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Research Article

Influence of quartz replacement by Iraqi porcelanite and silica fume on the properties of porcelain products

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Abstract

In this work, Iraqi porcelanite and silica fume were used as a silica source with replacement of quartz in the manufacture of porcelain products to investigate their influence on properties of these products. The specimens were prepared with composition (50: 30: 20) % as kaolin, silica source and feldspar respectively. Then, they dried at (383 K) temperature for 3 hrs. and sintered at (1473 K) temperature by soaking time for 2hrs. The fracture strength, hardness, apparent porosity, bulk density, linear shrinkage, and XRD, SEM and EDS analyses of the specimens were examined. The test results indicated that replacement of quartz by these sources improves the densification behavior and mechanical strength due to form of more amount from the glassy phase and interlocked crystals from mullite phase within the microstructure. Also, this replacement gives an economic interest for the energy saving and reduction of the material consumption, in addition to produce porcelain products with the best mechanical properties.

1. Introduction

Porcelain products are ceramic materials. Ceramic material can outperform its metal and polymer contestants in various applications due to its chemical inertness, low density relatively, that means lightweight, structural and thermal stability in high temperature, resistance of corrosion, involving hot corrosive gases or liquids [1-3]. Porcelain products are high-vitrified ceramic material manufactured from mixtures formulated from feldspar, quartz and kaolin. The feldspars [(K, Na)2O. Al2O3. 6H2O], that serve as fluxes; quartz or flint (SiO2), which retains the formed article shape through sintering; and kaolin [Al2Si2O5 (OH)4], which provides plasticity for the ceramic mixtures [4-6]. These three components put porcelain with the phases systems [(K, Na)2O-Al2O3-SiO2] by term of the oxides components, therefore it is named the triaxial porcelain ceramic [7]. The microstructure for the porcelain body constitutes from main phases which are needle shaped mullite crystals, a heterogeneous glassy matrix, irregular-shaped closed pores and some quartz particles. Mullite crystals, which form by the solid-state decomposition for feldspar with kaolin, donate the excellent chemical, thermal and mechanical properties. Porcelain composition consider from the most-complex ceramic systems because of the processing paths, the complex interaction among raw materials and the firing process kinetics [8, 9].

The increase of mechanical strength and the reduction of the production costs represent the quest over the period of time. In most attempts for increasing the strength, importance was concentrated for decreasing the quartz content within the formula of porcelain due to the phase transformation for β to α quartz that takes place during cooling at temperature (846 K). The phase transformation results into reduction of the volume for the quartz grain and may lead to crack in the ceramic body

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[10]. Therefore, there are researches to improve the mechanical properties via minimization of the quartz use. These involve substitutions of quartz by kyanite [11], Al2O3 [5, 12, 13], rice husk ash [8, 14-16], sillimanite sand [17], fly ash [18], partial substitution of quartz and feldspar with blast furnace slag and fly ash [19, 20], by a blend of silica fume and rice husk ash [21]. Also, there is an effort to part of quartz by fired porcelain that produces a negative result of the bending strength [22]. All these investigators observed a remarkable enhancement of the mechanical properties of porcelain bodies by use the substitution of quartz. In this work, the silica sources used for a substitution of quartz in porcelain bodies are Iraqi porcelanite and silica fume. Silica fume SF, which too defined as micro silica, produces into the metallurgical manufacturing as a waste product which arises from a manufacture of silicon alloys, ferrosilicon or metallic silicon. It involves of microscope sphere-shaped grains that approximately has 0.1 μm diameter and 20 m2/g surface area [23, 24]. It is described with flabby nature which makes it behaves such as a smoke or fume when spread in air, spherical shape, high surface area, and glassy nature [25-27]. In code ACI 116R, silica fume can be defined "very fine non-crystalline silica formed by the electrical arc furnace as a byproduct for the silicon element or by the production of alloys containing silicon" [28, 29]. While, Iraqi porcelanite is a term utilized for identification siliceous rocks by Iraqi geologist, and which is similar to diatomite. This rock is existent in diverse sites from Iraq. Iraqi porcelanite rock composed of Opal-CT (cristobalite-tridymite crystals deposits) derivative of biogenic amorphous opal silica (mainly of diatoms). Diatomite stratification present at several diverse countries and differs with its purity, quality, and utilization between the countries [30-32]. From the important physical properties of Iraqi porcelanite rocks are: porosity, fineness of pores, sorption capacity, light weight and low heat conductivity [32]. Table 1 displays the chemical composition for Iraqi kaolin clay [2], the Iraqi porcelanite rocks [31], and silica fume [21]. This work aims to study the influence of utilization of Iraqi porcelanite and silica fume as a silica source with replacement of quartz in the manufacture of porcelain products on the physical, mechanical and microstructural properties for these products.

Table 1. The chemical composition (% wt.) for some raw materials [2, 21, 31]

Constituents	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	P_2O_5	Cl	TiO_2	SO_4	LOI
Iraqi porcelanite rocks	70.36	2.6	1.0	5.4	6.1	0.73	0.14	1.42	1.08	-	0.37	10.8
Iraqi kaolin clay	47.4	38.5	0.5	0.3	0.3	0.3	0.6	-	-	0.1	-	12.0
Silica fume	96.05	1.53	0.39	0.60	-	0.34	-	-	-	-	-	0.90

LOI: loss on ignition.

2. Materials and Methods

In this study, Iraqi porcelanite and silica fume were used as a total replacement for quartz in porcelain manufacture. The specimens were prepared by a blend of raw materials with composition (50: 30: 20) % as kaolin, silica source and feldspar respectively. The mixing percentage for raw materials in the specimens is shown in Table 2.

Table 2. Composition of raw material (% wt.) for the specimens.

Raw material	Sample's symbol		
	A	B	C
Kaolin clay [$Al_2Si_2O_5(OH)_4$]	50	50	50
Sodium feldspar [$Na_2O.Al_2O_3.6H_2O$]	20	20	20
Quartz [SiO_2]	30	0	0
Silica fume [SiO_2]	0	30	0
Iraqi porcelanite [$SiO_2.CaO.MgO.Al_2O_3.(OH)_4$]	0	0	30

The powders of materials were mixed in an electrical mixer for 4 hrs in order to homogenize the mixture. The particle size averages of powders of kaolin clay and Iraqi porcelanite were tested by Bettersize 2000 laser particle size analyzer and that were 32.6 μm and 10.2 μm respectively. Table 3 shows the physical properties of Iraqi kaolin clay [2] and silica fume [6]. While, the bulk density and porosity are considered the main physical properties of Iraqi porcelanite rocks, which are (1.1) g/cm^3 and (38.5) % respectively [31].

Table 3. Physical properties of Iraqi kaolin clay and silica fume [2, 6]

Iraqi kaolin clay		Silica fume	
Property	Value	Property	Value
Color	Gray	Color	Grey
Density (g/cm^3)	1.8-2.6	Specific gravity	2.2
Melting point (k)	2043	Diameter (μm)	<1μm
Water solubility	Insoluble	Surface area (m^2/g)	15-30

Hydraulic pressing machine, 13 mm diameter of a steel die and 80 MPa pressure of pressing were used to form the porcelain samples. Polyvinyl alcohol PVA was employed as a plasticizer to bond particles of the materials powders with technique of a semi-dry pressing. Then, a drying furnace with (383 K) temperature was used for drying of the specimens from the moisture. After that, an electrical furnace with (1473 K) temperature, 278 K/min heating rate and 2 hrs soaking period was utilized for sintering of the specimens. Fig. 1 displays the specimens formed in this work.

Fig. 1. The specimens formed in this work: A (specimens with silica fume), B (specimens with Iraqi porcelanite), C (specimens with quartz)

After that, the samples properties were tested and these tests were included the physical properties (density, porosity and linear shrinkage), mechanical properties (fracture strength and hardness), XRD analysis and SEM of microstructure. They were made as the following:

Archimedes procedure was applied to calculate the porosity and density for porcelain specimens, which were tested depending on ASTM C373-88 standard, and Eqs. (1) and (2) were employed to calculate the density (D, g/cm^3) and porosity (P_o, %) for porcelain specimens respectively [33].

$$D = \frac{M_D * D_{water}}{M_S - M_P} \quad (1)$$

$$P_o\ \% = \frac{M_S - M_D}{M_S - M_P}.100 \quad (2)$$

where, D_{water} symbolizes the density of water (g/cm^3); M_D symbolizes a dry mass for the specimen (g); M_P symbolizes the suspended mass for sample (g); M_S symbolizes the water-saturated mass for sample (g). A linear shrinkage on firing (LS, %) for the sintered specimens was calculated depending on ASTM C1407 standard with usage Eq. (3) below.

$$LS = \frac{D_b - D_f}{D_b}.100 \quad (3)$$

where, D_b and D_f symbolize the diameters of sample before and after firing process respectively. Then, the mechanical testing machine was used to test the fracture strength of specimens ($б_f$, MPa) depending on ASTM standard C773-88 by use Eq. (4) below [34].

$$\sigma_f = \frac{R}{C_A} \tag{4}$$

where, R symbolize an applied force until fracture (N), and C_A symbolize the specimens' cross-section area (mm^2). The microhardness for specimens (HV, MPa) was tested by Vickers hardness test. 90 N indentation load applied for 10 s on the surface of the specimen and Eq. (5) were employed to measure Vickers hardness values depending on ASTM standard C1327- 90 [6, 35].

$$HV = 1.854.\ \frac{L}{n^2} \tag{5}$$

where, L symbolize indentation load (N), while n symbolize indentation diagonal on the sample surface (mm). After that, the powders of specimens were analyzed by XRD diffraction analysis for identification the crystalline phases developed in the specimens. The specimens were ground to take the fine powders that were utilized for XRD diffraction analysis. XRD pattern was gotten by employ SHIMADZU XRD – 6000 devices. This test was achieved by use the continuous scan mode with θ-2θ range as 20 °-70 °, 7 °/min scan speed and 2θ =0.02 ° the step size. Also, the microstructures and chemical composition of specimens were analyzed with scanning electron microscopy SEM and energy dispersive spectroscopy EDS to observe the surface morphology, structure and chemical composition for the specimens.

3. Results and Discussion

Different sources of silica were used as a substitution for quartz in porcelain specimens. These sources are Iraqi porcelanite and silica fume, and the content of silica in them was 70.36 % and 96.05 % respectively as shown in Table 1. These sources contain other chemical constituents. The chemical composition of these sources effected on the properties of porcelain specimens as explained below.

In Figs. 2, 3 and 4, the effect of silica source on the linear shrinkage on firing, apparent porosity and bulk density for the sintered samples is respectively shown. Where, the firing shrinkage and bulk density for specimen with Iraqi porcelanite C were the highest, and those for specimen with quartz A were the lowest among the porcelain specimens. The object in contrast with the porosity. Where, the apparent porosity for specimen with quartz A was the highest, and that for specimen with Iraqi porcelanite C was the lowest among the porcelain specimens. That because of the higher vitrification which happens in the specimens produced with Iraqi porcelanite and silica fume, due to form the glassy phase with more quality in these specimens, as a result to present the alkalis in the chemical composition of Iraqi porcelanite and silica fume which is shown in Table 1. In addition, it can be shown from Table 1 that the quality and quantity of alkalis in the chemical composition of Iraqi porcelanite are more than that of silica fume. Also, other authors emphasized that the sintering occurs in an existence of the liquid phase in many states. Particularly as various phases are existent, this sintering is named a liquid-phase sintering [4, 36, 37]. The liquid phase makes on the approach of particles under an effect of the forces of surface energy generated from tiny pores in specimens. Therefore, open porosity reduces with increasing the glassy phase formation in the specimen. Also, the firing shrinkage and bulk density for specimens increase with increasing of the sintering ability. That means an improving of the densification behavior for the porcelain specimens with replacement of quartz by Iraqi porcelanite or silica fume. This improved densification behavior for the porcelain specimens with presence of different silica sources was associated by the material reactivity, which means the higher surface area in comparison with the quartz powder. Similar results were observed by other works when quartz was replaced with different silica sources [14, 38, 39]. Also, the flabby nature of silica fume particles increased the firing shrinkage of specimen B [21]. It can be benefited from the results of this work in industrial environments, particularly that produced large amounts of porcelain products. Where, use of silica source contained considerable number of fluxes can be reduced or eliminated the use of fluxes in

porcelain manufacture and gave product with good properties. So, the economic interest in comparison with use of quartz from side of the material consumption, and the energy saving because of a presence of the flux's materials in a composition of these silica sources (Iraqi porcelanite and silica fume).

While Figs. 5 and 6 display the influence of silica source on the fracture strength and hardness for the porcelain specimens in that order. It can be shown that the fracture strength for specimen with silica fume B was the highest, and that for specimen with quartz A was the lowest among the porcelain specimens. While, the hardness for specimen with Iraqi porcelanite C was the highest, and that for specimen with quartz A was the lowest among the porcelain specimens. These results obtained because of the pores reduction and the bond process between the grains of specimens, due to form the glassy phase in the porcelain specimens, which means a higher vitrification for specimens. So, more rigid network formed in the specimens, that led to an enhancement in the mechanical properties. In addition, the results of XRD patterns and SEM images identify a presence of higher mullite phase in specimen B and C due to the existence of excess content from alumina in the composition of Iraqi porcelanite and silica fume, as shown in Table 1, that may be contributed in improving of the strength. Similar results were observed by other works [21, 38]. Improving of mechanical properties for products is an important point in the development of industry. Therefore, use of these silica sources (Iraqi porcelanite and silica fume) can be contributed in the development of porcelain industry.

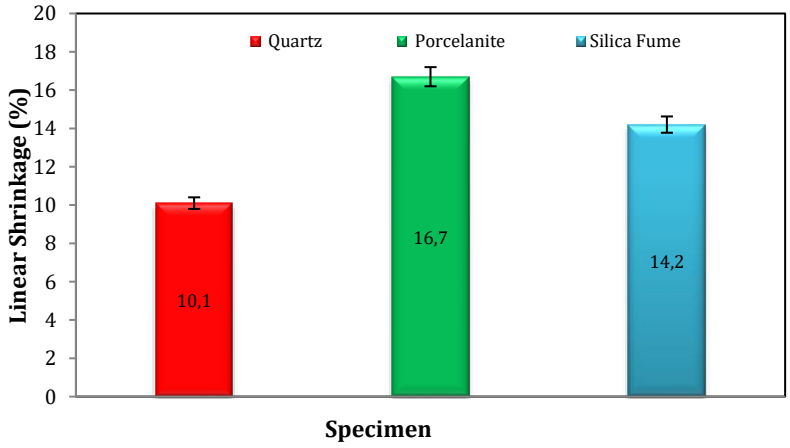

Fig. 2. Silica source effect on the firing shrinkage of porcelain specimens

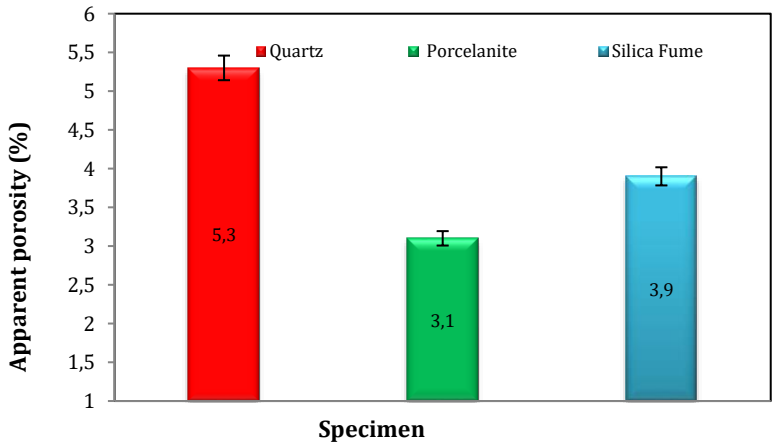

Fig. 3. Silica source effect on the apparent porosity of porcelain specimens

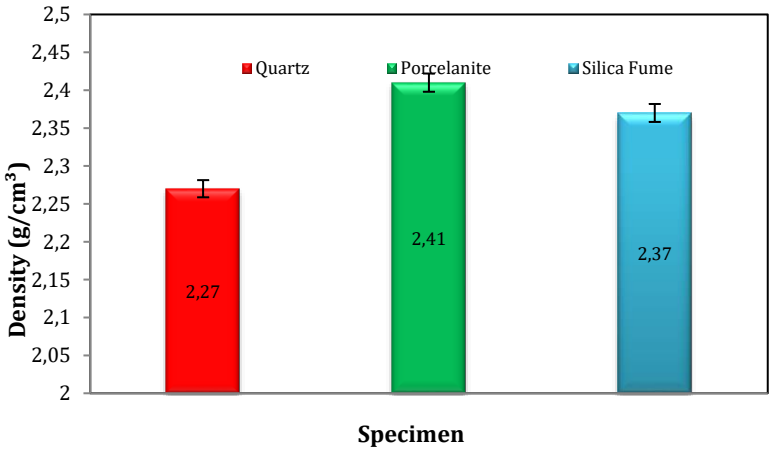

Fig. 4. Silica source effect on the bulk density of porcelain specimens

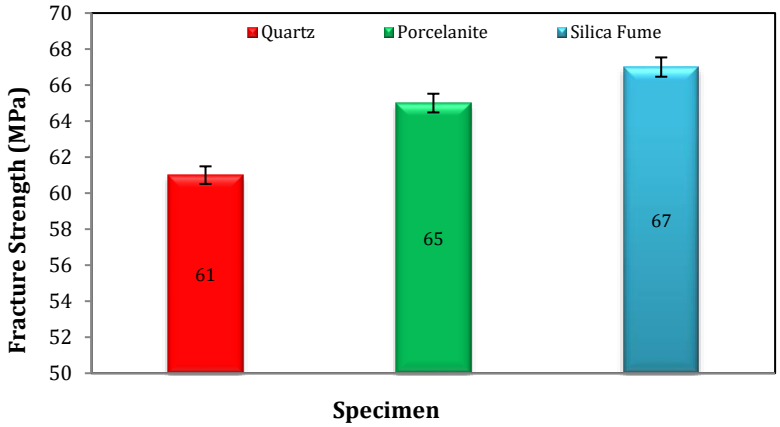

Fig. 5. Silica source effect on the fracture strength of porcelain specimens

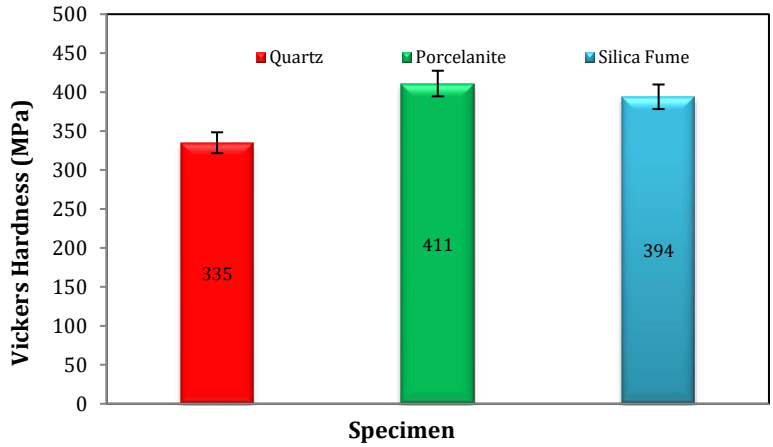

Fig. 6. Silica source effect on the hardness of porcelain specimens

The XRD patterns for the samples A, B and C, that are shown in Fig. 7, display the existence of two main crystalline phases that are, mullite (ICDD 074-4143) and quartz (ICDD 046-1045) in all the samples. It can be observed that the peaks of mullite phase increased and the peak intensity of quartz reduced considerably. XRD patterns showed that use of Iraqi porcelanite and silica fume significantly reduced an amount of the free quartz for the samples B and C, and increased the mullite phase due to the existence of excess content from alumina in their composition as shown in Table 1. Similar results were observed by other works [4, 15, 38].

While SEM images and EDS spectra for the specimens A, B and C are shown in Fig. 8 (a, c and e) SEM images and (b, d and f) EDS spectra for the specimens respectively. SEM images display the existence of a large quantity of remained undissolved free quartz in the microstructure for specimen A, and the reduction in content and size of the quartz crystals and the pores and also the increase in content of the needle-shaped, interlocked mullite crystals embedded with the glassy phase in the microstructure for B and C specimens due to replace the quartz with Iraqi porcelanite and silica fume. This replacement can be possible to remove a large amount from the inherent defects in the porcelain microstructure and to improve the mechanical properties for it. Similar results were observed by other works [21, 38, 40]. From EDS analysis results for the specimens, it can be shown that the Figs. 8 (b), (d) and (f) for the specimens A, B and C respectively are mainly composed of Si, Al and O elements with fewer amounts of Na, Mg, K, Ca and Fe elements in different percentages for all specimens. Where, Si element percentage in Fig. 8 (b) EDS spectra for the specimen A formed with quartz was higher in comparison with that in Fig. 8 (d) EDS spectra for the specimen B formed with silica fume and Fig. 8 (f) EDS spectra for the specimen C formed with Iraqi porcelanite. While, Al element percentage increases with respect to Si element percentage in Figs. 8 (d) and (f) due to the existence of excess content from alumina in composition of these specimens as shown in Table 1. In addition to existence of P and Cl elements in Fig. 8 (f) EDS spectra for the specimen C formed with Iraqi porcelanite due to presence of P_2O_5 and Cl in Iraqi porcelanite composition. These results corresponding with results of XRD analysis for the same specimens.

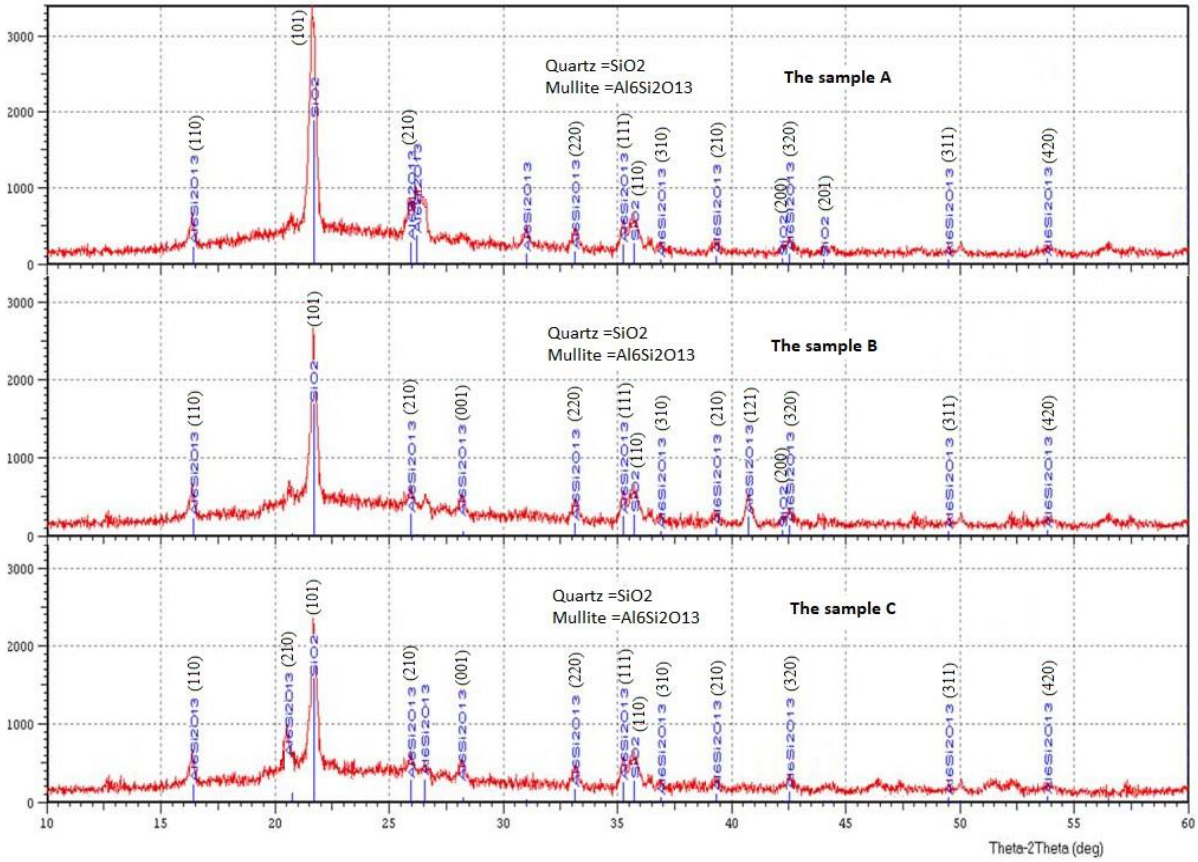

Fig. 7. XRD patterns of the sample A (with quartz), sample B (with silica fume) and sample C (with Iraqi porcelanite)

In comparison this work with researches utilizing alternative silica sources (such as references 4 and 21), these researches employ two alternative silica sources together in the same sample by partial and total substitution of quartz. While, this work employs the total substitution of quartz by one alternative silica source for each sample. Also, it employs Iraqi porcelanite (as alternative silica source) which has a rich chemical composition with the alkalis and an excess content from alumina in his composition. The alkalis are considered fluxes in the ceramic manufacture while alumina can

be contributed in formation a large amount of mullite phase. Thus, it can be obtained the best mechanical properties and densification behavior by use such this source.

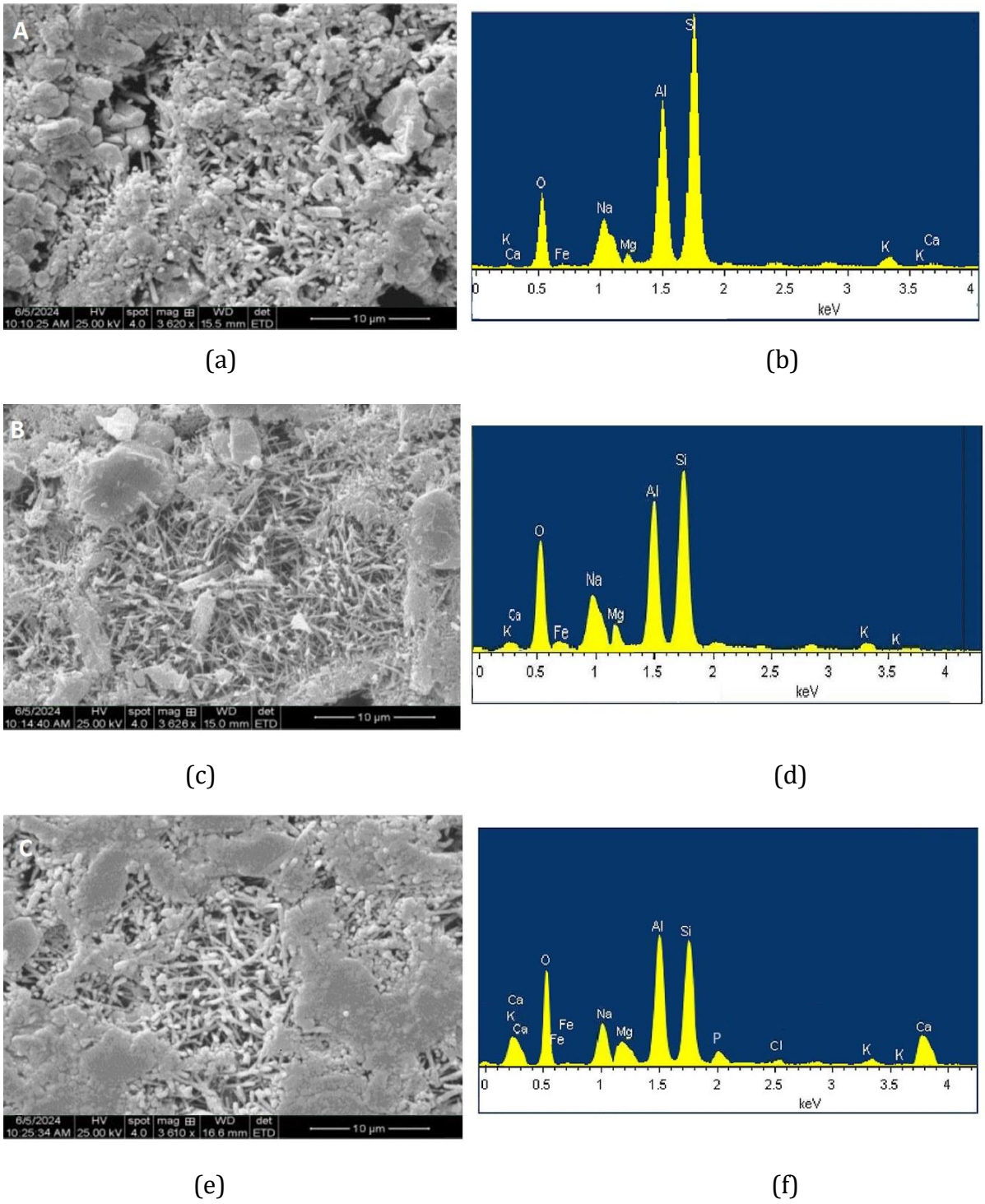

Fig. 8. (a) SEM image and (b) EDS spectra for the sample A (with quartz), (c) SEM image and (d) EDS spectra for the sample B (with silica fume), and (e) SEM image and (f) EDS spectra for the sample C (with Iraqi porcelanite)

4. Conclusions

In this work, Iraqi porcelanite and silica fume were used as a silica source with replacement of quartz in the manufacture of porcelain products to investigate their influence on properties of these products. The fracture strength, hardness, apparent porosity, bulk density, linear shrinkage, and

XRD, SEM and EDS analyses of the specimens were examined. From the results for these tests, it can be concluded that:

- The densification behaviour improves for the porcelain specimens with replacement of quartz by Iraqi porcelanite or silica fume due to form of more amount of the glassy phase in specimen B and C due to existence of considerable amount of the alkalis in their chemical composition.
- The mechanical strength for these specimens improves because of a formation of more quantity from well-interlocked mullite crystals, and a reduction of the content and size for both the quartz crystals and the pores in the microstructure of the specimen B and C.
- XRD patterns for the porcelain specimens show that use of Iraqi porcelanite and silica fume significantly reduce an amount of the free quartz for the samples B and C, and increase the mullite phase due to the existence of excess content from alumina in their composition.
- SEM and EDS analyses display that usage of Iraqi porcelanite and silica fume considerably reduce an amount of the free quartz for the samples B and C, and increase the mullite phase due to the presence of excess content from alumina in their composition.
- The replacement of quartz by Iraqi porcelanite or silica fume in the manufacture of porcelain products improves the physical, mechanical and microstructural properties for these products.
- The replacement of quartz by Iraqi porcelanite or silica fume in the manufacture of porcelain products gives the economic interest in comparison with use of quartz from side of the material consumption, and the energy saving because of a presence of the fluxes materials in a composition of these sources.

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