) [11].

The layer stacking of kaolinite is controlled by repulsion between highly charged Si and Al cations, which tend to avoid superposition. Unfortunately these functional groups are equally present in several other aluminosilicates. The Si-O, Al-O and OH play functional roles in identification and characterization for chemical composition of a kaolinite unit cell [12, 13].

2. Experimental Part

The starting materials used to prepare the basic porcelain were included 32 wt% feldspar, 24 wt% quartz, and 44 wt% clay. After selecting raw materials, the desired amounts were weighed then crushed and mixed together using hand-pestle. For fine grinding, ball mill for 1 hour consisting of rotating cylinder partially filled with spherical ceramic grinding media was used The particles were reduced to the desired size by sieving mesh equal and less to 53μm. Beryllium oxide was introduced at four different amounts; 1, 3, 5 and 7wt%. A powder metallurgy (PM) process was used to products 2g of compacted specimen in definite shape and size (as a disk shape with 1.5 cm in diameter) using uni-axial. These compacted powders are called "green compacts". Experimentally, the green density of the compact powder was determined using weight / volume ratio. Electronic balance with 5 mg accuracy was used to measure the weight of the elemental powders and also the green compact. The volume was computed by taking dimensions using a digital vernier caliper with an accuracy of 0.01mm.

The prepared samples were introduced in a furnace at sintering temperatures of 1100°C, 1300°C and 1450°C for 2h. Then the basic and different BeO content porcelain samples were leave in furnace till it reaches to room temperature at ambient conditions. During the firing process, a variety of reactions take place, such as, chemical water evolves (at 100 to 200°C), carbonates and sulfates begin to decompose (at 400 to 700°C) lead gases to be produced and escape from the sample. On further heating, some of the minerals break down into other phases, and the present fluxes (feldspar and flint) react with the decomposing minerals to form liquid glasses at 700 to 1100°C. These glass phases are one of causes for shrinking and bonding the grains. When the samples are cooled which causes the liquid glass to solidify, thereby forming a strong bond between the remaining crystalline grains, and finally, the porcelain is completed.

Bulk density (B.D) measurement was determined according to ASTM C373-88. The measurement procedure is outlined by the following steps:

Firstly, the dry weight (g) of the specimens in air (W_d) is determined, then the samples were placed in a baker filled with distilled water, then boiled for 2h. After that, the samples are kept immersed in water for 12h. Then the weight W_i is determined while the samples are suspended in water. Finally, each sample is blotted lightly with moistened smooth cotton cloth to remove excess water from the surface, and the saturated weight, W_s is recorded in air.

Bulk density were calculated using equation (1):

$$Bulk\ Density\ \rho_b(g/cm^3) = \frac{Weight\ in\ air}{Bulk\ Volume} = \frac{W_d}{W_i - W_s} \tag{1}$$

The compressive strength for prepared samples with 1.5 cm in diameter was calculating using equation (2). PHYWE test machine (model 1757793, Japan) has been used in its compression mode. The crosshead speed has been fixed to 0.07mm/sec.

$$\sigma_c = \frac{F}{A} \tag{2}$$

Where, F is the applied force in N, and A is the cross-section area.

The AC conductivity for the samples containing 3 and 7wt. % BeO were chosen for this measurement to clarify the ratios difference.

Experimentally, Loss index (Loss factor ε″), , Loss tangent (dissipation factor tanδ) have been measured directly and the results are read on the monitor and recorded on computer data sheet file. The frequency range extends from 40Hz to 10MHz. Dielectric constant (ε′) was obtained utilizing equation (3)

$$\varepsilon' = \frac{t_a \times c_p}{\pi \times \left(\frac{d}{2}\right) \times \varepsilon_\circ} = \frac{t_a \times c_p}{A \times \varepsilon_\circ} \tag{3}$$

$$\tan\delta = \frac{\varepsilon''}{\varepsilon'} \tag{4}$$

Where:

ε' = Dielectric constant of test material.
C_p = Equivalent parallel capacitance (F).
t_a = Average thickness of test material(m).
A = Area of guarded electrode (m^2).
d = Diameter of guarded electrode=5×10^{-3} (m)
$\varepsilon_\circ$ = Permittivity of space =8.854×10^{-12} (F/m)

For AC measurements, the device was first calibrated using a Precision Impedance Analyzer type Agilen technologies 42942 A made in U.S.A., standard capacitor was used.

Evaluation of a.c. conductivity for these samples was made using the available measurements of dielectric constant and tan δ at a given frequency:

$$\sigma_{ac} = \omega \tan\delta \varepsilon_\circ \varepsilon' \tag{5}$$

Where σ_{ac} is the conductivity, $\omega = 2\pi f$ is the angular frequency.

Infrared spectra for these samples were obtained by a Shimadzu FTIR spectrometer in the region of 4000-400 cm^{-1}. In order to continue improving the most common form of ceramic, namely insulator porcelain, the introduction of small amount of BeO into the composition of these ceramic was prepared to study the influence of this addition into the basic porcelain as well as sintering temperture which often believes it make a considerable improvement in the physic-mechanical and electrical resistance of porcelain.

3. Results and Discussion

The variation of BeO content relative to the green density (G.D) was listed in Table-1. The addition of different amount of BeO percentage into basic porcelain leads initially to slowly decrease in G.D. It can be expected that the increasing of BeO content introduces more voids among the particles, which produce a large volume with less dense material. In spite of BeO has higher density than the components of basic porcelain but the acceptance analysis for this behavior is either to the inclusion of voids by compression process, on, The properties and performance of PM parts (which is used in our work) are directly related to density; therefore high green density of powder compacts is an important first step. For powder particles in micron range, pressure and material composition are the dominant factors that affect green density. Particle size and size distribution factors have minor effect on the green density [14].

So, it can conclude that the particle size, shape, distribution, and composition are all of very important influence on green density.

Table 1-The variation of green density according to BeO wt% addition

Wt.% BeO	Density (g/cm^3)
1	1.824
3	1.823
5	1.812
7	1.810

In order to provide clear information about the influence of sintering temperature, 1300 °C and 1450 °C has been chosen for bulk density, compressive strength, AC conductivity and FTIR tests. Also, specific percentages of 3 and 7% BeO were chosen to supply for the same tests.

Table-2 shows the influence of sintering temperature on the bulk density for basic and new-compose porcelain. The basic porcelain has little enhancement in bulk density with increasing temperature, which means that increasing temperature leads to produce close particles , i.e., the volume of material is affected by decreasing linear shrinkage due to the movement of particles into the pores. This leads to densification in ceramic body. At 1450°C, the sintering is significantly improved and the transformation of open-into-closed porosity is expected.

In fact, it is apparent that open porosity decreases with increasing firing temperature due to the formation of a glassy phase that is mainly originated from the feldspar. Increasing temperatures cause both an increase in liquid phase amount and decrease in liquid phase viscosity. Under the surface energy forces created by the fine pores contained in the ceramic body, the liquid phase tends to approach the particles and, therefore, open porosity decreases [15].

Samples containing 3 and 7wt% BeO provide enhancement in bulk density in contrast to basic porcelain for all sintering temperature as well as sample contain 7wt% BeO introduce B.D. great than that contain less addition of BeO, this behavior is very acceptable according to high BeO density. Table-2 clearly revealed this behavior at different sintering and BeO contents.

Table 2-Bulk density as a function of sintering temperature for pure and new porcelain

Temp.	Pure Porcelain	3 wt.% BeO	7wt.% BeO
1100°C	1.879	2.130	2.232
1300°C	1.852	1.830	1.834
1450°C	1.835	1.578	1.493

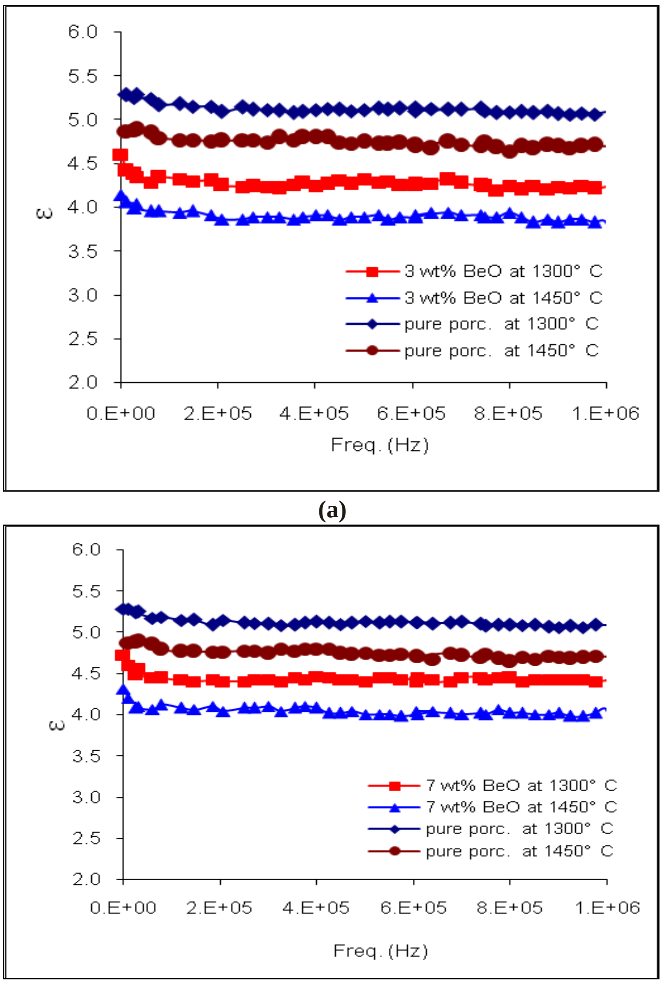
The dependence of compressive strength for basic and addition BeO porcelains at different sintering temperatures is shown in Table-3. It is observed that the basic has higher compressive strength, and increasing temperature leads to increase compressive strength. When the sintering temperature was raised to 1450°C, the molten part of the component starts to participate in the dissolution of the quartz phase and enhance the densification process. As a result, the sintering was improved and products with lower porosity and higher mechanical properties were obtained. This result was in agreement with that obtained from bulk density (dense materials produce higher strength). Samples containing less BeO show highly compressive strength at 1300°C than those sintered at 1450°C. Increasing BeO content and sintering temperature leads to less strength material. This result is reasonable if we know that Mechanical strength is normally lower than alumina, but can reach acceptable levels through control of the fabrication process [16].

Table 3- Compressive strength for pure and 3wt%&7 wt% BeO content at different sintering temperature

Temp.	Pure Porcelain	3 wt.% BeO	7wt.% BeO
1100°C	16.7	15	7.8
1300°C	20	13.22	6
1450°C	26.98	12.91	5.5

The variation of frequency-dependent dielectric constant (έ) for basic porcelain and porcelain containing different BeO contents at different temperatures (1300 and 1450°C) was shown in Figure-1. All samples revealed slightly decrease in dielectric constant at lower applied field 40 Hz to 200 kHz, and then show stable values in spite of increasing applied frequency. This can be explain as we know there are four type of polarization contribute to dielectric constant named as electronic, ionic, dipole orientation and space charge polarization. The net contribution of polarization to the dielectric constant is therefore frequency dependent. The electronic displacement responds rapidly at higher field reversals, no lag of this polarization contribution occurs up to 10^{17} Hz. Ions, which are larger than electrons are less mobile and have a less rapid response. The polarization effect of ionic displacement decreases at 10^{13}Hz. Dipole orientation and space charge polarization have slower frequency responses. So, the slightly decrease in έ behavior of specimens means that the electronic and ionic polarization contribution were eliminated and only the Dipole orientation and space charge polarization remain effective.

A comparison between the basic and new-composed porcelains revealed high value of dielectric constant for the basic porcelain with respect to those containing 3 and 7 wt.% BeO. As well as, porcelain with a lower sintering temperature (1300°C) produce higher έ values in contrast with higher temperature (1450°C). This result was also recorded for the porcelain samples containing 3 and 7 wt.% BeO. Effect of increasing temperature on reducing έ for basic porcelain can attributed to the contribution of all type of polarization in έ at lower temperature, increasing sintering temperature may be produce structure with less arrangement as well as less contribution and interaction with applied frequency. The composition of the basic porcelain, which has high quantities of SiO_2 (24wt% knowing as quartz), introduces material with high dielectric constant. Increasing sintering temperature leads to destroy Si-O-Si bonds and then formation of new composition with lower έ values.

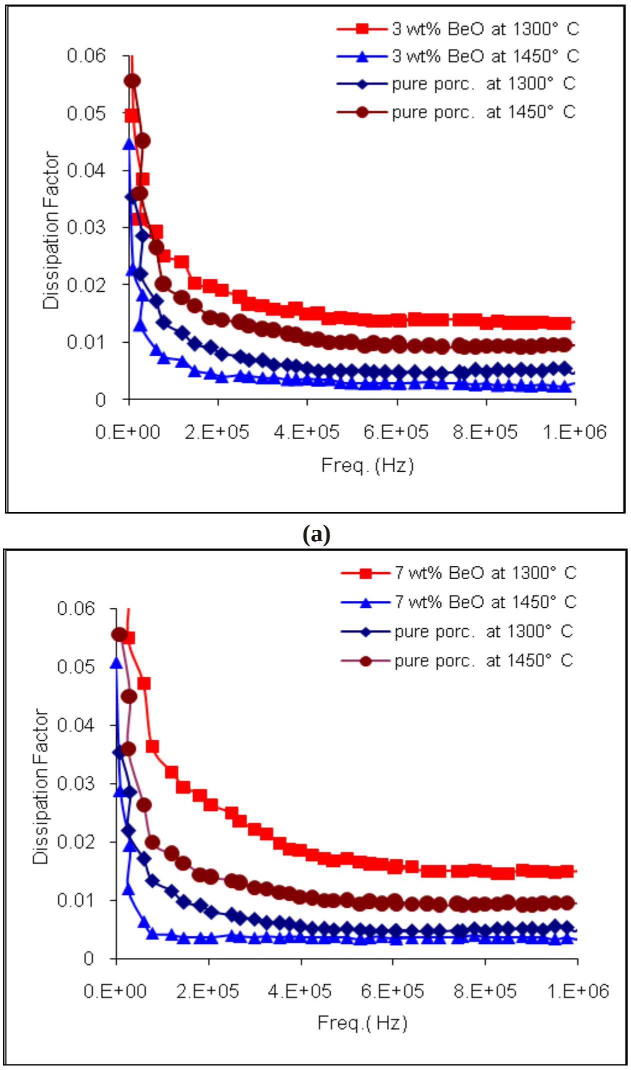


(a)

(b)

Figure 1- The dependence of dielectric constant for pure porcelain and addition (a) 3wt.% and (b) 7wt.% of BeO to it at different sintering treatment.

Figure-2 shows the relation between the dielectric losses or dissipation factor (ε″) and applied frequency for basic porcelain and porcelains containing 3 and 7 wt. % BeO. Increasing frequency leads to pronounced decreasing in ε″ within frequency range 40 Hz to 300 kHz. As well, increasing frequency for prepared samples shows linearly stable values of dielectric losses. Sample containing 7 wt.% BeO and subjected to 1300°C revealed higher dielectric losses than that containing 3 wt.% BeO at the same conditions.



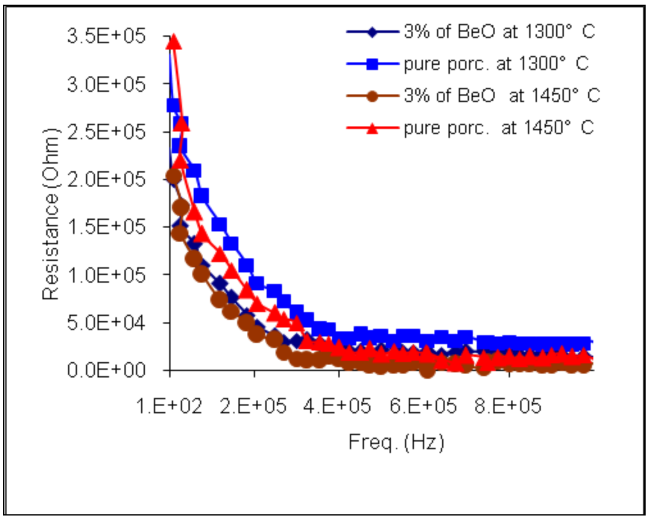
(a)

(b)

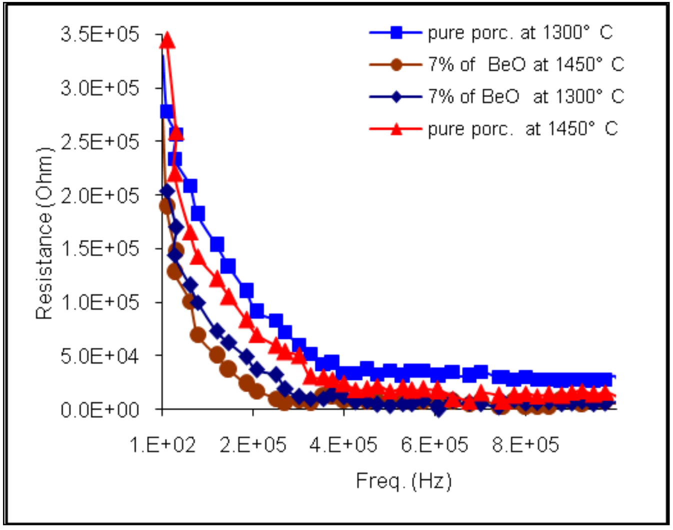
Figure 2- The dependence of dielectric losses for pure porcelain and addition (a) 3wt.% and (b)7wt.% of BeO to it at different sintering treatment

Figure-3 shows the resistance data taken for prepared samples. At the first sight, it can see that all samples have the same behavior, it have higher resistance at low frequency then the increasing of applied frequency leads to decreases in resistance. This result is very acceptable because an increasing in applied freq. means increasing of supplied energy subsequently movement of mass particles and inhibitor of currier movement.

Also, figure show that the basic porcelains have greater resistance than new one, as well as, sample with low sintering temperature provides higher resistance than higher sintering temperature. These results are similar for BeO addition porcelain. Porcelain body is composed of phases such as mullite, quartz and glass. Its electrical properties are dependent on the properties of each of these phases, both at room and at elevated temperatures. Besides, microstructural characteristics, such as crystal size and size-distribution also contribute to the electrical properties of the body because they influence the passage of current through the body [17].



(a)



(b)

Figure 3- The variation of resistance for pure porcelain and addition (a) 3wt.% and (b)7wt.% of BeO to it at different sintering treatment.

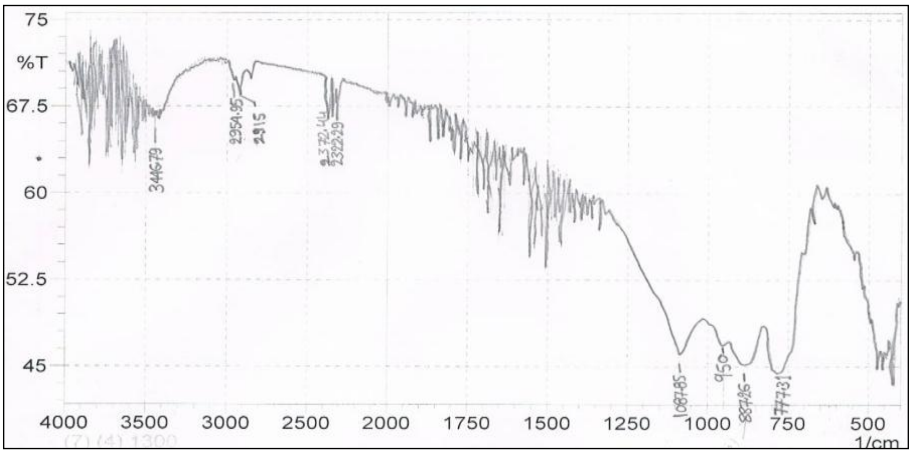
The general observations on FTIR show that there was no interference among peaks occurring at the highest frequencies where stretching OH vibrations occur. However, the interference of peaks was observed at lower frequencies at 430 cm^{-1} of bending vibration. The main functional groups within this range of wavelength are SiO and Al-OH. Muscovite and possibly quartz interference could be observed at 1029 cm^{-1}. The Al-OH absorption peak was identified at 913, 915 and 911 cm^{-1} for different kaolin sources, whereas the theoretical peak of kaolinite is at 912 cm^{-1}. In all prepared samples, the absorption at 800 cm^{-1}, due to Si-O-Si symmetrical stretching vibration, is generally accepted as the most suitable band for analytical peak measurement and commonly used for silica determination. In some cases, the 780 cm^{-1} band can be complemented to 800 cm^{-1} [18, 19]. The FTIR spectra of the sintered porcelain samples containing 3 and 7 wt.% BeO are shown in Figure-4. These spectra confirmed that the calcination at any conditions in these experiments produce dehydroxylation in the samples, i.e., elimination of the OH stretching from Si-OH and Al-Al-OH bond at 778 cm^{-1}.

In the region of OH stretching vibration of water, a band at 3446 cm^{-1} is observed which is typical of water-forming hydrogen bond and remained in all samples after the calcination. A weak band, assigned to the H-O-H bending vibration mode of H_2O molecules, can be seen at 1635-1623 cm^{-1} but cannot be seen in this case because of a noise distortion originated in the device. The OH stretching bands of kaolinite at 3698 and 3621 cm^{-1} were eliminated in all samples after the calcination. Absorption band at 533 cm^{-1} related to Al at octahedral sheet (Si-O-Al bending) of kaolin was not observed after calcination at 1300 and 1450°C. This can be explained as the octahedral sheet changes into tetrahedral sheet of metakaolin [20].

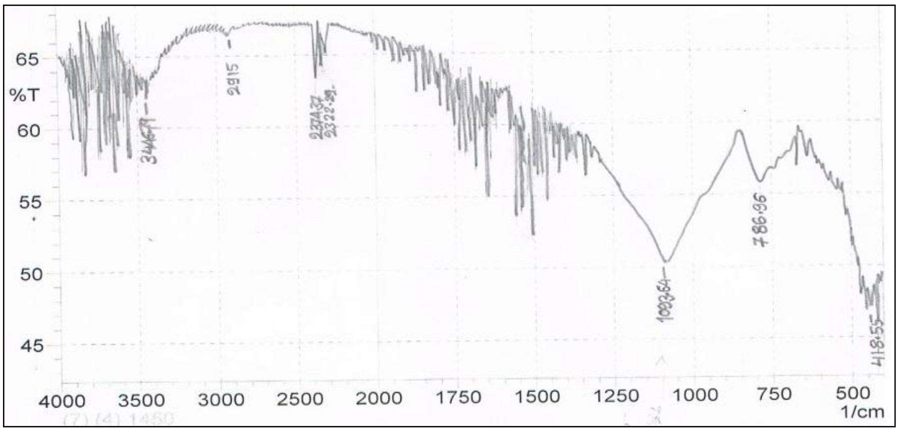
The Si-O vibration band of kaolinite at 1100 cm^{-1} was used as a reference. Raman band at 1076-1072 cm^{-1} band has been attributed to the Si-O bond, but it refers to the Be-O bond in other studies according to references listed by experimental results [21]. In all synthetic beryls, the position of this band was around 1067-1068 cm^{-1} and shifted to higher frequencies in natural samples than in synthetic ones. The assignment of this band to Si-O vibration was preferred rather than to Be-O vibrations.

For crystals containing the BeO_4 group and having a large number of degrees of freedom in the unit cell, the infrared spectrum is often interpreted as arising principally from the vibrations of the tetrahedral BeO_4, group. A tetrahedral unit exhibits a non-degenerate vibration of frequency υ_1, a doubly degenerate vibration of frequency υ_2, and two triply degenerate vibrations at υ_3 and υ_4 [22]. Generally, only υ_3 and υ_4 are infrared active, but distortions of the tetrahedral symmetry and couplings with other parts of the crystal can result in υ_1 and υ_2 appearing in the infrared and can break the degeneracy of the modes υ_2, and υ_4. A simple model is introduced for the band near 780 cm^{-1} which occurs in the infrared spectra of several minerals containing BeO. This band is attributed due to the vibration of a beryllium atom moving in a tetrahedron of fixed oxygen atoms. The frequency of this vibration band (ν_{Be}) would be triply degenerated for a perfect tetrahedron and the degeneracy is broken by slight distortions of the tetrahedron. Using the infrared spectrum reported by Ehlmanna and Mitchell [23], a band composed of peaks at 875, 780, 750 and 720 cm^{-1} was assigned to the vibration of the Be in a tetrahedron of fixed OH groups. The average frequency, 781 cm^{-1}, was taken as ν_{Be}. A band in the infrared spectrum of with peaks at 750, 775 and 820 cm^{-I} was tentatively assigned to BeO. The average of these peaks, 782cm^{-1}, is close to the assignment of 780 cm^{-1} made for υ_3 [24].

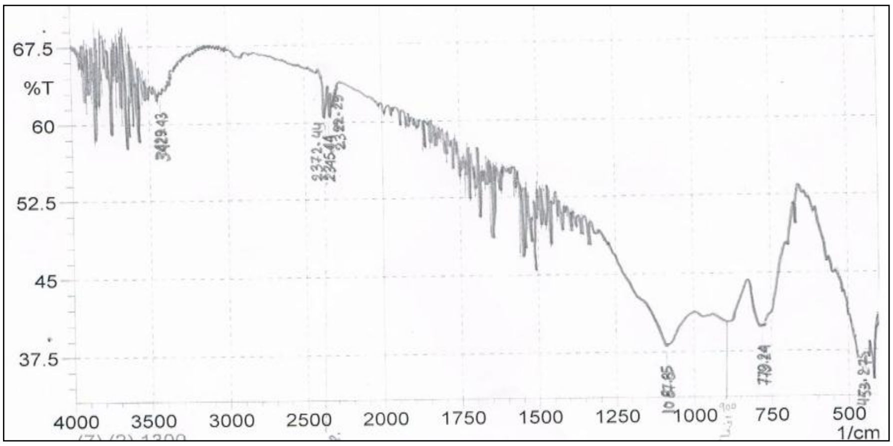
The presence of water during crystallization course exhibits a broad absorption band in the region 3200-3700 cm^{-1} and a somewhat sharper peak at 1625 cm^{-1}, while the absorption bands in the region 400-1500 cm^{-1} are due to the Be-O stretching and bending vibrations. The effect of heat treatment for 3 hours at temperatures of 1300 and 1450°C on the infrared absorption spectra of new feldspar composition is shown in Figure-4. The characteristic bands of SiO_4 tetrahedral units are observed at 400-485 cm^{-1} due to Si-O-Si bending mode [20], and at 675-775 cm^{-1} due to M-O-Si stretching mode [21].



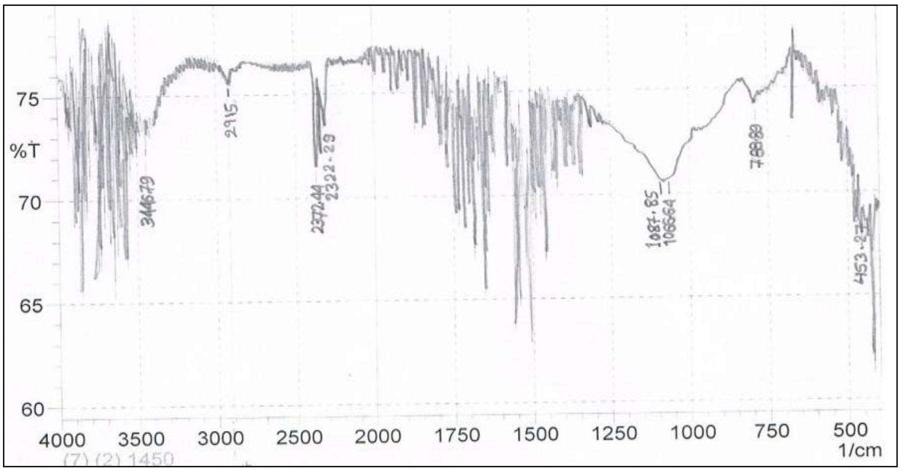
7 wt.% of BeO at 1300°C



7 wt.% of BeO at 1450°C



3 wt.% of BeO at 1300°C



3 wt.% of BeO at 1450°C

Figure 4- FTIR spectra of porcelain samples containing 3 and 7 wt% BeO at different sintering temperature.

4. Conclusion

Increasing sintering temperature of basic and new porcelain enhances the bulk density is expected because the inter-particle voids/pores were progressively filled up with increasing sintering temperature, the volume of the ceramic samples can be reduce with increased sintering temperature. This behavior is also due to the reduced porosity of the sample which lead to increase in the amount of matter in the sample per unit volume. Compressive strength was increased with increasing sintering temperature for basic porcelain whilst it behaves inversely with new porcelain, as sintering and addition content increase , compressive strength were decrease. High value of dielectric constant for the basic porcelain were recorded with respect to those containing 3 and 7 wt.% BeO. As well as, porcelain with a lower sintering temperature (1300°C) produce higher έ values. This result was also recorded for the porcelain samples containing 3 and 7 wt.% BeO. FTIR analysis shows that in all prepared samples, the absorption at 800 cm^{-1}, due to Si-O-Si symmetrical stretching vibration. The Si-O vibration band of kaolinite at 1100 cm^{-1} was seen which is used as a reference.

In all prepared samples, the octahedral sheet has changed into tetrahedral of metakaolin at calcination temperatures of 1300 and 1450°C, which was confirmed by the FTIR spectroscopy. Finally it can conclude that the properties of porcelain can be enhancing by controlling the fractional content of BeO as well as the sintering temperature of the composition.

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