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Effect of Air Blast Furnace Slag and γ-Alumina Content on Dielectric Properties and Physical Properties of Porcelain Insulators

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Abstract In this study, the effect of air blast furnace slag (ABFS) and γ-Alumina additions on densification, crystalline phases, microstructure, mechanical and electrical properties were investigated. ABFS was added partially in replacement of γ-Alumina and/or feldspar for preparing electro-porcelain compositions. The presence of slag from 15 up to 30 mass % to the standard mix (γAS0) which fired at 1300°C for 1h led to relatively low bulk density (BD) ranging between (2.21 and 2.32 g/cm^3) as compared with standard mix (γAS0) (BD 2.51 g/cm^3), this may be due to presence of much high fluxing oxides in the fired bodies. The main phases recorded were anorthite, corundum, cristobalite with traces mullite The relevant of electroceramic bodies produced exhibited high values for volume resistivity (VR) (25x10^{11} to 30x10^{11}Ω/cm) and relatively low values in the dielectric strength (DS) (11 to 12.43 kv/mm) as compared with standard mix (γAS0), (VR) (12x10^{11}Ω/cm) and (DS) (15.76 kv/mm), respectively. The present results show that it is possible application of recycled slag after grinding and elimination any iron contamination for the production of low voltage electrical insulators electro-ceramic bodies.

Keywords: *slag, γ-Alumina, densification, crystalline phases, microstructure, mechanical and electrical properties*

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1. Introduction

The porcelain is insulator materials because the electrical resistivity values of porcelain are also in this range which is 10^{12}–10^{14} ohm/cm [1]. The electrical insulators are materials with very low electrical conductivity. Their conductivity values are between 10^{-10} and 10^{-20} $(\Omega\text{-m})^{-1}$ [2] and are widely used in the microelectronic devices, ceramic heaters, heating elements, semiconducting material as well as in power transmission lines [1,3,4]. Porcelain insulator are processed from three types of raw materials, namely plastic materials (clay or kaolin [Al_2 Si_2 $O_5(OH)_4$], give plasticity to the ceramic mixture; flint or quart (SiO_2)/alumina (Al_2O_3), maintains the shape of the formed article during firing; and feldspar [K_x Na_{1_x} $(AlSi_3)O_8$], used as fluxing [5,6,7,8].

Recently, ceramics prepared from industrial waste and have been investigated, such as red mud, coal fly ash, and blast furnace slag [9,10,11,12,13]. Blast furnace slag is considered an inexpensive source of alkaline earth oxides. These alkaline earth silicates reduce shrinkage in comparison with alkali ones. Moreover, the produced silicate phases such as wollastonite, diopside and anorthite during firing form a meshwork of prismatic crystals. These phases are favorable properties for ceramic bodies such as higher mechanical strength, improved thermal shock resistance, low thermal expansion and superior electrical properties to the sintered ceramic bodies [14,15,16]. Therefore, it may be advantageous to replace silica containing raw materials such as flux (feldspar), silica and part of clay by blast furnace slag to produce ceramic recipes with desired properties.

The aim of present work is to investigate the feasibility of producing alumina porcelain insulators from air blast furnace slag (ABFS) and γ-Alumina. The physico-chemical and mechanical properties of the porcelains obtained were studied. X-ray diffraction (XRD) was used to characterize the mineralogical phases and scanning electron microscopy (SEM) to evaluate the microstructure.

2. Experimental Work

2.1. Raw Materials:

Local Egyptian raw materials namely; Tih clay (Sinai), potash feldspar (Aswan), air cooled blast-furnace slag from Egyptian Iron and Steel Co.(EISCO), which is cooled slowly in air, it solidifies into a gray crystalline material and commercial aluminum hydroxide $Al(OH)_3$ from (S D Fine – Chem Limited) (Prod C :37077-K05)

(India origin) were used. The chemical analysis of the raw materials was determined via a computerized (X-ray fluorescence Axios – Advanced Panalytical) are shown in Table 1.

Table 1. Chemical analysis of the raw materials

Constituents/Mass-%	Tieh Clay	Potash Feldspar	Aluminum Hydroxide	Blast furnace slag
L.O.I	13.5	0.8	43.77	0.30
SiO_2	44.80	63.90	0.025	35.47
Al_2O_3	35.13	19.20	54.34	12.33
Fe_2O_3	0.52	0.06	0.12	0.39
TiO_2	3.87	-	-	0.63
CaO	1.58	0.13	0.17	40.88
MgO	0.19	0.06	0.02	4.59
K_2O	-	13.24	0.04	1.11
Na_2O	-	2.22	0.03	2.00
SO_3	-	-	-	2.54
Total	99.59	99.61	98.51	100.24

2.2. Particle Size Distribution

The samples grain size of the used raw materials studied with X-Ray monitored gravity sedimentation (using Sedi Graph III V1.04).The results of grain size distribution of raw materials, Figure 1, show that, blast furnace slag: clay 10%, medium and fine silt 80%, coarse silt 10%, potash feldspar: clay more than 10% and Silt 90%, tih clay :clay more than 19.7% and $Al(OH)_3$: pass through 45 µ sieve.

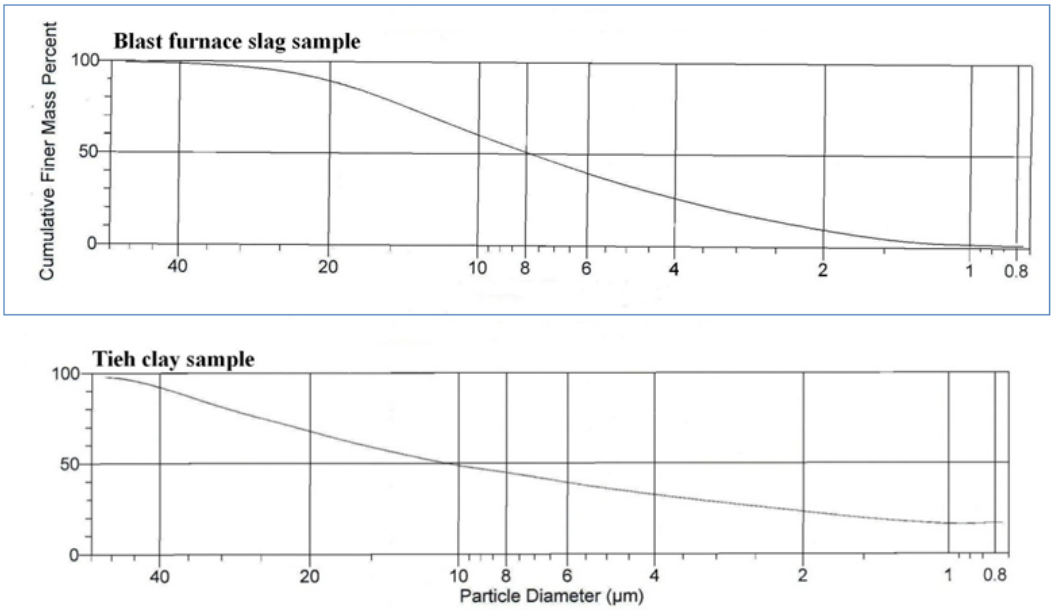

Figure 1. Cumulative fine mass percent curves of raw materials

2.3. Processing

Raw materials were separately ground to pass 250 mesh sieve. The chosen porcelain mix with the following mass percent; 40 Tih clay, γ-Alumina and 30 mass % feldspar was as the base composition for the study. Slag was added partially in replacement of γ-Alumina and/or feldspar. The proposed mixes are listed in Table 2. Slag as powders was used after magnetic separation and grinding with acetone in a ball mill for 3 hrs. The batches were prepared from the fine materials, wet mixed in a porcelain ball mill for one hour to attain proper homogeneity, then dried at 110°C.

Table 2. Batch composition containing γ- Alumina and air blast furnace slag, mass %

Batch symbol	Composition / mass %			
	γ-Alumina	Tieh clay	Potash feldspar	Blast furnace slag
γAS0	30	40	30	-
γAS15	25	40	20	15
γAS20	20	40	20	20
γAS25	15	40	20	25
γAS30	15	40	15	30

Discs of 25 mm diameter and 3 mm thickness were processed by semi-dry pressing under a pressure of 30 KN, dried for 48 h at room temperature, then over night at 110°C in an electrical dryer (Raypa.dod-50, Espanol), then specimens were fired between 1150 to 1350 °C with temperature interval of 20 °C /min and 1 hour soaking time in a muffle furnace(Carpolite,20-900522,England).

The physical properties of the fired discs were followed in terms of linear shrinkage, bulk density, apparent porosity and water absorption were determined according to the ASTM specification Nos. C_{373}-$_{72}$, C_{372-56} and $C_{326–56}$, respectively [17]. Bars with dimension (5.0 x10 x 0.5 cm) were pressed at 200 bar and dried under the same conditions and fired at selected maturing temperatures to measure the modulus of rupture by three point method using (Gabberlli,n.219. Italy). Discs 50 mm diameter and 2-3 mm thickness were fabricated then fired at selected maturing temperature in order to determine electrical properties in terms of volume receptivity and dielectric strength according to the ASTM (D_{257} and D_{149}), respectively [18].

Crystalline phases developed in the fired bodies were determined by XRD technique using (X'Pertpro. Panalytical Cu Kα Target with secondary monochromator, KV=45, Ma=40, Holland). Microstructure was studied by scanning electron microscope of the type Joel TSM. T200 attached with an EDX unit. Selected specimens fired at the optimum conditions were washed, dried and sputtered with a layer of gold of about 200-300 Å thickness for SEM examination.

3. Results and Discussion

3.1. Physical Properties

The effect of partial substitution of γ-Alumina and /or feldspar by various proportion of slag ranging between 15 and 30 mass % on physical properties of the bodies fired between 1150 and 1350 ℃ at 1h are summarized in Table 4 and graphically represented in Figure 2. The results obtained show that the vitrification of all the studied samples occur at 1300 ℃. The vitrification range of all samples is very short. Complete vitrification takes place suddenly and the bodies start to soften suddenly on further increase of temperature. The presence of slag from 15 up to 30 mass % to the standard mix (γAS0) led to relatively low bulk density ranging between (2.21 and 2.32 g/cm^3) and water absorption (zero%) as displayed in Figure 2, as well as reduce in the linear shrinkage (≈11- 14%) Table 4, as compared with standard mix (γAS0) fired at 1300 °C (BD 2.51 g/cm^3, WA zero% and LR≈18%). The low bulk density values of the γ–Alumina insulator bodies containing slag (γAS15, γAS20, γAS25, γAS30) are mainly due to the presence of much high fluxing oxides in the fired bodies. From Table 3 clears that the level of these oxides ranges between (13.50 and 20.12). Also, the presence of high content of alkali (K_2O + Na_2O), and alkaline earth oxides (CaO + MgO) within the fluxing oxides (feldspar and slag) leads to lowering the viscosity of the liquid phase developed at vitrification temperature. This assisted speedier formation of glassy phases and promoted the reaction with clay minerals present at this temperature. Harms [19] also observed similar effect of alkaline earth oxide in a porcelain body. Hence, very short vitrification range is expected on firing of the all γ–Alumina bodies containing slag. The additions of slag to γ–Alumina bodies show always low linear shrinkage as compared with standard mix (γAS0). This result is agree with Marghussion and Yekta [20]. They reported that alkaline earth oxide cause less shrinkage in slag containing composition.

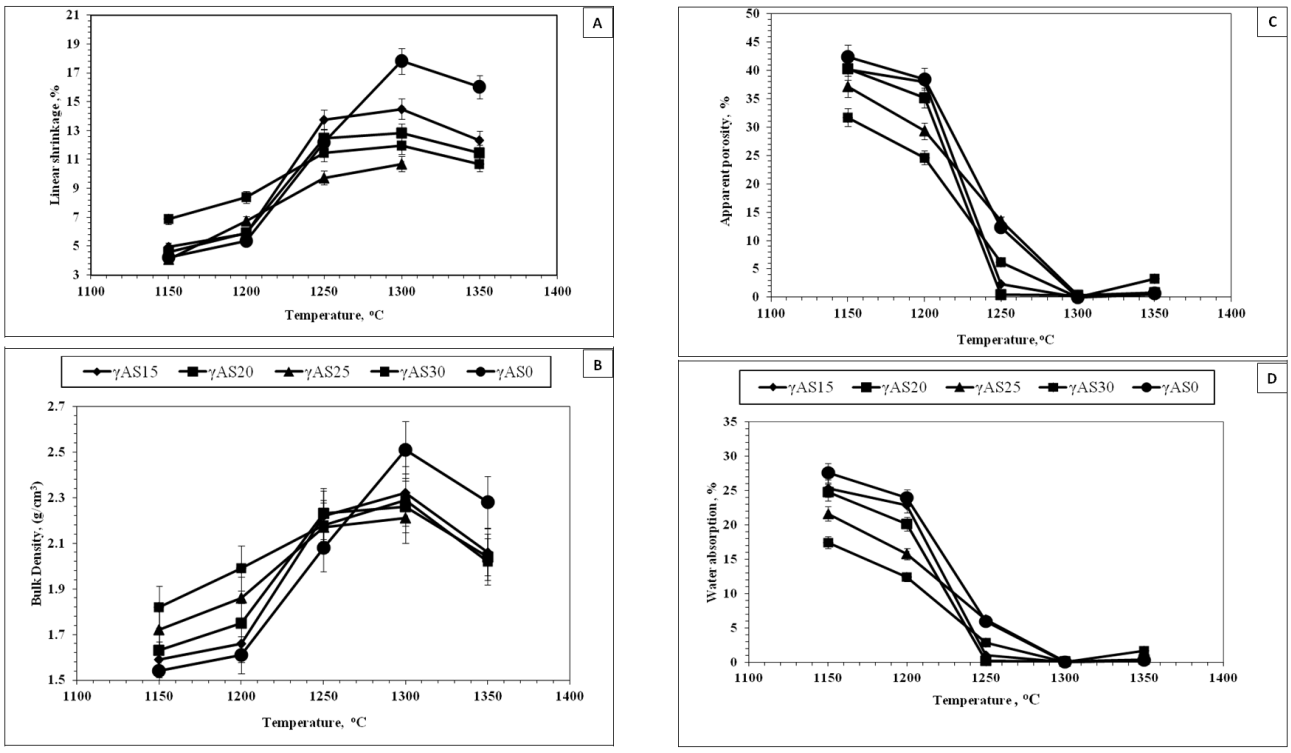

Figure 2. Physical properties of base composition (γAS0) and γ–Alumina bodies containing air blast furnace slag at different sintered temperatures, (A) linear shrinkage, (B) Bulk density, (C) Water absorption, (D) Apparent porosity

Table 3. Chemical composition of the studied batches

Sample symbol	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	K_2O	Na_2O	SO_3	Total fluxing oxide
γAS0	40.21	51.99	0.28	1.79	0.81	0.1	4.03	0.68	-	7.69
γAS15	39.06	46.92	0.33	1.88	6.92	0.77	2.85	0.75	0.38	13.50
γAS20	40.83	42.56	0.34	1.91	8.96	1.00	2.90	0.85	0.50	15.96
γAS25	42.60	38.20	0.36	1.94	11.00	1.23	2.95	0.95	0.63	18.43
γAS30	41.15	37.85	0.37	1.98	13.03	1.46	2.34	0.94	0.76	20.12

Table 4. Physico-mechanical properties of the base composition (γAS0) and/ γ–Alumina bodies containing air blast furnace slag at vitrified temperature

Sample symbol	vitrified Temperatures ◦C	B.D g/cm^3	A.P %	MOR N/mm^2
γAS0	1300	2.51	zero	101.01
γAS15	1300	2.32	zero	95.84
γAS20	1300	2.26	0.29	74.07
γAS25	1300	2.21	0.28	88.60
γAS30	1300	2.29	zero	88.50

AP: Apparent porosity BD: Bulk density MOR: modules of rupture

3.2. Crystalline Phase

The phase changes at the 1300 ℃ fired alumina bodies were investigated by X-ray diffraction analysis and the patterns are exhibited in Figure 3. XRD patterns of standard body (γAS0) which fired at 1300°C showed that γ-Alumina phase transformed into corundum and quartz into cristobalite as the major crystalline constituents with a higher real intensity as shown in Table 5. XRD patterns of standard body appear trace amount mullite phase. The absence of the standard peak at 2 Theta =16.34 belonging to mullite phase in the in the parent sample. Probably, a small amount of mullite was formed in γAS0 sample by the reaction of the metakaolinite caused by the dehydroxylation process of kaolin. However, this mullite phase was difficult to detect, may be due to the volume fraction and extremely small size. These results are agreement with results reported by authors [21] and [22]. Results of x-ray analysis of all specimens containing slag Figure 3 show that the main crystalline phases present are corundum, anorthite and cristobalite as the major crystalline phases. Alumina–slag ceramic bodies containing ceramic bodies considerable amount of CaO, SiO_2 and Al_2O_3 melted with feldspar, quartz and clay and the melted glassy phases, crystallized as anorthite ($CaO \cdot Al_2O_3 \cdot 2SiO_2$) [16]. Also, the presence of CaO caused the conversion of free quartz into cristobalite Figure 3. The disappearance of mullite and presence of more anorthite and cristobalite phases with a high real intensity are observed with increasing slag content from 15 up to 30 mass % for all samples, Figure 3 and Table 5.

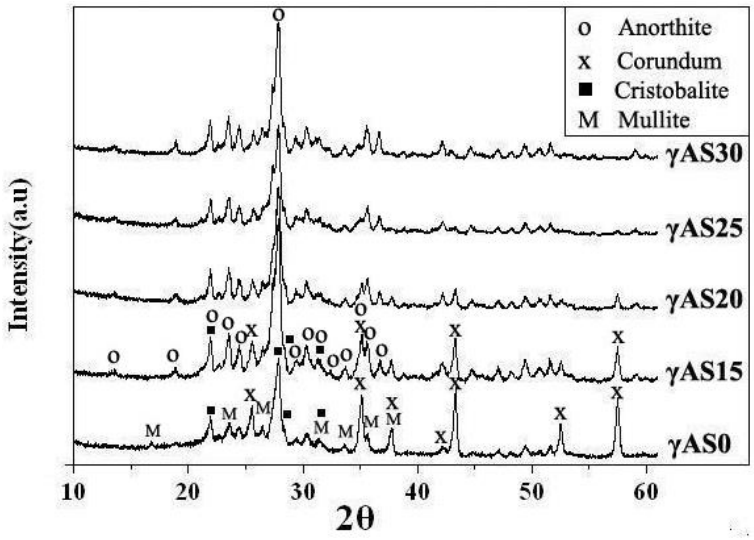

Figure 3. XRD patterns of (γAS0) and γ–Alumina bodies containing air blast furnace slag at vitrified temperature

Table 5. Real intensity of crystalline constituents and electrical properties of base composition (γAS0) and/ γ–Alumina bodies containing air blast furnace slag

sample symbol	Maturing firing Temp. ℃	Phase composition, Real Intensity				Electrical properties	
		mullite	corundum	anorthite	Cristobalite	Volume resistivity (Ω /cm)	Dielectric strength (kv/mm)
γAS0	1300	trace	70.04	-	27.70	12X10^{11}	15.76
γAS15	1300	-	31	100	28.01	25X10^{11}	11.00
γAS20	1300	-	18.12	100	25.43	25X10^{11}	11.68
γAS25	1300	-	11.37	100	26.29	15X10^{11}	12.39
γAS30	1300	-	25.28	100	28.77	30X10^{11}	12.43

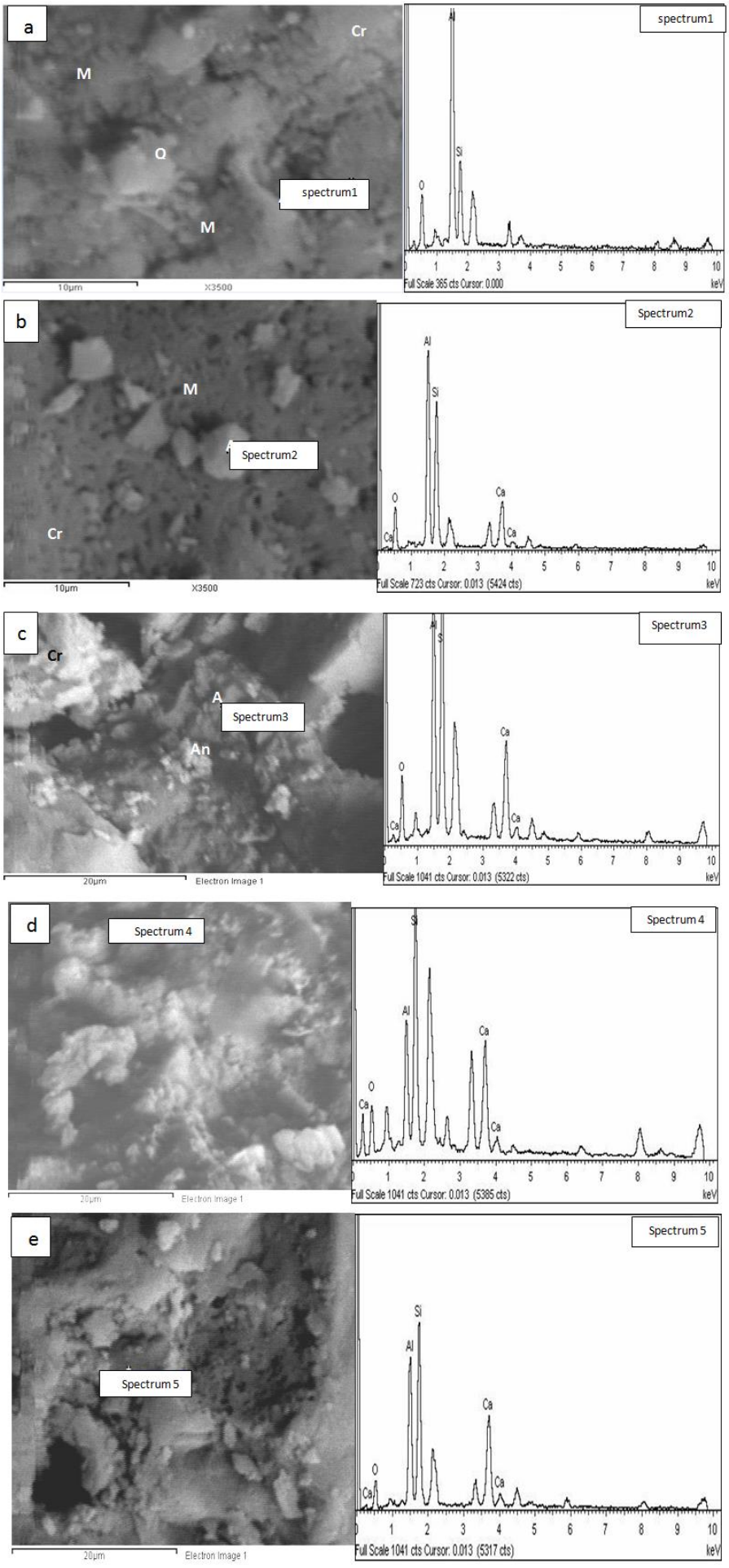

Figure 4. (a-e) SEM images with EDX spectra for bodies (γAS0, γAS15, γAS20, γAS25 and γAS30, respectively) at vitrified temperature

3.3. Microstructure

The microstructure of the vitrified base composition of alumina porcelain bodies based on slag using γ-Alumina (γAS0 body vitrified at 1300 ℃) is demonstrated in Figure 4 (a-e). It is observed from the SEM of fired specimens containing (30 γ-Alumina, 40 Tih clay and 30 Feldspar, mass%) at vitrified temperature have a crystalline phases with different sizes occurring as aggregates such as, trace mullite, scattered prismatic alumina crystals in addition to cristobalite grains. A mismatch between the glass and different crystalline phases appeared as cracks and fissures as seen in Figure 4 (a). It also shows that the mullite is recrystallized as secondary mullite having a stout prismatic form interlocking network located in the neighborhood of the alumina crystals. It will know that the secondary mullite forms by the reaction of clays with feldspar relicts after firing at 1000 ℃, in particular it originates from the surface of the clay relict, growing into the less viscous feldspar relicts, the more the matrix viscosity decreases, and the more size of secondary mullite increases.

To confirm the mullite and corundum phase crystallized, EDX analysis for γAS0 were performed with spectrum (1). The results indicated that the elements such as Al, Si and O are detected. It was suggested that mullite and corundum phases crystallized. These results agreement with the XRD patterns as given in Figure 3.

Figure 4 (b-e) show the microstructure of prepared γ-alumina ceramic bodies containing air blast furnace slag (γAS15, γAS20, γAS25 and γAS30 bodies) which fired at 1300 ℃ for 1h. The microstructure of all samples showed that the grain size and morphology are sensitive to the partial substitution of feldspar and /or γ-Alumina by slag in the γ-Alumina ceramic bodies. The SEM images Figure 4 (b-e) show that a dense microstructure with fine anorthite crystals, cristobalite and scattered alumina grains. Inhomogeneous distribution of grains, disconnected pores and inter-granular liquid phase formation along with extensive grain are declared with increasing slag additions. Circumferential cracks are also observed around grain probably due to larger difference in thermal expansion coefficient between glassy matrix and the other faces detected during cooling process. The EDX analysis was carried out for the aggregates fine grains of the γAS15, γAS20, γAS25 and γAS30 bodies in spectrums (2-5), Figure 4(b-e). As result, the EDX analysis indicated that the presence Al, Ca, Si and O elements suggests that calcium aluminum silicate, Ca Al Si_3 O_8).

3.4. Mechanical Properties

The values of the Modulus of rupture (MOR) for determination mechanical strength of the γ–Alumina insulator bodies containing slag (γAS15, γAS20, γAS25, γAS30) which fired at 1300 ℃/1h are presented in Table 4. For all series, the pure γAS0 (101.01 N/mm^2) sample has highest bending strength. However, the MOR is significant decline when 15 mass % to 20-30 mass% of slag added. The results indicate that the highest bending strength corresponds to the highest densities and the lower amount of slag: 101.01 N/mm^2 for γAS0, 95.84 N/mm^2 for γAS15, 74.07 N/mm^2 for γAS20 and ≤ 88.50 N/mm^2 for both γAS25 and γAS30. So, addition 15% slag to standard γ–Alumina porcelain composition enhances the densification and strength significantly increase in LR BD and decrease in AP as clear in Figure 2 followed by increasing in bending strength, in comparison with the other bodies containing slag Table 4. The increase in bending strength of standard γ–Alumina porcelain body is attributed to lower porosity values and free alumina grains, that remained in the microstructure, along with well-interlocked secondary mullite needles. While the bending strength decreasing with increasing slag content, may be related to the disappearance the mullite phase, high closed porosity, increased amount of liquid phase certainly affects negatively on the mechanical strength ceramic and to the additional crystallization during cooling. This observation agrees with the three main theories proposing explanations for the mechanical properties of porcelain [5] which consider that the crystalline phases present dispersed in the vitreous phase are the ruling factor influencing the mechanical properties.

3.5. Dielectric Properties

Table 5 summarizes the calculated electrical properties of the samples at room temperature. It is evident that the partial substitution of feldspar and /or γ-Alumina by slag from 15 to 30 mass % in the ceramic bodies containing γ-Alumina improves over two times the volume resistivity values varied between ($25x10^{11}$ and $30x10^{11}$Ω/cm) as compared with standard γ-Alumina body ($12x10^{11}$ Ω/cm). From results ,it is clear that the γAS25 sample recorded a reduce in volume resistivity value but still have a relatively high amount of volume resistivity ($15x10^{11}$Ω/cm) in comparison with standard γAS0 sample Table 5. Moreover, the produced silicate phase such as anorthite (Calcium aluminum silicate, Ca Al Si_3 O_8) during firing form a meshwork of prismatic crystals. This phase characterized by having relatively low thermal expansion and good mechanical strength, leading to a good thermal shock resistance and electrical properties [15,16,23].

Therefore, the high volume resistivity of the bodies containing slag, may be due to the higher activation energy of calcium ions which makes them less mobile as reported by kingery et al. [24], the less mobile modifier ions e.g. alkaline earth (Ba, Ca and Sr) with comparatively large ions and high charge (divalent). They are regarded CaO as one of most effective addition for dielectric bodies. Also, the increasing in volume resistivity up to 30 x 10^{11} Ω/cm with 30 mass % slag content can probably be attributed to the composition of resultant a fine texture microstructure formed in the alumina–slag ceramic body fired at 1300 ℃.

The variation of dielectric strength from (11 to 12.43 kv/mm) with composition of the samples is shown in Table 5. Above 25% slag content, the dielectric strength increase with increase in slag content but still with the occurrence of relatively low amount of dielectric strength as compared with γAS0 sample (15.76 kv/mm) Table 5. The strength values obtained for the samples are all within or above the range of 6.1–13 kv/ mm, which is the specified range for porcelain insulators [25]. Generally, the relative changes in dielectric strength values for the samples are very small.

4. Conclusion

The presence of slag from 15 up to 30 mass % to the standard mix (γAS0) which fired at 1300 °C for 1h led to relatively low bulk density (BD) ranging between (2.21 and 2.32 g/cm^3) as compared with standard mix (γAS0) (BD 2.51 g/cm^3), may be due to the presence of much high fluxing oxides in the fired bodies. The main phases recorded were anorthite, corundum, cristobalite with traces mullite. The relevant of electro-ceramic bodies produced exhibited high values for volume resistivity(VR) ($25x10^{11}$ to $30x10^{11}$ Ω/cm and relatively low values in the dielectric strength (DS) (11 to 12.43 kv/mm) as compared with standard mix (γAS0) (VR) ($12x10^{11}$Ω/cm) and (DS) (15.76 kv/mm), respectively. The present results show that, it is possible application of recycled slag after grinding and elimination any iron contamination for the production of low voltage electrical insulators electro-ceramic bodies.

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