

## Utilization of sugar processing filter cake instead of calcite in production of anorthite based ceramics

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In recent years, the reuse of industrially generated residues in ceramic bodies has been an important issue. Reuse of industrial waste products is not only environmental responsibility, but also cost reduction and efficient use of raw materials. The aim of the study was to investigate the possibility of using press filter cake (PFC) instead of calcite in the production of anorthite-based ceramics. The PFC is an industrial solid waste, which is a by-product in the production of sugar from sugar beet. This solid waste quantity is about one million ton per year in Turkey. PFC was evaluated as alternative raw material in the preparation of anorthite based ceramic bodies. In this study, anorthite based ceramics were produced by using two different types of calcium oxide source. While PFC and calcite were used as CaO source, clay was used as the source of aluminum silicate. Compositions were prepared from 30% calcium oxide sources and 70% clay by weight. The ceramic bodies were sintered at different temperatures from 1000 °C to 1200 °C. The micro-porous structure of ceramics were obtained by burning of organic substances and by decomposing of carbonate compounds of the PFC wastes during firing process. The differences between the physical, mechanical and microstructural properties of anorthite based ceramics produced from two different CaO sources were investigated. Also, their phase analysis results were compared. While the specimens with 30 wt% PFC addition fired at 1200 °C contained anorthite ( $\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ) and cristobalite ( $\text{SiO}_2$ ) as major phase, the specimens with 30wt% calcite addition fired at same temperature included anorthite as major phase and minor secondary phases such as cristobalite and quartz.

**Keywords:** Sugar processing wastes, Firing, CAS ceramics, Phase analysis, Microstructure.

### Introduction

Anorthite ( $\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ) is one of the crystalline phases of the ceramic material, which has a calcium-aluminum-silicon oxide (CAS) composition. Due to its low thermal conductivity, low theoretical density, high melting point, low dielectric constant, this ceramic composition has a great potential for using in insulation, refractory and electronic applications. [1-6]. Anorthite-based ceramics can be commonly obtained as glass-ceramics or ceramics, sometimes by controlled crystallization of glass, or sometimes from kaolin and calcium carbonate mixtures. These ceramics produced in dense and porous forms are preferred in many different applications due to their interesting properties. For instance, the mechanical strength of clay-based ceramics can be increased by forming anorthite crystals embedded in the matrix in their microstructures when sintered even at low temperatures using clay and calcite mixtures. In general, in the formation of such a microstructure

during thermal transformations, the reaction sequence takes place as metakaolinite-gehlenite-anorthite [7]. Many researchers have used low-cost natural raw materials such as clay, kaolin, calcite, dolomite, quartz and feldspar to prepare these ceramics. In many studies, the synthesis of anorthite powders and the production of dense anorthite ceramics have been extensively studied using different methods such as sintering of raw materials, mechano-chemical processes and the use of different sintering aids [8-16]. Kurama et al. [8] evaluated the effect of different calcium oxide sources in order to produce anorthite ceramics. Also, sintering behaviors of anorthite based porcelain bodies was investigated by using additives such as wollastonite and dolomite as a calcium source [17, 18]. Moreover, the formation of anorthite crystalline phase was determined in glass-ceramic glazes derived from industrial wastes such as blast furnace slag and fly ash [19]. In addition to research on the development of dense anorthite ceramics, some studies have recently attracted attention on the development of porous anorthite ceramics from different sources of raw materials, especially industrial wastes. Porous anorthite ceramics are generally utilized as lightweight thermal insulation materials in

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refractory applications [12, 13]. Some researchers have reported their findings in their effort to develop the properties of anorthite based lightweight insulating materials produced by using of some wastes such as eggshell, marble powder, recycled paper processing residues as a source of calcium oxide [11-14]. The use of industrial wastes as a source of calcium for the anorthite composition as well as the pore former in the production of porous anorthite ceramics has gained importance due to its economic, environmental and energy saving advantages [8, 11, 14, 20-24].

In this study, press filter cake (PFC), an industrial solid waste of sugar factory, was used to produce anorthite based ceramics. The PFC is an industrial waste that is exposed in the carbonation treatments in sugar factories. It contains organic and inorganic substances and consists mainly of calcium carbonate ( $\text{CaCO}_3$ ). This residue could not be reuse for the sugar process. The resulting wastes are either drained into the environment or stored at the factory site. This process negatively affects the quality of the rivers in terms of environment and can damage the soil in the region due to the high calcium carbonate content. PFCs stored at the factory site also cause various problems in terms of waste management. As a result of industrial processes performed in the sugar factory in Turkey it is produced about one million tons of PFC wastes annually [25]. Therefore, the different recovery or reassessment studies are performed to overcome this problem. This waste from the sugar beet industry has been used by some researchers in the production of anorthite-based ceramics as a source of calcium oxide [21-24]. Using this waste together with a local clay, El-Maghraby et al. [21] obtained samples with a dominant anorthite crystal phase, bending strength of about 25 MPa, and a relative density of 64.5%, after sintering at high temperatures above 1200 °C. Naga et al. [22] investigated the effects of the ratio of crystalline phases on the physical, mechanical, electrical and thermal expansion coefficient properties of the cordierite/anorthite ceramic composites prepared by using sugar beet filter cake and talc. In another study, Naga et al. [23] prepared anorthite powder by using sugar beet industry waste and kaolin, then used it to prepare anorthite-alumina composites. The physical and mechanical properties of these composites were studied as a function of the amount of added alumina. Moreover, Man et al. [24] prepared the porous bricks that consist of 80 wt% diatomite, 20 wt% sugar filter cake, which have open porosity of 50% and sufficient flexural strength of 10 MPa after firing.

The aim of the study was to investigate the possibility of using PFC as a source of calcium and pore former to replace calcite in the manufacture of a porous anorthite-based ceramic product. In this study, the physical, mechanical and also, microstructural characteristics of the produced samples were investigated.

## Experimental

The sugar filter cake is obtained from the Eskişehir Sugar Factory (in Turkey), which produced 50,000 tons of sugar cake per year. Clay was provided by Omya Inc. (Kırşehir, Turkey) and calcite was supplied by Kale Mine Company (Çanakkale, Turkey). The raw materials were analyzed for particle size, chemical composition, phase content and thermal behavior. Particle size distribution of the raw materials was measured by Malvern Mastersizer 3000. An Energy Dispersive X-ray Fluorescence Spectrometer (XRF, Rigaku NEX CG Model) was used for determining their chemical composition. The phase compositions of the raw materials were identified by X-ray diffraction (XRD, Bruker D2 Phaser) with Cu tube of 1,54 Å at voltage of 30kV. Thermogravimetric analysis (TGA) of the raw materials was carried out under nitrogen atmosphere at heating rate of 10 °C/min using the TA Instruments TGA-SDT Q600 equipment. The particle morphology of raw materials was observed by a scanning electron microscope (SEM, Carl Zeiss 300VP). Before SEM observation, all samples were gold coated.

The mixture compositions composed of 30% PFC + 70% clay (CP mixture coded), and 30% calcite + 70% clay (CC mixture coded) by weight. The slurry of batch compositions were prepared at 1215 g/l density and mixed with rotational speed of 200 rpm for 45 min to provide homogeneous blend. Then, the slurries were dried at 100 °C for 24 h and ground in a mortar to obtain powder mixtures. The mixtures were uniaxially compacted in form of cylindrical pellet with 20 mm diameter at hydraulic press under a pressure of 25 MPa. After the samples were dried at 100 °C for 24 h, they were fired at 1000, 1050, 1100, 1150 and 1200 °C for 2 hours to obtain anorthite ( $\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$ ) as major crystalline phase.

The fired samples were characterized for their physical properties, crystalline phases and microstructural properties as well as compressive strengths and thermal conductivities. The physical properties of fired samples such as bulk density, apparent porosity and water absorption were evaluated by the water immersion method according to ASTM C20 [26]. Crystal phase formations and microstructures in the fired samples were examined with XRD and SEM, respectively. Compressive strength of the samples was measured by Shimadzu testing machine with 100 kN capacity at a crosshead speed of 0.5 mm/min. For measuring the thermal conductivity of samples, C-Therm Thermal Conductivity Analyzer was utilized.

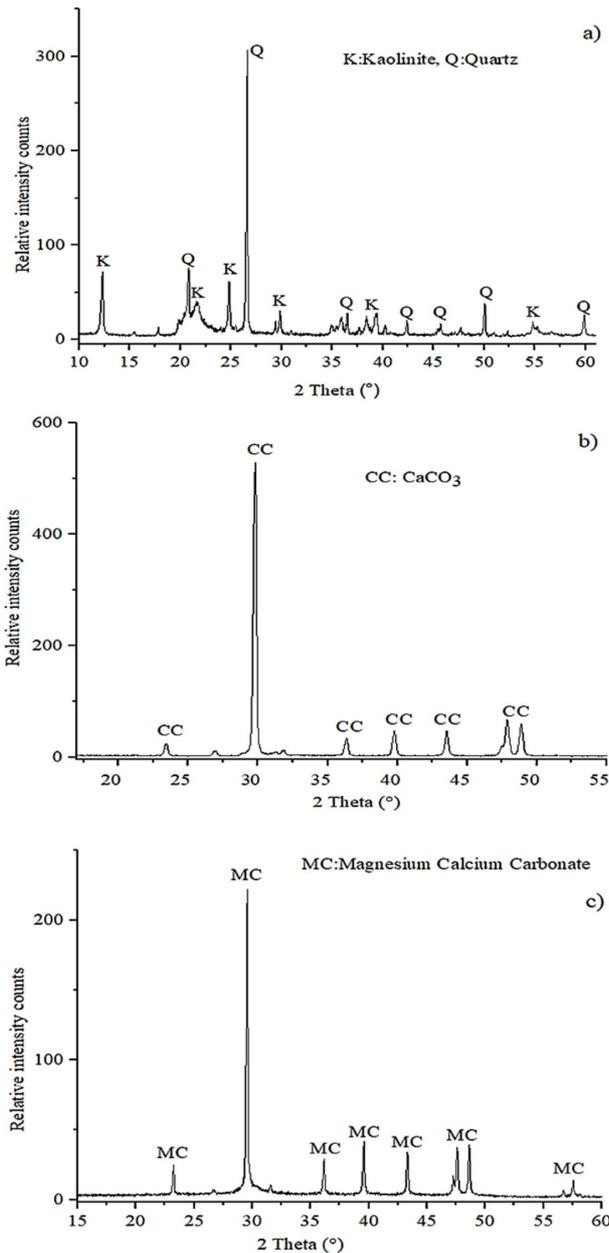
## Results and Discussion

### Characterization of the raw materials

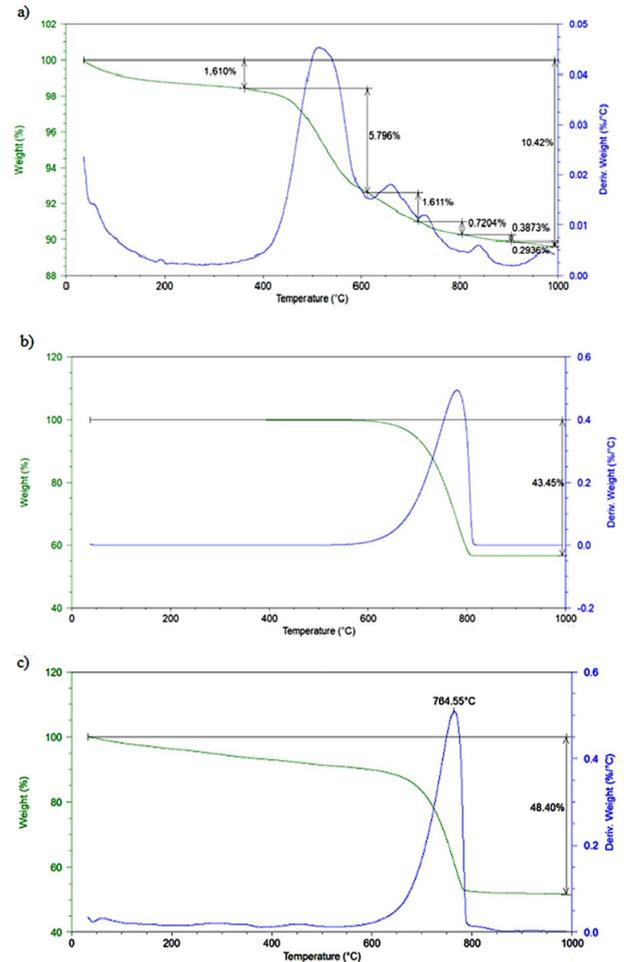
Table 1 shows the chemical composition of raw materials. The loss on ignition values of clay, calcite

**Table 1.** Chemical composition of the raw materials (% by weight)

Raw materials	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	LOI
Clay	63.32	18.77	1.00	0.28	2.20	0.20	0.32	0.35	9.60
Calcite	0.81	0.21	55.48	0.63	0.05	0.02	0.08	0.01	42.67
PFC waste	0.70	0.45	45.84	3.36	1.64	0.01	0.25	0.02	46.0

**Fig. 1.** XRD phase analysis of the raw materials: clay (a), calcite (b) and PFC waste (c).

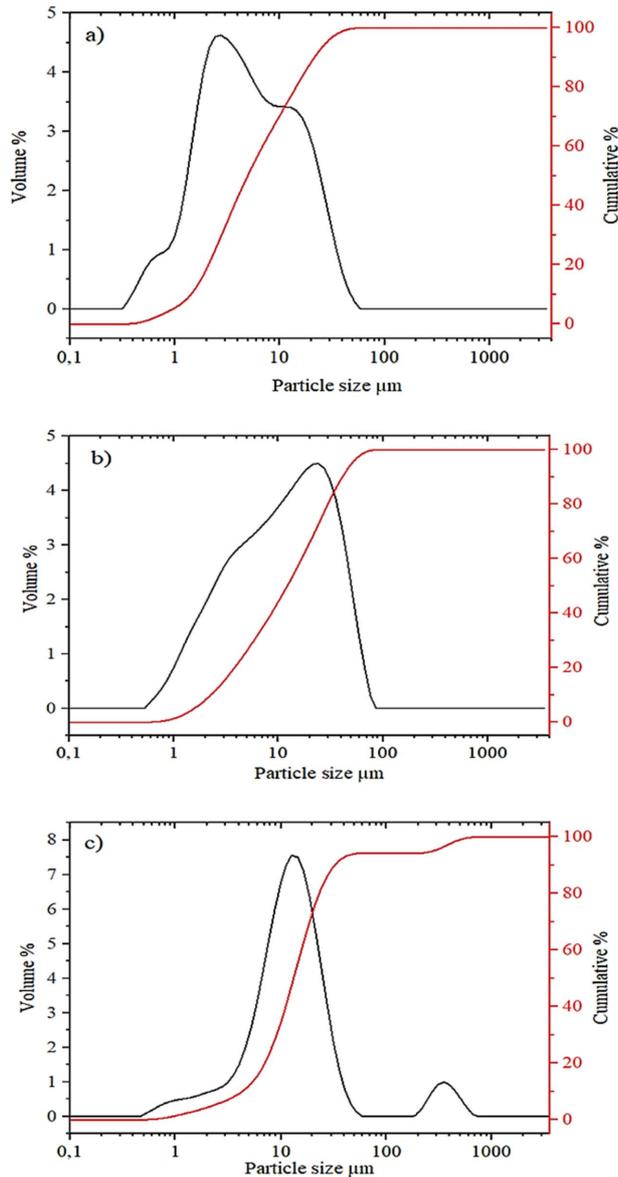
and PFC waste were measured as 9.60, 42.67 and 46.0% by weight, respectively. The clay raw material mainly includes about 63.3% SiO<sub>2</sub> and 18.8% Al<sub>2</sub>O<sub>3</sub>. It contains also minor constituents such as 2.2% Na<sub>2</sub>O and 1% CaO. Purity of the commercial calcite is quite high. The content of PFC waste has mainly calcium oxide content as well as minor amounts of the oxides

**Fig. 2.** TG analysis of the raw materials: (a) clay, (b) calcite and (c) PFC waste.

of magnesium and sodium.

XRD analysis was performed to identify the mineral phases found in raw materials. Because these phase definitions are important to identify certain phases that are likely to occur after firing the ceramic compositions. Fig. 1 shows the XRD patterns of clay, calcite and PFC waste. The analysis result of clay raw material indicates that quartz (SiO<sub>2</sub>) was the major crystalline phase as well as kaolinite (Al<sub>2</sub>O<sub>3</sub>·2SiO<sub>2</sub>·2H<sub>2</sub>O). The main crystalline phase of calcite raw material is calcium carbonate (CaCO<sub>3</sub>), while PFC waste was observed as magnesium-calcium carbonate phase.

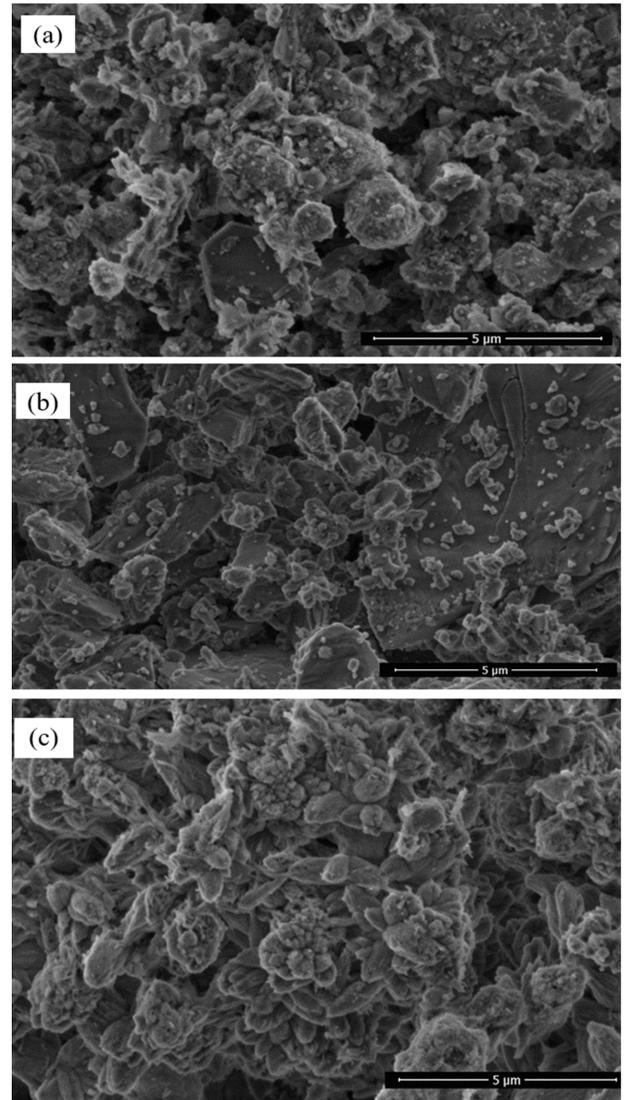
Fig. 2 indicates the TGA results of the raw materials. According to the TGA curves, total weight loss of clay raw material is 10.42% after firing at 1000 °C, the significant mass loss is due to the dehydroxylation of



**Fig. 3.** Particle size analysis of the raw materials: clay (a), calcite (b) and PFC waste (c).

kaolinite between 400 °C and 600 °C. For the calcite raw material, TGA analysis showed that there is only an endothermic reaction (total weight loss of 43.45%) in the range of 600 to 800 °C, which may be due to the thermal decomposition of  $\text{CaCO}_3$  into calcium oxide and carbon dioxide. In PFC waste, the total mass loss is 48.4%. The carbonate decomposition that occurs between 600 °C and 800 °C appears to be less (about 38.4%) compared to calcite raw material. In addition, it is observed that the removal of physical water and organic compounds that are likely to be present in the waste before the decomposition of carbonates is carried out at 100 °C and 300-500 °C, respectively.

Fig. 3 indicates the log-normal particle size distributions of the raw materials and the curve of cumulative distribution is also presented. The average



**Fig. 4.** SEM images of the raw materials: clay (a), calcite (b) and PFC waste (c).

particle sizes of clay, calcite and PFC appear to be about 5 μm, 10 μm and 15 μm, respectively. The clay particle sizes are less than 50 μm, while the calcite is less than 100 μm. PFC has a bi-modal particle size distribution curve; in addition to particles smaller than 60 μm, a small amount of agglomerated or coarse particles (<600 μm) are also present. It is known that particle size fineness is an important parameter for the rapid reaction of powder mixtures during firing process.

Fig. 4 shows the particle morphology of raw materials. It has been observed that the raw materials have some agglomerated powders composed of fine primary particles less than 5 μm. The SEM images confirm the results of particle size analysis. The particle morphology of PFC wastes appears to differ from the calcite raw material. It is also clear that calcite particles have sharper edges than that of PFC waste.

#### Characterization of the fired samples

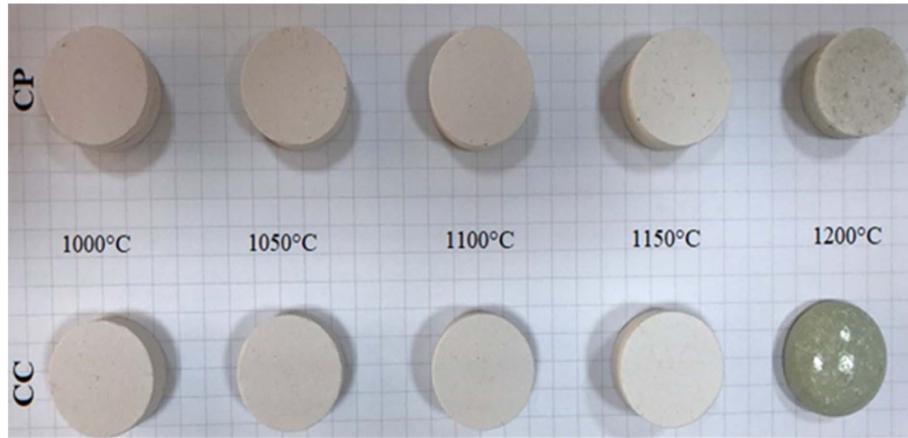


Fig. 5. Fired ceramic samples.

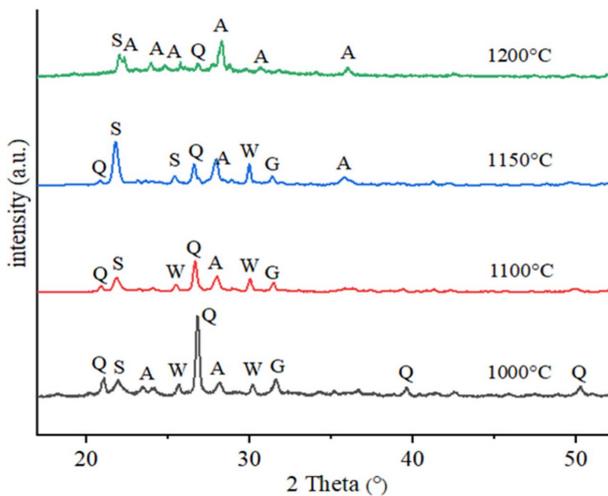


Fig. 6. Phase analysis of CC samples fired in different temperatures (Q: Quartz-low, S: Cristobalite, W: Wollastonite, A: Anorthite, G: Gehlenite).

The physical, mechanical and thermal properties of the fired samples were characterized. Fig. 5 shows the photograph of pellet samples fired at different temperatures. From the image of the fired samples, it can be seen that the firing behavior of the two different calcium carbonates additive admixtures is different at the applied temperatures.

Figs. 6 and 7 present XRD spectra acquired from CC (30% calcite + 70% clay) and CP (30% PFC + 70% clay) samples fired at 1000–1200 °C, respectively. The XRD patterns from CC and CP samples show the characteristic peaks of quartz as major phase at low firing temperature of 1000 °C. Also, in the both composition fired at 1000 °C, it was observed that the calcium alumina silicate (CAS) crystalline phases such as anorthite and gehlenite started to form. Gehlenite and wollastonite peaks appeared in the samples fired at 1000 and 1100 °C, which completely transformed to anorthite at 1200 °C. At high firing temperatures, especially at 1200 °C, it was observed that peaks of

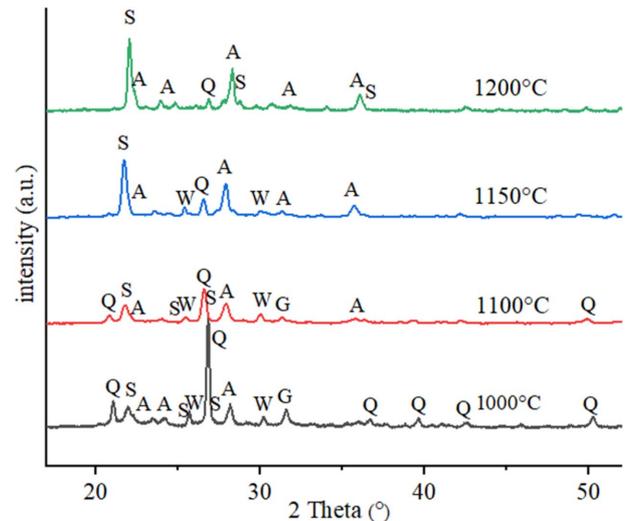


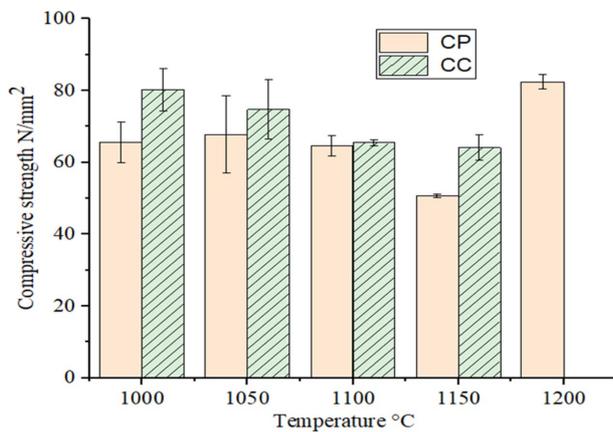
Fig. 7. Phase analysis of CP samples fired in different temperatures (Q: Quartz-low, S: Cristobalite, W: Wollastonite, A: Anorthite, G: Gehlenite).

quartz have decreased while characteristic peaks of anorthite and cristobalite have increased. At 1200 °C, in the CC composition samples, the major crystalline phase is anorthite as well as glassy phase. Due to the low percentage of CaO in the CP composition, the main crystalline phase is cristobalite, and followed by anorthite crystals. Gehlenite and wollastonite phase peaks were dissappeared at 1200 °C. The formation of the anorthite phase in CP sample is more pronounced than CC sample at 1150 °C. While the CC sample began to partially melt at 1200 °C, the anorthite crystals became evident with the glassy phase and the silica in the melt partially dissolved and decreased.

Physical properties of samples such as linear firing shrinkage, apparent porosity, bulk density were determined from average of three samples for each composition and firing temperature. Table 2 gives linear shrinkage of the samples versus temperature. It was observed that the linear firing shrinkage (LFS) values of the samples partially decreased from 1000 to

**Table 2.** Physical properties of CP and CC samples fired at different temperatures (LFS: linear firing shrinkage, WA: water absorption, AP: apparent porosity, BD: bulk density)

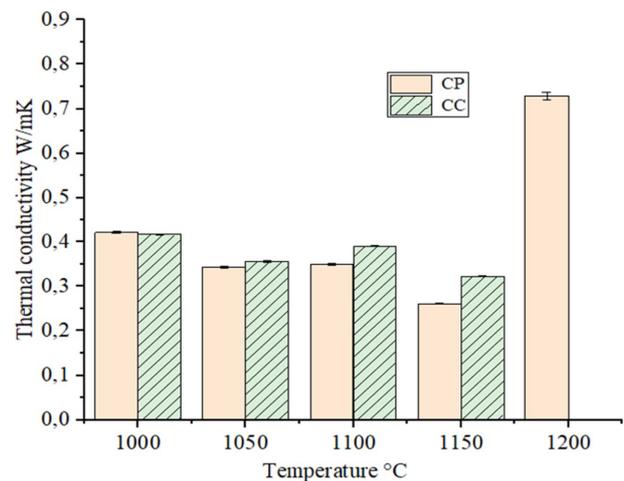
Temp.	Sample	LFS, %	WA, %	AP, %	BD, g/cm <sup>3</sup>
1000 °C	CP	1.8±0.1	28.4±0.5	42.4±0.6	1.49±0.01
	CC	2.2±0.1	24.6±1.2	38.9±1.6	1.58±0.02
1050 °C	CP	1.6±0.1	29.3±0.3	43.6±0.3	1.49±0.01
	CC	1.7±0.1	24.9±1	38.7±1.6	1.55±0.01
1100 °C	CP	1.5±0.1	29.9±0.7	43.6±1	1.46±0.01
	CC	1.3±0.1	27.9±0.4	42.3±0.3	1.52±0.02
1150 °C	CP	1.8±0.1	29.1±0.5	42.8±0.6	1.47±0.01
	CC	1.7±0.1	24.4±0.6	37.4±0.9	1.53±0.01
1200 °C	CP	10.4±0.6	4.8±0.2	9.7±0.2	2.04±0.01
	CC	deformed	0.1±0.01	0.2±0.01	2.33±0.01

**Fig. 8.** Compressive strengths of CP and CC samples fired at different temperatures.

1100 °C. The reason for this decrease may be the result of the quartz crystals dissolving and thus the increasing of the CAS crystal phases. The CC and CP samples tend to have larger shrinkage value from 1150 to 1200 °C, and also show a considerable liquid phase at 1200 °C.

While the apparent porosity ratios decreased, the bulk densities of the fired samples increased with rising to 1200 °C. Because of partial vitrification and pore closure, the CC and CP samples indicated a rising in density values at 1200 °C. It was observed that the CP samples demonstrated more stable behavior according to the CC samples. The apparent porosity values of CP samples are partially higher than that of CC samples. The CC samples, which contain anorthite as major phase, vitrified at 1200 °C and their apparent porosity values reduced to almost zero.

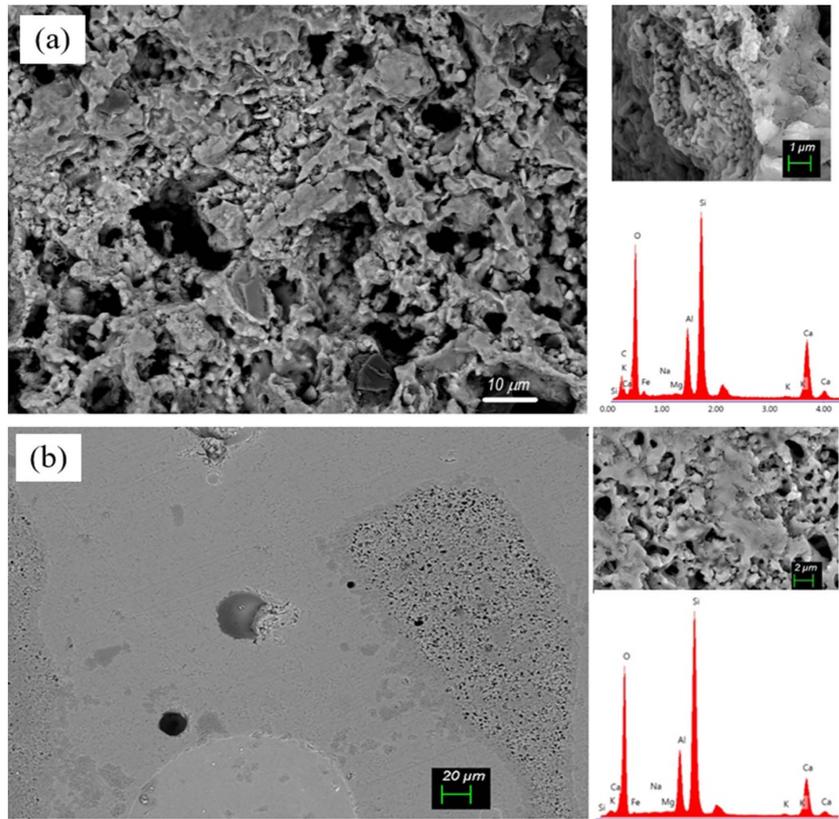
The compressive strength results of the CP and CC samples which were fired from 1000 to 1150 °C are shown in Fig. 8. The results are ranged from 63 to 50.7 MPa and 80.3 to 64 MPa, respectively. By increasing of firing temperature up to 1150 °C of the both samples, the compressive strength values partially decreased. In the samples fired at 1200 °C, the strength of CP sample showed an increase trend to about 82 MPa, however,

**Fig. 9.** Thermal conductivities of CP and CC samples fired at different temperatures.

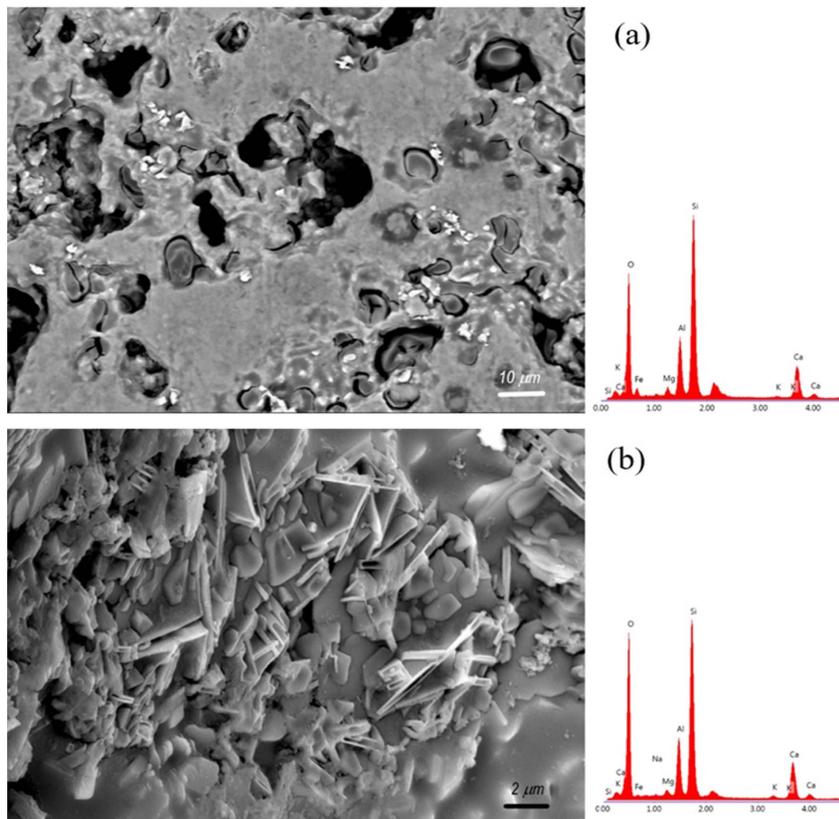
the strength of CC sample was not measured because of melting of the samples.

Thermal conductivity values for each composition and firing temperature were gotten from ten measurements of one sample. As seen from Fig. 9, the thermal conductivity values of the CP and CC samples ranged from 0.42 to 0.8 W/mK. By increasing the firing temperature up to 1150 °C of the both samples, the thermal conductivity values showed a decreasing trend. Thermal conductivities of the CP samples decreased from 0.42 to 0.25 W/mK. These results are promising for use as thermal insulation bricks of the ceramic samples produced from sugar processing filter cake waste. In the samples fired at 1200 °C, the thermal conductivity values of CP sample showed a rapid increase to about 0.73 W/mK. This case is a result of partial vitrification and densification. The thermal conductivity values of CC sample was not measured because of melting the samples at 1200 °C.

Figs. 10 and 11 show SEM micrographs of the etched surfaces of the ceramic samples. In the figures, it was observed that the CC and CP samples fired at 1150 °C have a porous structure. Also, cristobalite and



**Fig. 10.** SEM micrograph and EDS patterns of the CC samples: (a) fired at 1150 °C, (b) fired at 1200 °C.



**Fig. 11.** SEM micrograph and EDS patterns of the CP samples: (a) fired at 1150 °C, (b) fired at 1200 °C.

quartz particles in the ceramic matrix are present as spheroidal crystals. The ceramic matrix is formed from anorthite crystals which appear as plate-like and sub-micronfine particles. In the CP sample fired at 1150 °C, it is observed that the silica particles embedded in the anorthite phase have voids around the particles due to the thermal expansion difference from the ceramic matrix. In the CC sample fired at 1200 °C, a dense structure as well as local porous regions are observed in the structure. In the CP sample fired at 1200 °C, plate-like anorthite crystals are observed in the structure. According to EDS results taken from the SEM images, the ceramic bodies have similar Ca-Al-Si-O content.

### Conclusions

The differences in CaO content between calcite and sugar filter cake (PFC) waste, as well as organic content of PFC, caused differences in physical, mechanical and microstructural properties. As the result of higher loss of ignition of PFC waste, CP (Clay + PFC waste) ceramics have higher porosity percentages. As a function of high porosity, the bulk density, compressive strength and thermal conductivity values of CP ceramics are lower than that of CC (Clay + Calcite) ceramics. However, the dimensional stability of CP samples at higher temperatures was found to be better than CC samples. Depending on the firing temperature, the compressive strengths and apparent porosity of fired samples produced from sugar filter cake waste ranged from 50.7 to 80.3 MPa and from 9.7% to 43.6%, respectively.

Phase analysis results show that due to lack of sufficient CaO content in CP ceramics, the highest amount of cristobalite phase and secondly anorthite are formed at 1200 °C. In contrast, CC ceramics at the same temperature showed the strongest anorthite phase as well as the glassy phase. In microstructural investigations, it was observed that porosity decreased and glassy phase increased by dissolving silica with increasing temperature of samples. In addition, calcium alumina silicate crystals appeared embedded in the glassy matrix of CC samples. Sugar filter cake waste can be used instead of calcite in the production of CAS ceramics, but due to the difference in CaO content, the chemical composition of the desired phases in the ceramic body should be considered. This study is important to show that this waste can be used in the production of porous anorthite based ceramics. It is envisaged that in the next process waste can be used as an alternative raw material to produce thermal insulating materials with higher porosity.

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