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Influence of Fe_2O_3 content on the dielectric behavior of aluminous porcelain insulators

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Abstract

Due to the increasing availability of substitute materials for electrical porcelain, research is needed to adapt formulations involving these materials to the current economic realities of the industry. This study assessed the effect of iron oxide concentration (0, 1, 2, 3, 5, and 8 wt%) on the dielectric properties of an aluminous porcelain composition commonly employed for electrical insulation based on different values of temperature and frequency. Samples with iron oxide contents of 0, 3, and 5 wt% were analyzed using dilatometry, X-ray diffraction, and scanning electron microscopy to evaluate the thermal, structural, and microstructural changes related to their Fe_2O_3 concentrations. Both the dielectric constant (ε_r) and the loss tangent (tan δ) were measured and evaluated in every sample. Results indicated that the presence of Fe_2O_3 increased the dielectric constant and loss tangent, which could result in an increase in heating by dielectric losses. Fe_2O_3 contents of up to 5 wt% had no significant effect on the performance of these insulators at room temperature (~30 °C) and a high frequency (1 MHz), especially when the hematite phase was completely solubilized in the porcelain phases.

Keywords: D. Porcelain; Iron oxide; Dielectric constant; Loss tangent

1. Introduction

Materials used for electrical insulation perform the basic function of acting as a physical barrier to the passage of electric current between two conductive materials, thus regulating or preventing the transport of current [1]. The selection of an insulating material begins with detailing the specifications for its use, which involve a compromise between the electrical properties inherent in insulation, mechanical strength compatible with the application, and the application's economic viability. The magnitudes of these factors have undoubtedly been responsible for the adequacy and consequent increased use of porcelain as an insulator in electricity distribution lines. Moreover, functional properties have been improved by substituting quartz by alumina [2,3]. The alumina addition increases the electrical resistivity and reduces the loss tangent (tan δ) [4].

These porcelains for electrical insulation should have a high electrical resistivity that minimizes the energy conducted. In addition to electrical resistivity, two other important electrical properties are the dielectric constant (ε_r) and loss tangent. Porcelain should exhibit the smallest possible dielectric constant value (or relative permittivity). In other words, it should not store loads or have species that can be polarized when subjected to an electric field. Of all of the polarization mechanisms usually present in

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porcelains, ionic polarization is the most relevant [1]. The loss tangent represents in a certain way the inefficiency of the polarization process. As with the dielectric constant, the loss tangent must be minimal in high-performance electrical insulators. The reason for the low values of these two properties is due to the combined effect they have on the heating of the insulator. Heating occurs as a result of the conversion of work related to the movement of polarizable species into heat, so the larger the number of species (high ε_r), the greater their mobility with respect to the field direction and the more inefficient the polarization processes (high tan δ), and the greater the rates of thermal energy generation. The problem with heating the insulator is that, as will be discussed later, the functional insulation properties of porcelains are reduced with increasing temperature. Therefore, a potential exists for insulation failure as a result of thermal rupture that increases with temperature increasing, due to atoms inside the dielectric are in a higher energy state to ionize together, developing a progressive effect of excitation [5].

Recent studies have analyzed the behavior of the dielectric constant and loss tangent of siliceous porcelain in relation to the concentration of the existing phases. Chaudhuri and Sarkar [6] have evaluated whether the presence of mullite can influence the dielectric properties of porcelain using mineralizers in porcelain formulations to increase their concentrations. But the role of mineralizer oxides should be studied. In that work, several oxides of transition metals were used and it was considered that they did not influence in other ways except in the formed crystalline phases concentration, i.e. mullite crystals nucleated from iron oxide addition were dielectrically identical to those grown by the addition of vanadium oxide, for example. The dielectric constant exhibits a parabolic trend as a function of mullite concentration. The researchers observed an initial increase in the dielectric constant with the increase in mullite content, up to a maximum ε_r equal to approximately 3 with 40 wt% mullite. After this maximum, the dielectric constant progressively decreases. The effect on the loss tangent is such that it increases with higher concentrations of mullite and increasing size of the mullite crystals. The latter involves the movement of carriers across the boundaries of the mullite crystals [6]. In relation to the presence of quartz, after reaching a maximum value of ε_r equal to 2.9 (5 wt% SiO_2), there is a progressive decrease with the increase in the concentration of this phase, identified as a nonlinear trend.

A study by Chaudhuri and Sarkar [6] has shown how the concentration of the glassy phase in porcelain can affect its insulation properties. The dielectric constant and loss tangents exhibit a parabolic profile, going through a minimum and then increasing with the increased concentration of the glass phase.

Taking into account the complexity of the logarithmic dependence of dielectric variables shown in the Chaudhuri and Sarkar [6] work and the difficulty of finding related works, complementary investigations to define a general effect of the relationship between microstructure and dielectric response are needed. By considering the effect of mullite, for example, we cannot state its influence before defining the concentration.

When interpreting the values of the dielectric constant and loss tangent, the results cannot be extrapolated to different measurement conditions from those used during sampling. There are significant changes in the values of these properties with changing temperature and frequency.

The goal of this study was to evaluate the effect of iron oxide content on the dielectric constant and loss tangent in aluminous porcelains at different frequencies and temperatures. Simply put, the intent of the present study was to illustrate how harmful the presence of iron oxide is in the raw materials that make up a porcelain composition in terms of its electrical properties. Furthermore, the study assessed whether there was a maximum Fe_2O_3 concentration that would result in a significant increase in the porcelain's dielectric characteristics and hinder the use of porcelain as an electrical insulator.

2. Material and methods

Samples were prepared based on a triaxial porcelain formulation containing feldspar, kaolin, and alumina. Table 1 shows the chemical analysis of the raw materials and the additive obtained by X-ray fluorescence (Philips PW 2400, Eindhoven, Netherlands). The formulation of the test specimens was prepared considering the addition of iron oxide (1–8 wt%) as a substitute for the corresponding proportion of the other components of the base composition, Cb (30 wt% kaolin, 30 wt% feldspar, and

Table 1
Chemical analysis of the raw materials used and the additive (hematite).

Raw material	**Concentration (wt %)**											
	Al_2O_3	SiO_2	Fe_2O_3	CaO	Na_2O	K_2O	MnO	MgO	TiO_2	P_2O_5	Others	L.o.I.[a]
Feldspar	17.32	71.32	0.20	0.60	5.50	3.50	0.02	0.03	0.01	0.50	0.00	1.00
Kaolin	33.93	53.16	0.73	0.22	0.03	0.36	0.00	0.06	0.25	0.05	0.07	11.15
Alumina	99.59	0.07	0.02	0.02	0.04	0.00	0.00	0.00	0.00	0.00	0.06	0.26
Hematite	0.00	0.00	99.95	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.05	0.00

[a] Loss on ignition.

40 wt% alumina). This method seeks to minimize the effects of the removal of initial constituents. Table 2 shows the code for the formulations applied and the corresponding levels of addition. The molar ratio of silica to alumina was constant in all of the formulations ($SiO_2/Al_2O_3=0.68$). Table 3 shows the chemical compositions (wt%) of the oxides in the formulations applied. Each formulation was then ground in a porcelain mill, dried, and shaped by uniaxial pressing with a compaction pressure of 125 MPa in cylindrical steel molds 10 mm in diameter and 3 mm thick. The thermal behavior of each formulation was determined by dilatometric analysis (BAHR dilatometer Thermoanalyse Gmbh 2000 model DIL801L, Hüllhorst, Germany). The sintering temperatures were 1200 °C for formulations Cb.F5 and Cb.F8 and 1300 °C for formulations Cb.F0, Cb.F1, Cb.F2, and Cb.F3, using heating rates of 10 $^{\circ}C\,min^{-1}$ and then maintaining for 2 h at room temperature in all cases. The microstructures of the ceramic bodies were determined by scanning electron microscopy (SEM; HITACHI S-4100, Tokyo, Japan). For this procedure, samples of each formulation after heat treatment were prepared in the form of pellets whose surfaces were polished and chemically attacked with an aqueous solution of hydrofluoric acid (10% v/v) for 60 s. Identification of the crystalline phases was performed by X-ray diffraction (Rigaku Geigerflex, Tokyo, Japan) using a copper tube (Cu Kα radiation) in a 2θ angle interval from 10° to 80° with a 0.02°-step. To perform the electrical measurements, the samples were polished to obtain parallel surfaces, and palladium/gold electrodes measuring 5.5 mm in diameter were placed on them. The electrical properties were analyzed at frequencies between 0.1 and 1 MHz and between temperatures of 30 and 200 °C according to the electrometer method, using an HP 4284A Precision LCR Meter (Hewlett-Packard Company, Santa Clara, USA).

Table 2
Formulations applied.

Code	Description
Cb.F0	Base composition (Cb) without addition of Fe_2O_3
Cb.F1	Base composition (Cb)+addition of 1 wt% Fe_2O_3
Cb.F2	Base composition (Cb)+addition of 2 wt% Fe_2O_3
Cb.F3	Base composition (Cb)+addition of 3 wt% Fe_2O_3
Cb.F5	Base composition (Cb)+addition of 5 wt% Fe_2O_3
Cb.F8	Base composition (Cb)+addition of 8 wt% Fe_2O_3

3. Results and discussion

Fig. 1 shows the linear shrinkage (LS) curves as a function of temperature, the corresponding first derivative curves of LS as a function of temperature, and the microstructures acquired by SEM for compositions Cb.F0, Cb.F3, and Cb.F8. The figure shows that sintering began at approximately 1085 °C for these compositions. However, the addition of iron oxide progressively reduced the temperature of the maximum linear shrinkage rate. Compared to composition Cb.F0, composition Cb.F8 showed a reduction of approximately 100 °C. This phenomenon was explained by the reduction in the temperature of formation of the first liquid and the iron oxide acting to reduce the viscosity and surface tension of the liquid formed, providing conditions for the rearrangement of the particles and increased densification at lower temperatures. It should be noted that the presence of iron oxide had a positive effect on densification. However, when the maximum densification temperature is exceeded, the iron oxide can result in the emergence of open and interconnected pores in the porcelain due to the increased pressure of internally formed gases and the high fluidity of the Fe_2O_3-rich liquid formed. This sequence of events can cause the deformation and even expansion of the pieces, especially for composition Cb.F3.

The X-ray diffractograms of compositions Cb.F0 and Cb.F3 (sintered at 1300 °C for 2 h) and Cb.F8 (sintered at 1200 °C for 2 h), shown in Fig. 2, illustrate the effect of the iron oxide concentration on the progression of the porcelain composition's crystalline phases. Fig. 2 shows the disappearance of residual quartz peaks in composition Cb.F3 compared to Cb.F0 and the increase in the relative heights of the peaks related to mullite. These findings demonstrated the effect of iron oxide as a mineralizer. The presence of hematite favored the consumption of residual quartz in the formation of new mullite crystals, observed in the micrographs, in which the mullite crystals amount in the compositions Cb.F3 and Cb.F8 is increased, besides modifying the crystals morphology. As discussed previously, iron oxide accelerates the process of liquid phase

Table 3
Chemical compositions of the formulations applied.

Formulation	Concentration (wt%)									
	Al_2O_3	SiO_2	Fe_2O_3	CaO	Na_2O	K_2O	MnO	MgO	TiO_2	P_2O_5
Cb.F0	57.38	38.84	0.29	0.26	1.74	1.20	0.01	0.03	0.08	0.17
Cb.F1	56.78	38.44	1.33	0.26	1.72	1.19	0.01	0.03	0.08	0.17
Cb.F2	56.19	38.04	2.37	0.26	1.70	1.18	0.01	0.03	0.08	0.17
Cb.F3	55.60	37.63	3.39	0.26	1.68	1.17	0.01	0.03	0.08	0.17
Cb.F5	54.41	36.83	5.44	0.25	1.65	1.14	0.01	0.03	0.08	0.16
Cb.F8	52.64	35.63	8.52	0.24	1.59	1.11	0.01	0.03	0.07	0.16

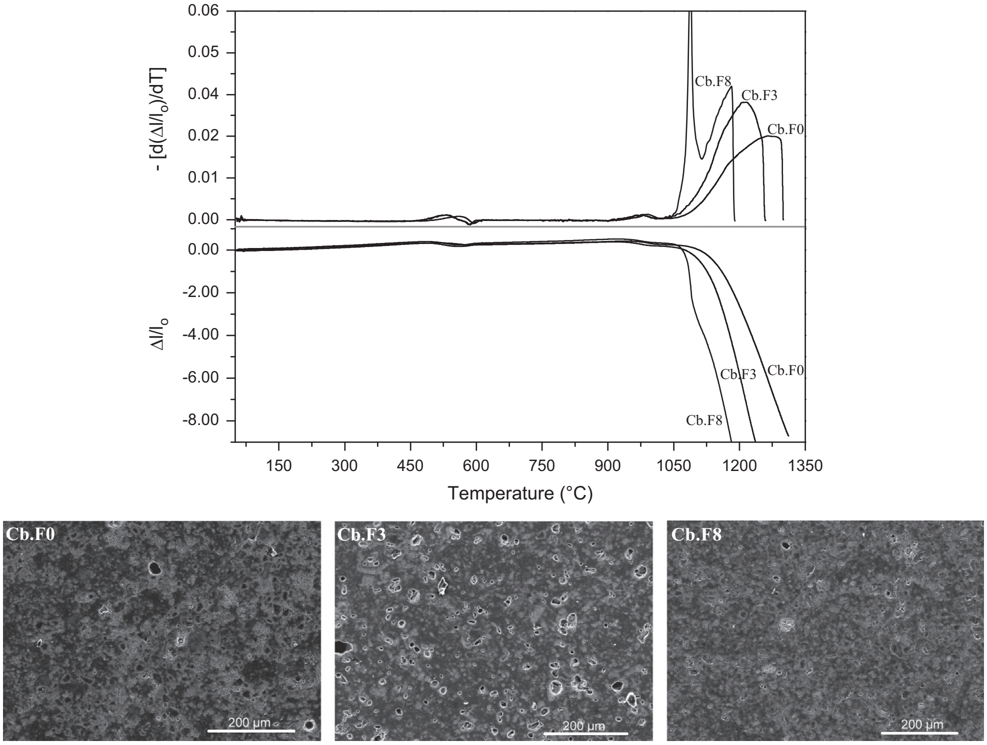

Fig. 1. Linear shrinkage as a function of temperature, first derivative curves, and microstructure of compositions Cb.F0, Cb.F3, and Cb.F8.

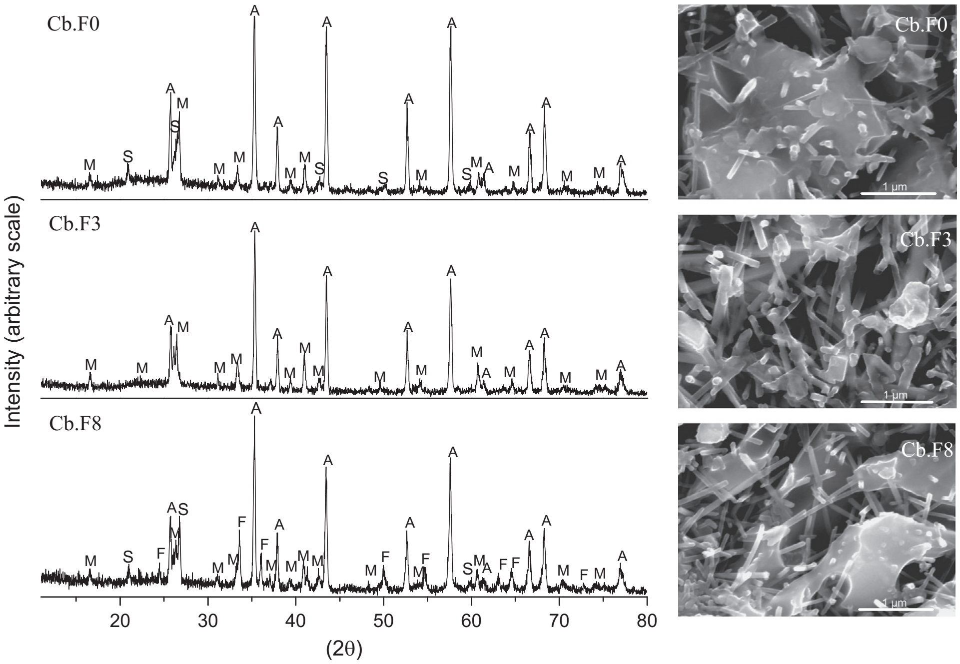

Fig. 2. X-ray diffraction pattern and microstructure of compositions Cb.F0, Cb.F3, and Cb.F8. A–alumina, S—quartz, M–mullite, and F–hematite.

formation, and may also modify the properties of the liquid formed. These aspects provide favorable conditions for diffusion, stimulating the nucleation and growth of mullite crystals induced by the addition of Fe_2O_3. In this case, an acceleration of the mullite formation processes occurs [7].

The presence of hematite was detected in composition Cb.F8. There were apparently no conditions for the complete solubilization of Fe_2O_3 in the porcelain phases of this composition. The action of iron oxide as a catalyst for mullite formation is a function of its concentration and temperature. At a constant temperature, higher concentrations of Fe_2O_3 enable greater formation of mullite crystals, and at a fixed concentration, the mullite formation is facilitated at higher temperatures. In addition, the size and form of mullite crystals are strongly controlled by the liquid fluidity of the matrix where the mullite crystals precipitate and grow. Those phenomena depend on the temperature and composition [8,9].

As shown by Chaudhuri and Sarkar [6], the effect of mullite may be positive or negative due to its parabolic behavior. Although iron oxide induces the formation of larger quantities of mullite, it has been observed that Fe_2O_3, as an additional phase, exerts an effect more pronounced than its mineralizer effect.

The dielectric constant is dependent on the extrinsic variables frequency and temperature, and also has an intimate relationship with the constituent phases [10]. Thus, these variables were considered when preparing a three-dimensional graph, the response surface of which was employed to understand their interrelation. Fig. 3 shows the variation in the dielectric constant as a function of frequency and Fe_2O_3 concentration at constant temperatures of 30 and 200 °C.

At 30 °C, the dielectric constant had an almost linear dependence on Fe_2O_3 concentration. Consequently, low dielectric constant values are observed, around 8.5, in the region between 1 and 2 wt % Fe_2O_3 concentration and 2×10^5–4×10^5 Hz. The two-dimensional contour map indicates the optimum values (minimum values) of the dielectric constant. At 200 °C, the dependence of the dielectric constant on these variables became more marked. Dielectric constant shows a parabolic profile related to Fe_2O_3 concentration. Moreover, values are higher than for those obtained at 30 °C. Considering the response surface and contour map at 200 °C, it was observed that the dielectric constant displayed a more accentuated decrease at higher frequencies, whereas this reduction was less sensitive to higher Fe_2O_3 concentrations. Dielectric constant values around 11 were obtained in the region between 2 and 3 wt% of Fe_2O_3 and 9×10^5–10×10^5 Hz.

Fig. 4 shows the effects of temperature and iron oxide concentration on the dielectric constant at 0.1 and 1 MHz. In this figure, the dielectric constant exhibits a parabolic trend in relation to the concentration of Fe_2O_3 and an exponential trend in relation to temperature. At near-room temperatures and low Fe_2O_3 concentrations, the dielectric constant was virtually independent of frequency. However, at high temperatures and Fe_2O_3 concentrations, the dielectric constant decreased with increasing frequency. At the same dielectric constant value, the validity field of ε_r values was greater at the frequency of 1 MHz than at 0.1 MHz. Thus, the increase in frequency resulted in an increase in the low dielectric constant values, even when the porcelain was exposed to high temperatures or displayed a high Fe_2O_3 concentration. At the maximum values of Fe_2O_3 addition (8 wt%) and temperature (200 °C), the maximum value of the dielectric constant was 18 at a frequency of 0.1 MHz and 15 at 1 MHz. ε_r varied from 8.7 to 9.5 in the temperature range of 30–80 °C and Fe_2O_3 concentrations between 2 and 3 wt%, regardless of the frequency.

Independent of the absolute values, all of the compositions (Cb.F0–Cb.F8) showed similar behavior. In general, the dielectric constant decreased with increasing frequency and increased with increasing temperature. Moreover, the

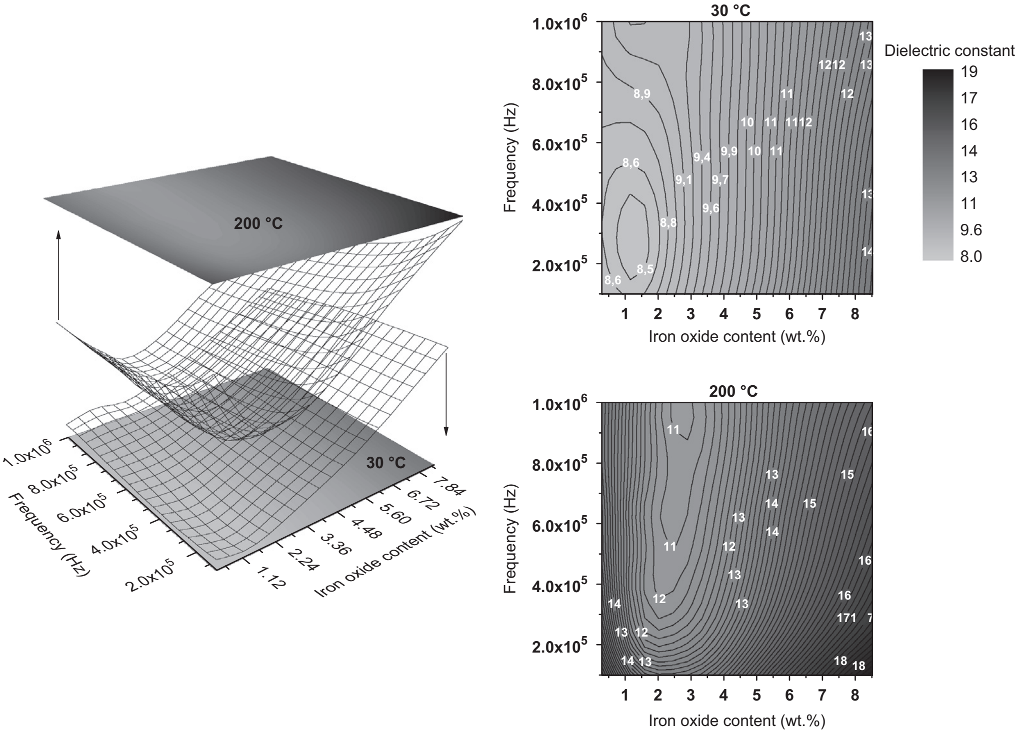

Fig. 3. Influence of frequency and iron oxide content on the dielectric constant at 30 and 200 °C.

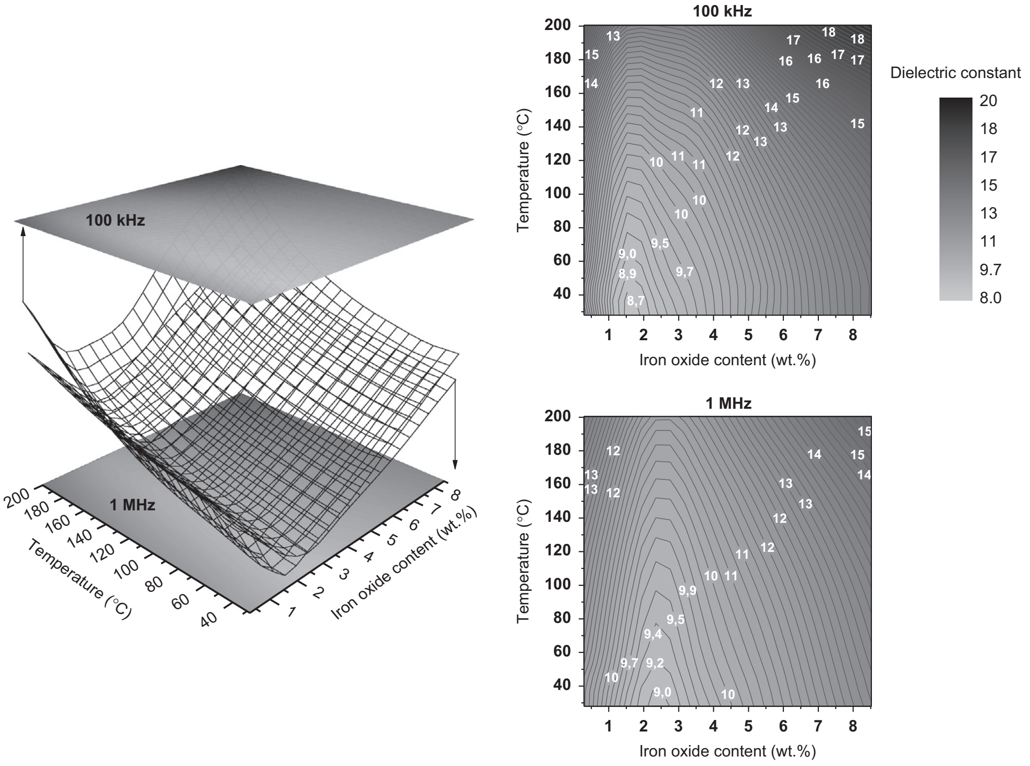

Fig. 4. Influence of temperature and iron oxide content on the dielectric constant at 0.1 and 1 MHz.

addition of Fe_2O_3 up to a concentration of 2.5 wt% reduced the dielectric constant and then increased it again even for low concentrations of Fe_2O_3. This increase in the dielectric constant with temperature was due to the increased number of species susceptible to polarization per volume unit [11]. Temperature increases also intensified the dependence of ε_r on frequency, a characteristic phenomenon of the glass phase relative to ion jump polarization (Na^+ and K^+) [12]. At high temperatures, these aluminous porcelains are exposed to conditions with increased ionization capacity. At high frequencies (1 MHz), there is a reduction in the dielectric constant due to the inability of the less mobile species to keep up with the electric field (AC) and be sufficiently displaced to measurably contribute to the total polarization.

These effects were exploited as a result of the influence of Fe_2O_3 on the development of the different phases in the studied porcelains. A qualitative increase in the dielectric constant clearly depended on the quantity of non-solubilized hematite and its mineralizer effect. According to the X-ray diffractograms (Fig. 2), the composition displaying the highest dielectric constant was that which included hematite as an additional phase. In addition to this, minimum values were observed around 3 wt % Fe_2O_3, a region in which all quartz has been consumed. So, the existence of Fe_2O_3 in content capable of being supported in solid solution causes a beneficial effect since it act in the quartz dissolution. Any change caused by the increasing mullite concentration or related to the glassy phase became irrelevant when hematite was in excess or insoluble in the crystalline phases. This precipitated phase added new mechanisms that increased the possibility of polarization by introducing more mobile species at high frequencies that were activated at low temperatures, significantly reducing the intrinsic inertia of the hematite-free aluminous porcelain. In this case, polarization occurred at the interfaces of the existing phases because of differences in conductivity when there were multiple phases.

When all of the phases displayed high resistivity, polarization occurred only at very low frequencies, but when only one of the phases exhibited low resistivity, in this case hematite, the relative response to polarization extended to higher frequencies, as in aluminous porcelains with hematite as a dispersed phase.

Regarding the loss tangent, Fig. 5 shows its dependence on temperature and Fe_2O_3 concentration for three frequencies. At 30 °C, tan δ presents negligible variation with frequency for the compositions Cb.F0–Cb.F5 (tan $\delta < 0.04$) up to temperatures of about 90 °C. However the frequency dependence is increased with the content of Fe_2O_3 increasing. For composition Cb.F8, tan δ was equal to 0.03 at 0.1 MHz and increased to 0.18 at 1 MHz. It can be clearly seen that there should be losses associated to other mechanisms related to hematite and not evaluated in this work. From composition Cb.F5, it can be seen that the tendency of reducing the loss tangent with increasing frequency is opposite to that shown by the lower Fe_2O_3 contents, i.e. losses are greater for higher frequencies. Basically, the mechanism related to hematite has a relaxation frequency closer to 1 MHz, making

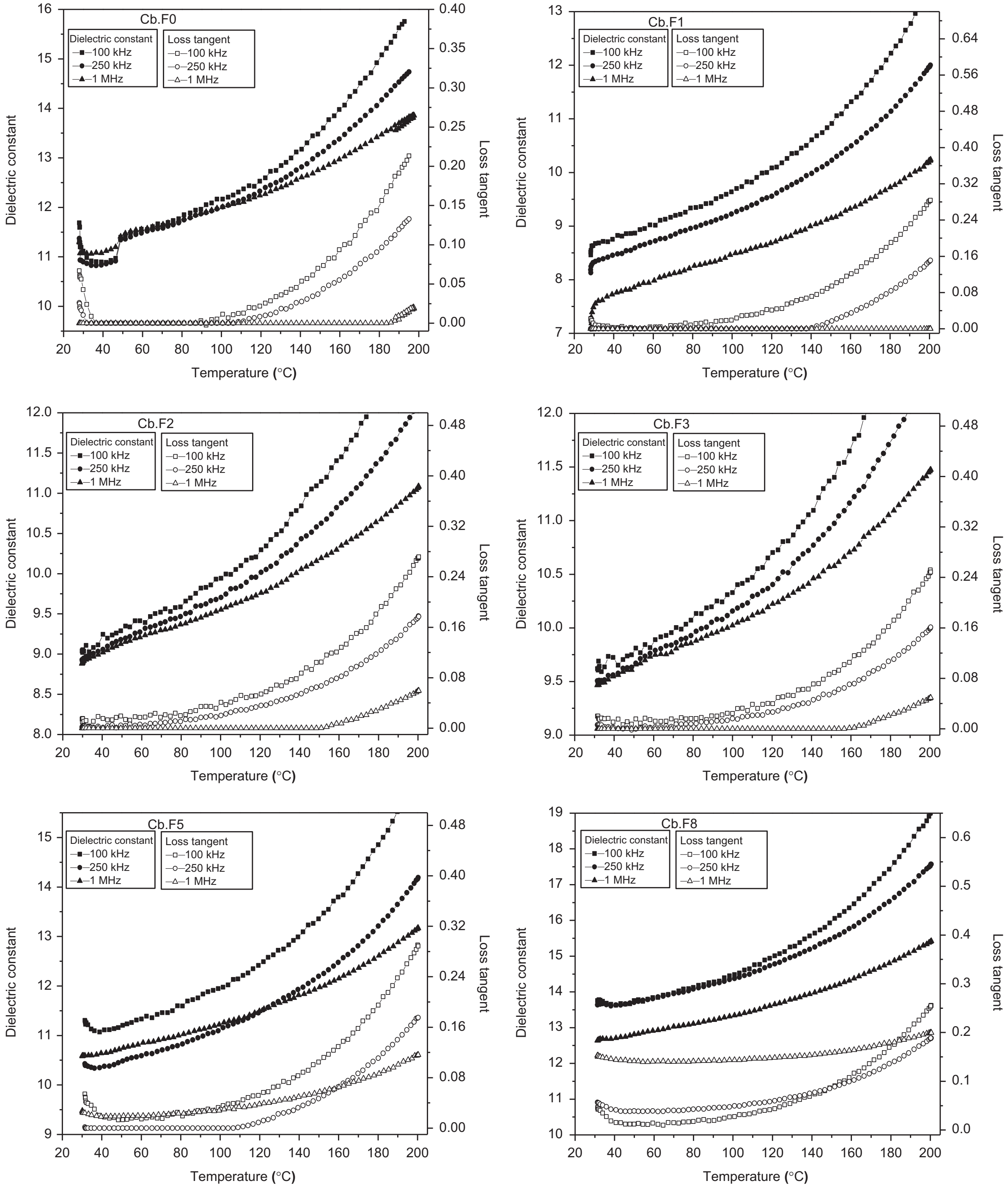

Fig. 5. Dielectric constant and loss tangent versus temperature in the investigated compositions for frequencies of 0.1, 0.25, and 1 MHz.

the tan δ values of porcelains with high hematite concentration greater at higher frequencies, in an opposite way to that shown by samples with low concentration or complete solubility. When samples were exposed to higher temperatures ($\sim$200 °C), the loss tangent showed almost no variation in relation to the Fe_2O_3 concentration at low frequencies. Taking into account the temperature, the increase in loss tangent is related to conduction losses, caused by increased concentration of space charges and defects in the existing phases.

Both ε_r and tan δ increased with the presence of hematite. The presence of Fe_2O_3 at high levels induced heating of the insulator due to the increase of the product of the dielectric constant and loss tangent ($\varepsilon_r \times \tan\delta$).

So, taking into account conditions such as

– low temperatures, at which the dielectric constant depends almost entirely on the Fe_2O_3 content and the loss tangent is nearly constant, and

- high frequency, which for samples without hematite crystalline phase reduces loss tangent,

would have enabled concentrations of up to 3 wt% of Fe_2O_3 in the porcelain without any significant damage to the insulation characteristics.

4. Conclusions

The presence of iron oxide in aluminous porcelains increased the dielectric constant by inducing mechanisms more amenable to polarization for samples in which the hematite (Fe_2O_3) was found to be an additional phase. When hematite was present in amounts between 1 and 3 wt%, loss tangent showed a minimum. There was an increase in the loss tangent, which rises with temperature and frequency to Fe_2O_3 contents above 3 wt%. Loss tangent can be admitted to be invariant for lower Fe_2O_3 contents. The heating of the insulator due to dielectric loss caused by the presence of $F_{e2}O_3$ may only be a risk factor in the case of insulators to be used under extreme conditions such as high temperature and low frequency.

Compositions containing up to 5 wt% Fe_2O_3 did not show significant variations in their dielectric properties at near-room temperatures. The deleterious effects caused by the presence of Fe_2O_3 were clearly linked to its non-solubilization in the aluminous porcelain phases. The use of raw materials with Fe_2O_3 concentrations higher than that currently used would not yield large changes in the product's insulation properties but would allow for significant economic gains.

In the future, issues whose resolution would minimize the influence of Fe_2O_3 include the conditions for complete solubilization of the existing hematite, with sufficient time and temperature for the formation of its solid solution with the remaining porcelain phases and complete dissolution of the residual quartz.

Acknowledgments

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