



Recycling of Ceramic Wastes for the Production of High Performance Mullite Refractories

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Received: 30 June 2019 / Accepted: 12 August 2019 / Published online: 29 August 2019
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Abstract

This work aims at benefit from the huge quantities of the ceramic wastes in KSA through recycling them to produce mullite-based refractories with expected outstanding properties. Because the ceramic wastes are mainly aluminosilicate, bauxite as a source of aluminum oxide is added to obtain mullite based refractory ceramics. The mixes containing different proportions of ceramics waste and raw bauxite were well mixed then shaped into cylindrical bodies using a compressing machine. After drying at 110 °C the obtained bodies were fired at 1500 °C. The phase compositions of the fired samples were followed using X-Ray diffraction technique (XRD). The microstructure of some selected samples was depicted using scanning electron microscope, spot analysis of some selected crystals was carried through energy dispersive x-ray technique. Densification parameters, mechanical as well as refractory properties of the fired samples were tested according to the International Standards. Up to 40 wt.% of ceramic waste could be consumed for production of high quality mullite-based refractories through mixing with 60 wt.% raw bauxite which decrease the beneficial problems resulting from the accumulation of ceramic waste material. The relatively higher mullite content with recognized mechanical and refractory properties reflects its properties on such mix which enable its use at high temperature and various engineering applications.

Keywords Ceramics · Wastes · Recycling · Mechanical · Mullite

1 Introduction

Saudi Arabia has witnessed a significant development over the last few years in the ceramic industry in line with the large increase in its uses due to the rapid urban development witnessed in the country. Some studies have recorded a rise in the demand for ceramic materials by more than 100% between the years 2001 and 2009, the production of ceramic tiles and porcelain increased from 35 million square meters in 2001 to 93 million square meters in 2009 while reaching 107 million square meters in 2012. As a result of the construction boom in the Kingdom

through the establishment of several mega projects, it is expected that the demand for these products will increase to about 300 million square meters by 2030. Therefore, the Kingdom attaches great importance to maintaining the equilibrium of the markets of the different production sectors in accordance with the requirements of development plans and programs and the objectives of the national strategy for industry until 2030. However, the increasing consumption of ceramics is accompanied by huge amounts of ceramic wastes whether during industry or demolition and construction. Elimination of this wastes has become more and more problematic, since the huge amount of this waste produced each year, leads to an increase of disposal cost.

In recent years, there is a scientific and social interest with the waste management problem especially the industrial and construction wastes. This is occurred through recycling or reuse such wastes which would be otherwise lost causing many ecological and economic problems. [1–3] Due to economic (energy saving) and ecological (air pollution) reasons the recycling or reuse of the wastes becomes more valuable in case of high temperature industries e.g. ceramics industry.

The annual worldwide consumption of ceramics reaches about 9000 million m² [2], this is accompanied with huge

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Table 1 Batches composition

Batch No.	% wt. Ceramic waste	% wt. Local raw bauxite
M1	100	00
M2	80	20
M3	60	40
M4	50	50
M5	40	60
M6	20	80

quantities of ceramic wastes (estimated by 30% of the total production) come either during ceramic industry or during construction or demolition process. In Europe it was reported that about 5–10% of the daily produced ceramics are go into wastes reaching to millions ton per year [3] which cause air, ground water or soil pollution because of the nature of these wastes. So, scientists and researchers try to solve this problem through exploitation of these wastes for other important applications. [1] several research works utilize ceramic wastes for concrete, ceramic or glass industries [2, 3].

Daigo et al., [4] could enhance the recyclability of ceramic materials for tiles and glass industries. Nepomuceno et al., [5] evaluate the possibility of utilizing the ceramic wastes as an aggregate in ordinary concrete, they incorporated natural fine aggregates (NFA), natural coarse aggregates (NCA) and the ceramic recycled coarse ceramic waste as aggregates (RCA). They could replace up to 70% of the aggregate with the ceramic wastes. Andreola et al., [6] used different industrial wastes e.g. scraps from packaging waste glass and cathode rays crushed tubes as a partial replacement of clays in ceramic industry. Reig et al., [7] improved the microstructure and hence mechanical properties of ceramic bodies through the alkali-activation process using tile ceramic waste TCW as a recycled aggregate.

In one of my previous works, I exploited the ceramic waste for nano-alumina powder extraction [8]. In this work we are going to reuse the ceramic waste to produce mullite-based high technical refractories.

Mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) is known as one of the most important minerals in engineering and metallurgical industries as well as many other advanced functional applications. This is correlated with its outstanding thermomechanical properties as well as high thermal stability [9–16].

Since the ceramic waste is mainly aluminosilicate, it may be suitable for the preparation of mullite refractories through addition of suitable proportion of local raw bauxite as a source of alumina to adjust the silica/alumina ratio to 3: 2 to produce high technical mullite based refractories used in a wide range of high temperature and engineering applications.

Because of the nature of the raw materials used in the manufacture of ceramics, its waste is expected to compose mainly of silica and alumina. So, they may be considered as a base of aluminosilicate (mullite) based refractories [17–19].

This work aims at benefit from the huge quantities of the ceramic wastes accumulated during its industry or during destruction and construction works together with the widespread bauxite raw mineral in KSA to produce high performance mullite based refractories for use at high temperature industries as well as in many advanced engineering applications. This will decrease the ecological problems arising from the accumulation of ceramic wastes and will assist in developing the local refractories industry.

The process is doubly valuable since it exploits the useless ceramic wastes that may cause many ecological problems to produce high technical mullite based refractories which are valuable to use as kiln furniture, lining of high temperature furnaces used in various industries e.g. iron and steel, cement, glass, petrochemicals...etc., they are used also in electronics and various engineering applications because of their outstanding properties.

2 Material and Methods

2.1 Material

- Ceramic waste material was collected from ceramic company, Jeddah, KSA.
- Local bauxite was collected from Zuberia region, eastern area of KSA.

2.2 Experimental

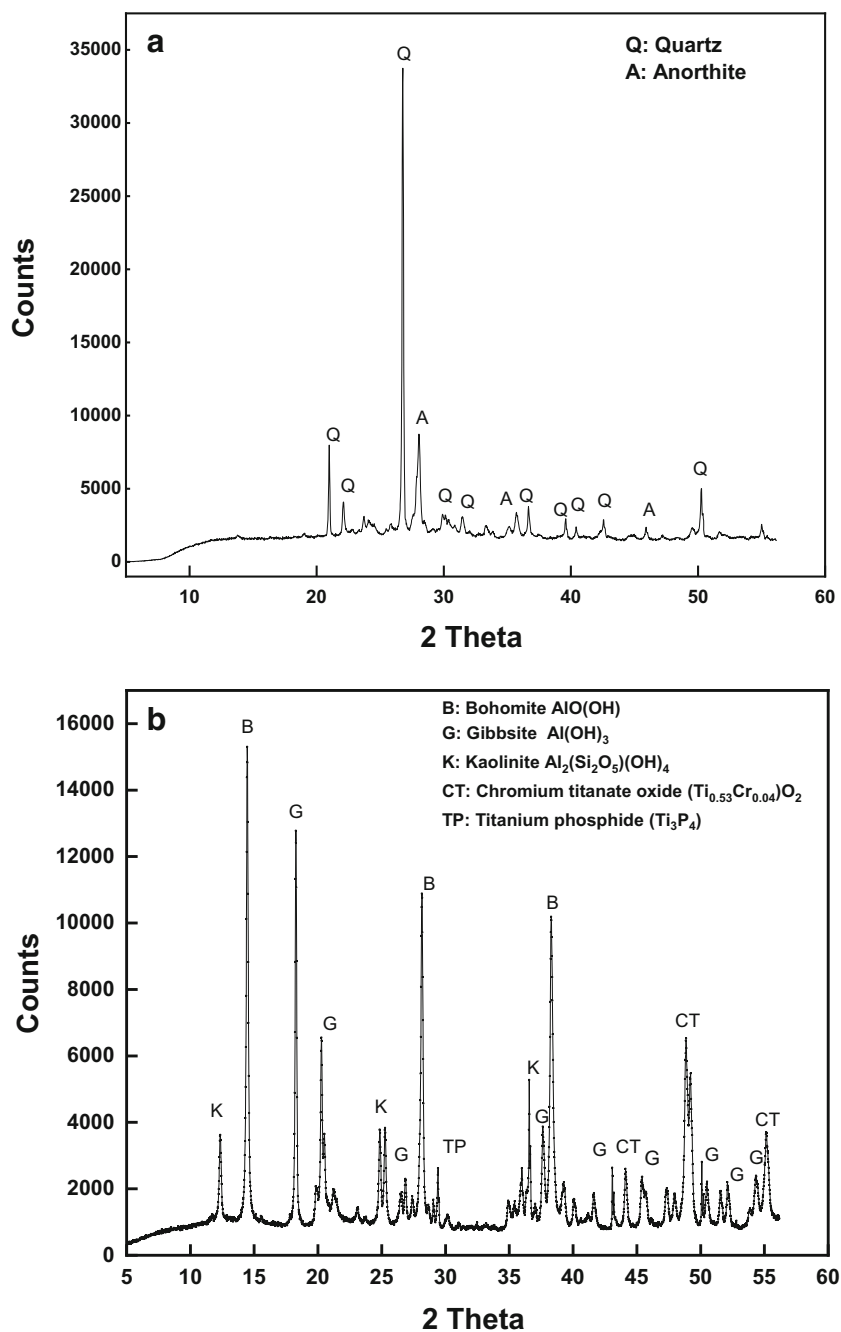
2.2.1 Preparation of the Ceramic Bodies

The collected ceramic wastes and local raw bauxite were dried, grounded, sieved to a particle size lesser than 100 μm .

Table 2 The chemical composition of the ceramic waste and local raw bauxite

Starting material	Chemical composition %, ± 0.01												
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	Na ₂ O	MnO	K ₂ O	P ₂ O ₅	SO ₃	Cr ₂ O ₃	LOI
Ceramic waste	60.30	17.36	5.54	0.87	11.57	1.11	1.09	0.05	1.59	0.20	0.19	–	0.79
Local raw Bauxite	6.30	77.05	1.22	2.11	0.75	0.75	–	–	–	0.01	–	0.80	11.74

Fig. 1 a XRD of ceramic waste. b XRD of raw bauxite



Six batches (each is 100 g) were prepared from ceramic wastes with different percentages of raw bauxite (as given in Table 1). After 5 min of hand mixing and 1 h mechanical mixing to assure the homogeneity of the batches. Five samples

(20 g of each) were uniaxially pressed into 3 cm high and with 4 cm width cylindrical bodies using a hydraulic compression machine at a constant uniaxial pressure of 800 kg/cm^2 . The green bodies were dried at 110 in an electric dryer for 24 h.,

Fig. 2 The batches M1-M6 at 1500°C

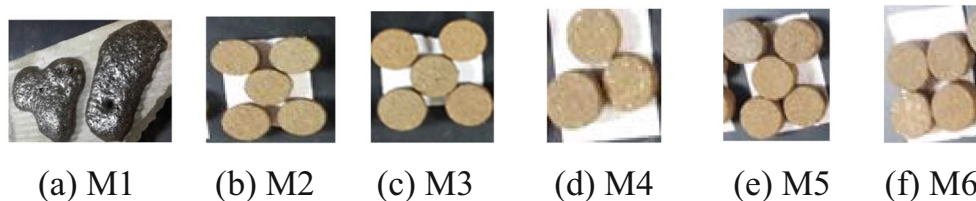


Table 3 Linear shrinkage of the ceramic bodies at 1500 °C

Batch	Linear shrinkage (%)
M2	3.10
M3	2.98
M4	2.70
M5	1.60
M6	1.64

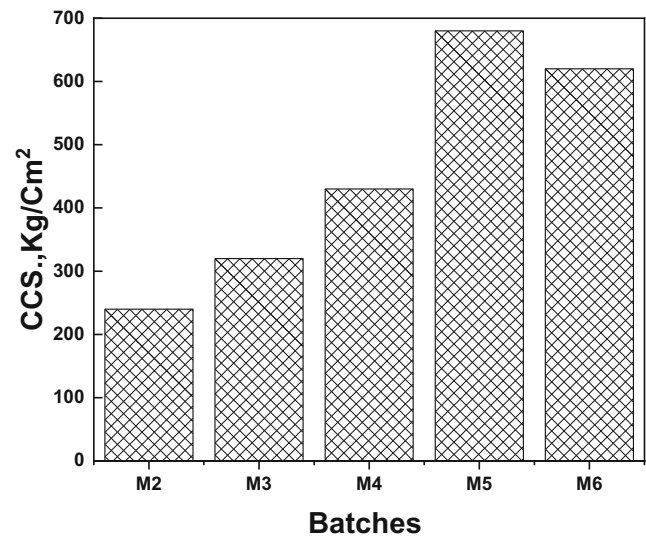
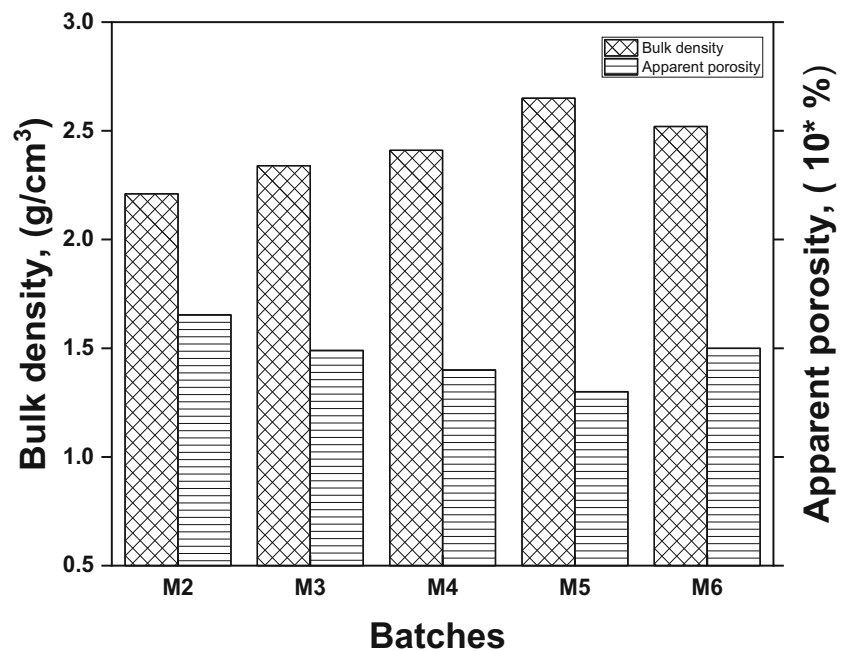
then calcined at 1000 °C before finally firing at 1500 °C for 1 h soaking time with 20 °C /min heating rate.

2.2.2 Instrumentation

The chemical and mineral composition of the ceramic waste and raw bauxite were carried out using Philips X-ray fluorescence (XRF) machine and X-ray diffractometer (XRD), D8 ADVANCE, Bruker, Germany, (diffractometer with Ni-filtered Cu K α radiation operating at 40 mA and 40 kV). The mineralogical compositions of the prepared ceramic bodies were also investigated after firing at 1500 °C, through X-ray diffraction (XRD) technique. The microstructure and spot analysis of some selected samples were tested by SEM (SEM; JEM-6460LV, Japan) attached with energy dispersive x-ray unit (EDAX).

2.2.3 Testing of the Prepared Ceramics Bodies

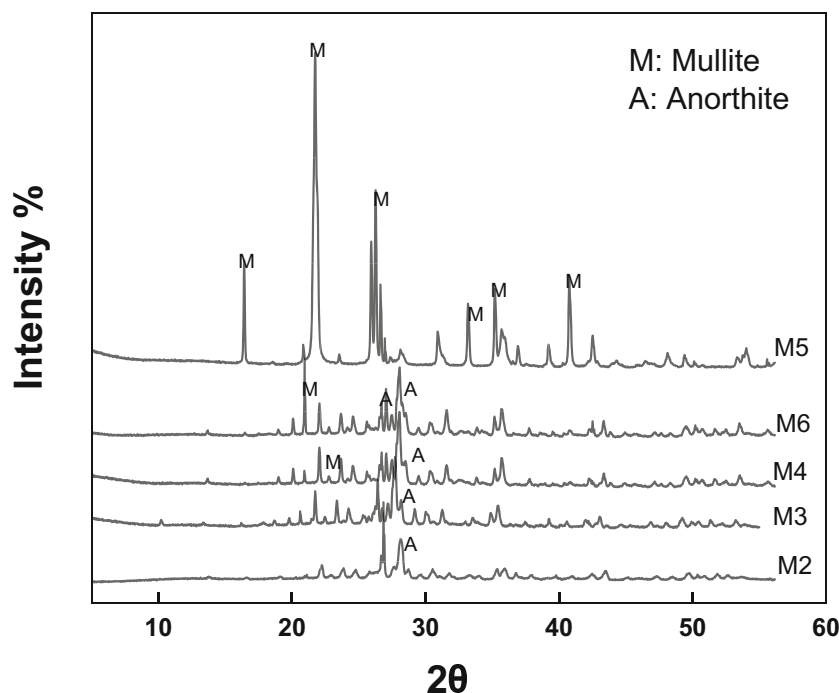
- According to the standard test method of linear change of refractories ASTM 350–10, linear shrinkage of the prepared bodies was calculated at each firing temperature from the following equation; $\frac{L_0 - L}{L_0} \times 100$ where L_0 is the side

Fig. 3 Bulk density & apparent porosity of batches M2-M6 at 1500 °C**Fig. 4** Mechanical strength of batches M2-M6 at 1500 °C

length of the green sample while L is the side length of the fired sample [20].

- Standard test methods for apparent porosity (A.P) and bulk density (B.D) of burned refractory brick and shapes using kerosene displacement method ASTM C838–96 (2010) was adopted to follow the sintering properties of the prepared samples [21].
- Standard test method ASTM C133–97 (2008) was adopted to test cold crushing strength (C.C.S) of the prepared bodies using a semi-automatic compression testing machine (2000 kn, model CN0123, UK) [22].
- Refractory properties involving; thermal shock resistance and refractoriness under load were tested according to the following International Standard tests, respectively;

Fig. 5 XRD patterns of the batches M2–M5



method for quantitatively measuring the effect of thermal shock and thermal cycling on refractories ASTM C1171–16 (2016) [23], method of measuring thermal expansion under load ASTM C832–00 (2015) [24].

3 Results and Discussions

3.1 Chemical Composition of the Ceramic Waste and Local Raw Bauxite

Table 2 shows the chemical composition of the ceramic waste and local raw bauxite determined through X-ray fluorescence analysis. The results indicate that ceramic waste contains mainly SiO_2 (60.30%) with considerable contents of Fe_2O_3 (5.54%), Al_2O_3 (17.36%), CaO (11.57%), in addition to little contents of other impurities. The bauxite mineral contains a high proportion of aluminum oxide Al_2O_3 (77.05%) and a considerable content of silicon dioxide SiO_2 (6.30%), in addition to small contents of other impurities. The loss of ignition (LOI) in the used raw bauxite was (11.74%). These data indicate that mixes of starting materials will compose mainly of alumina and silica which may favor the mullite formation especially at high contents of bauxite additions.

3.1.1 Mineral Composition of Ceramic Waste

Figure 1(a) shows that the ceramic waste consists mainly of Quartz (SiO_2), card no. 1011176 and Anorthite ($\text{CaAl}_2\text{Si}_2\text{O}_8$) card no.00–002–0523.

3.1.2 Mineral Composition of Local Raw Bauxite

Figure 1(b) shows that local raw bauxite consists mainly of the following minerals:

- Boehmite ($\text{AlO}(\text{OH})$) -50.8%, Card no. 00–021–1307).
- Gibbsite ($\text{Al}(\text{OH})_3$)–25.7%, Card no. 01–070–2038).
- Kaolinite ($\text{Al}_2\text{Si}_2\text{O}_5 (\text{OH})_4$)–15.8%, Card no. 01–079–1570)
- Chromium titanium oxide ($\text{Ti, Cr} \text{O}_2$)–2.2%, Card no. 01–076–7111).
- Titanium phosphide (Ti_4P_3)–5.6%, Card no. 01–077–3189).

The results of XRD are in an agreement with the those of XRF, the relatively higher contents of SiO_2 , Al_2O_3 and CaO in ceramic waste are presents as quartz and anorthite phases. Bauxite mineral is composed mainly of boehmite ($\text{AlO}(\text{OH})$) and gibbsite ($\text{Al}(\text{OH})_3$) in addition to a relatively lower percentage of kaolinite ($\text{Al}_2\text{Si}_2\text{O}_5 (\text{OH})_4$) which is confirmed by the presence of a high percentage of aluminum oxide (77.05%) in the chemical analysis of the bauxite.

These results led to the idea of this research work, as both the ceramic waste and local raw bauxite contain appreciable amounts of high temperature oxides, namely, Al_2O_3 (2050 °C), and silicon (SiO_2) (1710 °C). These oxides can interact together at high temperatures giving mullite mineral ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) with high melting point (1930 °C) [25–28].

3.2 Effect of Firing Temperature on the Form of the Prepared Ceramic Bodies

Figure 2(a-f), shows the effect of firing temperature on the shape of the cylindrical bodies prepared from mixtures of different proportions of ceramic waste and bauxite mineral (M1-M6). The ceramic bodies M1 containing 100 wt.% of ceramic wastes –00 wt.% of bauxite mineral were completely defused at 1500 °C (Fig. 2(a)) whereas the remaining samples (M2-M6) could stand at that temperature (Fig. 2(b-f)), this is correlated with the absence of bauxite in M1 batch and hence the deficiency of its alumina content and increase in other impurities compared with other batches resulting in the formation of high content of low melting point compounds and hence deformation of the samples [10, 12]. Therefore, M1 batch was excluded from further study since they didn't meet the minimum requirements of the International Standards for refractories specifications (cannot stand >1500 °C) [25].

3.3 Technological Properties of the Prepared Ceramic Bodies

3.3.1 Linear Shrinkage (%)

Table 3 shows a pronounced decrease in linear shrinkage as the content of bauxite increases, (i.e., M2 → M5) this is due to the increase in the formed mullite content which decreases the effect of impurities as a result of the consumption of Fe_2O_3 and TiO_2 in solid solution formation during the densification process [9, 10, 16, 17]. At higher content of bauxite (M6 containing 80 wt.%) the decrease in linear shrinkage becomes less pronounced may be due to the deficiency of the main source of silica (ceramic waste) and hence the decrease in the formed mullite content these results are in accordance with those published by Maldhure et al., [29]. The lowest linear shrinkage of batch M5 containing (60 wt.% bauxite) is correlated with its relatively higher contents of mullite.

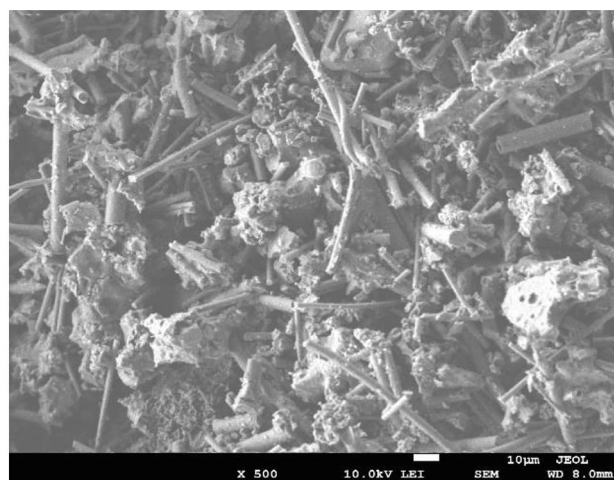
3.3.2 Bulk Density and Apparent Porosity

As shown in Fig. 3 the densification (bulk density) increases while apparent porosity decreases as the content of bauxite increases on the expense of ceramic waste up to M5 (60 wt.% bauxite +40 wt.% ceramic waste) at which the density shows its maximum value (2.65 g/cm^3) while the porosity is minimum (13.00%). Further increase in bauxite content in M6 (80 wt.% bauxite +20 wt.% ceramic waste) leads to a pronounced decrease in densification (i.e., decrease in bulk density and increase in apparent porosity). The gradual improvement in densification of M2 to M5 is due to gradual increase in the content of mullite in addition to glassy phases resulting from the impurities present in ceramic waste which fill pores and voids in the matrix resulting in a dense body.

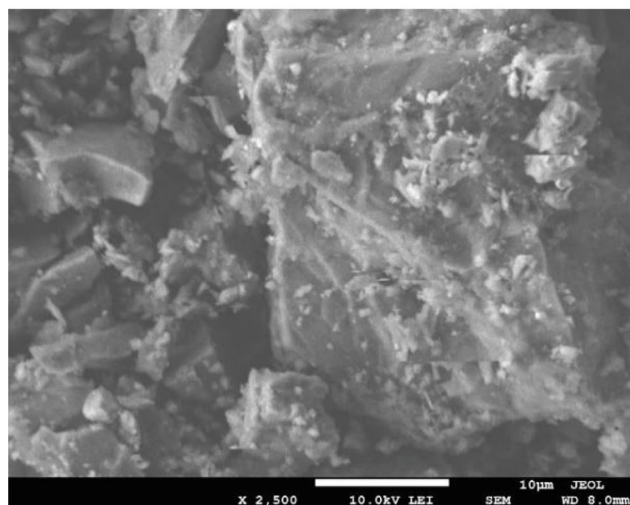
Also increasing the bauxite content means increasing in the content of Fe_2O_3 and TiO_2 which form at high temperature solid solution ($\text{Fe}_2\text{O}_3 - \text{TiO}_2$) that assists sintering [10, 28, 29]. In M6 the contents of impurities and silica decrease as a result of the deficiency of ceramic waste in this batch and hence the decrease in the content of glassy phases and the chances of mullite phase formation which leave porous matrix and hence lower densification [29].

3.3.3 Mechanical Strength of the Prepared Ceramics Bodies

Figure 4 shows a pronounced improvement the mechanical strength (cold crushing strength, CCS) as the content of bauxite in the batch increases on the expense of ceramic waste reaching its maximum value (680 kg/cm^2) in batch M5 beyond which (in batch M6) the mechanical strength is markedly decreased. The improvement in mechanical strength as we proceed from M2 to M5 is correlated directly with the system



(a): SEM for M5



(b) : SEM for M6

Fig. 6 a SEM for M5. b SEM for M6

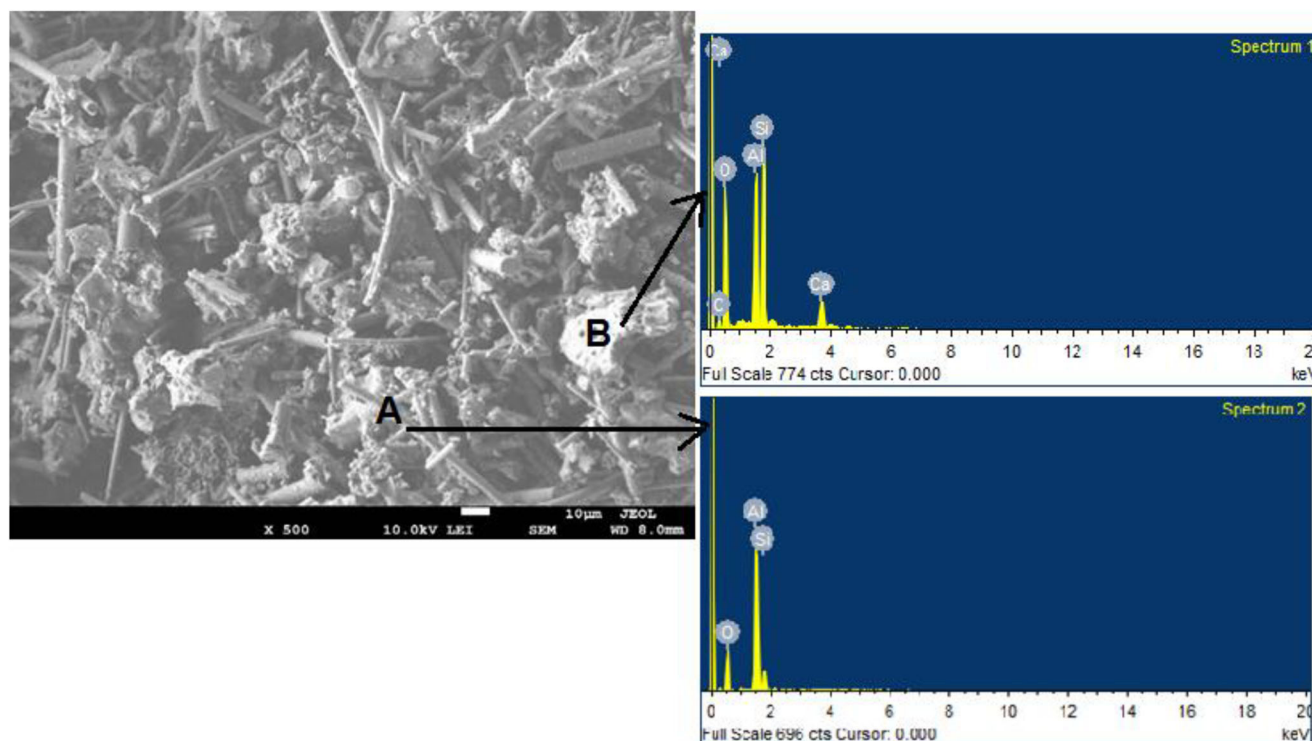


Fig. 7 EDAX analysis of some crystals in samples of M5 batch

mullite-glassy phases-solid solution, this system predominates in batch M5, so it shows the highest mechanical strength. This system decreases in M6 as a result of the sharp decrease in the ceramic content in this batch leading to a relatively decrease in mechanical properties [10, 29].

3.4 Phase Composition and Microstructure

The XRD patterns of the fired batches shown in Fig. 5 confirm our explanation of the improvement of densification and mechanical properties from M2-M6 in which the lines characterizing mullite content increases with the increase in bauxite contents in addition to the presence of considerable content of anorthite (low melting glassy phases). In M6 the intensity of mullite content and anorthite phase is sharply decrease as a result of deficiency in ceramic waste (source of silica and

impurities) which explain the deterioration in sintering and hence mechanical properties of M6.

Microstructure of some selected fired batches shown in Fig. 6(a & b) show a uniform development of rod-like crystals characterizing mullite in M5 batch (Fig. 6(a)) which interlock with the glassy phases and solid solution leading to a compact texture which reflects on their physicomaterial properties.

By calculating the percent of anorthite ($\text{CaOAl}_2\text{O}_3 \cdot 2\text{SiO}_2$) formed in mix 5 (40% ceramic waste +60% bauxite) based on the ignited weight of the starting materials [11, 28] it was found that this mix will contain about 20% wt. anorthite and about 44.5 mullite. The presence of this content of glassy phase together with mullite mineral assists and improves sintering and hence mechanical properties. On further mixes (containing relatively higher contents of ceramic wastes) the content of glassy phase will be increased on the expense of mullite mineral as a result of increasing the calcium oxide content in the mixes associated with the increase of its source

Table 4 EDAX analysis data of Crystals A and B

Element	Composition, %	
	Crystal A	Crystal B
C	—	5.14
O	31.70	37.08
Al	56.44	17.04
Si	11.86	28.24
Ca	—	12.50
Total	100.00	100.00

Table 5 Thermal shock resistance of the prepared batches

Batch No.	Thermal shock resistance (cycle)	% of retained strength
M2	16	—
M3	22	—
M4	30 <	58
M5	30 <	80
M6	30 <	64

Table 6 (T_a °C) of batches M2–M6

Batch No.	T_a , °C
M2	1410
M3	1430
M4	1490
M5	1530
M6	1515

(ceramic waste), Table 2 which affects adversely on the sintering and hence mechanical properties.. These theoretical predictions were confirmed with experimental observations of XRD and SEM.

In batch M6 (Fig. 5(b)) the content of mullite crystals are less than in M5 with a noticed deficiency in glassy phases as a result of the deficiency of impurities this creates a less compactness microstructure and hence decrease in sintering and mechanical properties. Figure 7 and Table 4 shows the EDAX analysis of some crystals in samples of M5. The rod like crystals (point A) are rich in aluminum (56.44%), its content is approximately five folds the content of silicon (11.86%), this crystal is also rich in oxygen content (31.70%), this chemical compositions confirm the presence of mullite mineral in which the aluminum oxide is one and a half folds the silicon oxide content ($3Al_2O_3 \cdot 2SiO_2$). Non uniform crystal (point B) contains a considerable content of calcium (12.53%) in addition to aluminum (17.04%) with larger content of silicon (26.34%) and oxygen (58.95%) which confirm the possibility of the presence of glassy phase (anorthite; $CaOAl_2O_3 \cdot 2SiO_2$). The data of SEM and EDAX confirm the results of XRD and hence support our explanation to the improvement in sintering and mechanical properties of M5 samples.

3.5 Refractory Properties of the Prepared Ceramic Bodies

3.5.1 Thermal Shock Resistance

As given in Table 5 only samples of batches 4,5 and 6 could withstand 30 cycles of heating at 1000 °C and quenching of water without spalling or any sign of cracking. The table indicates also that samples of batch 5 could maintain 80% of its initial strength while samples of batches 4 and 6 could maintain only 50 and 60 of their strengths. The recognized thermal shock resistance of batch 5 samples is due to its higher relatively content of mullite mineral with relatively low reversible thermal expansion, excellent thermomechanical properties and chemical stability [30]. On the other hand, relatively high content of glassy phases in samples of batches M2–M4 decrease the chance of mullite formation which affect adversely their spalling resistance. Samples of batch 6 with relatively lower contents of mullite compared with those of batch 5, show relatively lower spalling resistance. The presence of

considerable content of glassy phases in batch M5 bonded tightly the mullite crystals resulting in intergranular fracture while the lack of glassy phases in batch M6 leads to transgranular fracture of mullite [17–19, 29].

3.5.2 Refractory under Load (Load Bearing Capacity)

Table 6 indicates that samples of batches 4, 5 and 6 show relatively higher T_a (temperature of beginning of subsidence) corresponding to 6% deformation of the samples; 1480, 1530 and 1515 °C, respectively. T_a of other batches M2 and M3 are relatively lower; 1410, 1430 due to their relatively higher contents of impurities leading to relatively higher content of low melting compound (anorthite) [29], which affect adversely also on the contents of the formed mullite mineral. Again, the better assemblage and balance of minerals; mullite - glassy phase in samples of batch M5 results in relatively higher refractory characteristics [9–12].

4 Conclusion

Up to 40 wt.% of ceramic waste could be consumed together with 60 wt.% raw bauxite to produce high quality mullite-based refractory ceramics. The process is doubly valuable since it exploits the useless ceramic wastes that may cause many ecological problems to produce high technical mullite based refractories which are valuable to use as kiln furniture, lining of high temperature furnaces used in various industries e.g. iron and steel, cement, glass, petrochemicals...etc., they are used also in electronics and various engineering applications because of their outstanding properties.

Acknowledgements This work was funded by the Deanship of Scientific Research (DSR), University of Jeddah, under grant no. (UJ- 42- 18- DR). The authors, therefore, acknowledge with thanks DSR technical and financial support.

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