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RESEARCH ARTICLE / ARAŞTIRMA MAKALESI

Production and Characterization of Porous Anorthite Ceramics Using Sugar Process Solid Waste, Chamotte and Coal Powder Additives

Gözenekli Anortit Seramiklerin Şeker Proses Katı Atığı, Şamot ve Kömür Tozu Katkıları Kullanılarak Üretimi ve Karakterizasyonu

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Abstract

Press filter cake is a solid waste produced during sugar production process and includes carbonates and organic components. In this study, coal powder in different percentages (from 10 wt.% to 50 wt.%) was mixed with 35 wt.% press filter cake and 65 wt.% chamotte composition to produce porous anorthite ceramic products with different apparent porosity percentages. The anorthite phase was observed as the dominant crystalline phase in all fired samples. The firing shrinkage values sharply increased after adding higher than 40 wt.% coal powder to composition. In addition, apparent porosity values were changed from 49% to 70%. Compressive strength values reduced from 18 MPa to 1.73 MPa while percentages of ground coal were increasing. Microstructural analysis also showed that the porosity values of the sintered samples showed important changes depending on the amount of coal additive. The thermal conductivity values showed a reduction from 0.113 W/mK to 0.064 W/mK while increasing the percentage of ground coal. Micro-computed tomography (Micro CT) analysis was performed for 40wt. % coal powder added composition and the total porosity value was found as 60.2%.

Keywords: *Porous ceramic, Anorthite, Press filter cake waste, Coal powder*

Öz

Pres filtre keki, şeker üretim sürecinde ortaya çıkan, karbonatlar ve organik bileşenler içeren katı bir atıktır. Bu çalışmada farklı görünür gözeneklilik yüzdelerine sahip, gözenekli anortit seramikler üretmek için, ağırlıkça %35 pres filtre keki ve ağırlıkça %65 şamot bileşimi ile farklı yüzdelerde kömür tozu (ağırlıkça %10'dan %50'ye kadar) karıştırılmıştır. Pişirilen tüm örneklerde anortit fazı baskın kristal faz olarak gözlenmiştir. Bileşime ağırlıkça %40'tan fazla kömür tozu ilave edilmesiyle, pişirme büzülme değerleri hızlı bir şekilde artmıştır. Ayrıca görünür gözeneklilik değerleri %49'dan %70'e çıkmıştır. Kömür tozu yüzdeleri artarken basma dayanım değerleri 18MPa'dan 1,73MPa'a düşmüştür. Mikroyapı analizleri de sinterlenmiş numunelerin gözeneklilik değerlerinin kömür katkı miktarına bağlı olarak önemli ölçüde değiştiğini göstermiştir. Bileşimdeki kömür tozu yüzdesi arttırılırken, termal iletkenlik değerleri, 0,113 W/mK'den 0,064 W/mK'e düşme göstermiştir. Ağırlıkça %40 kömür tozu katkılı bileşim için Mikro bilgisayarlı tomografi (Micro CT) analizi yapılmış ve toplam gözeneklilik değeri %60,2 olarak bulunmuştur.

Anahtar Kelimeler: *Gözenekli seramik, Anortit, Pres filtre keki atığı, Kömür tozu*

1. Introduction

Anorthite porous ceramics have an area of usage in the insulation field in favor of low thermal conductivity, high purity, and high strength [1]. In recent years, many studies have been published in the literature on the processing of porous anorthite ceramics using both mineral raw materials and waste materials [1-19]. In a study carried out using mineral raw materials, porous gehlenite-anorthite-based ceramics were produced by using calcined kaolin and 28 wt.% calcite and firing at 1250°C for 1h. In the structure, it was measured to have a high porosity of 54% [2]. Another study related to the production and characterization of porous anorthite ceramic based on mineral materials found that the resulting bodies showed apparent porosity values between 28 and 32% [3]. The utilization of various wastes in the production of insulation refractory is a remarkable method of recycling for the annihilation of several wastes. A significant quantity of waste is used to produce insulation refractory. Rice husk, fly ash, fired refractory grog, and rice husk ash are current wastes. Several specimens are worked on compositions based on partial and full replacement of clay [4]. Calcium carbonate and fly ash cenosphere were put to use in fabricating high apparent porosity ceramics. High apparent porosity was investigated with the addition of calcite. Calcium carbonate constituted the apparent porosity in two ways: the first was that CO_2 emission from $CaCO_3$ decomposition induced the formation of pores in ceramics; the second was that CaO from calcite decomposition formed into anorthite with fly ash cenosphere. The apparent porosity of a ceramic composition with 30 wt.% $CaCO_3$ fired at 1250°C achieved 59.25% [5]. Through direct foaming and slip casting, a mixture of gypsum and fly ash constituted porous anorthite ceramics with low thermal conductivity. While 10%

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gypsum content was added, anorthite came in the main phase with the least amount of impurity. In addition, with sodium hexametaphosphate as dispersant and using a two-step foaming process, the highest open porosity of up to 94% and the lowest thermal conductivity of 0.042 W/mK were achieved [6]. In research, porous anorthite ceramics were produced from metakaolin, talc, fly ash, and red mud. A sample containing 30 wt.% red mud and 40 wt.% fly ash fired at 1150°C had 38% apparent porosity and 42 MPa compressive strength. Anorthite was detected as the main phase, and the minor phases were mullite, α-quartz, and diopside ferrian. Abundant open pores and closed pores were examined in SEM images, which indicated high porosity and low heat conductivity. In addition, mullite grains with rod-like shapes were present in the porous ceramics. These grains prevented the propagation of cracks in intergranular fractures to raise the compressive strength [7]. In another study, steel slag and high-alumina fly ash microbeads were mixed to produce feldspar-porous ceramics over high-temperature solid-phase sintering by adding pulverulent coal as a pore-forming agent.The ratio of fly ash microbeads to steel slag was chosen at 6:4, and the pore-forming material content was 10%. Porous ceramics with a porosity of 49.21% and a flexural strength of 12.88 MPa were obtained by firing at 1170°C for 2 hours. The main phase of porous ceramics was anorthite [8]. For the production of porous and lightweight anorthite-based insulating ceramics, mixtures of clay, recycled paper processing waste, and sawdust as pore-forming materials were also studied. Recycled paper processing waste consists of organic and inorganic contents. The reaction between calcium oxide from paper processing waste and aluminum silicate from clays resulted in anorthite in the minor secondary phase. Sawdust-added mixtures with anorthite composition promoted the porosity of the samples. It was found that the bulk densities ranged from 1.12 to 0.64 g/cm^3, and the thermal conductivities varied from 0.25 to 0.13 W/mK depending on their porosity content [9]. Another study is about the use of three different clays, such as aluminum silicate, clay with alkalis, and fireclay, in the manufacturing of anorthite-based lightweight insulating firebricks. All samples were mixed with 30–40 wt.% paper processing residues, and they were fired at 1200–1400°C. Sintered samples consisted of anorthite as the major phase and mullite or gehlenite phases examined as minor secondary phases in some mixtures [10]. Press filter cake (PFC) is a sugar industry waste that consists of magnesium calcium carbonate, and organic substances. This waste is generally used for fertilization of sugarcane fields and other fields of agriculture [11, 12]. PFC wastes were used for the production of anorthite-based ceramics as CaO sources in the anorthite compositions [13, 16]. Research has shown that it is a useful raw material for the production of anorthite/cordierite and anorthite/alumina composites [17, 18]. In our previous study, it was determined that a 35% press filter cake (PFC) and 65% chamotte (PCH-35) composition, which was fired at 1300°C for 2 hours, showed 49.6% apparent porosity for porous anorthite ceramic [19]. In this study, it was aimed to increase the porosity ratio of the PCH-35 composition by adding coal powder as the pore-forming material in different percentages (10%–50% by weight) for insulation field usage.

2. Materials and Methods

2.1. Characterization of raw materials

PFC was obtained from Eskişehir Sugar Factory, where 50.000 tons of sugar beet filter cake waste are sorted per year. Chamotte was chosen as the alumina silicate and it was supplied from Kaolin Industrial Minerals Co. Coal was procured from an industrial company located in Soma, Turkey. Coal was ground for 5 minutes at 700 rpm by using a disc mill. Ground coal was used as the pore-forming material in the PCH-35 composition [19]. In the beginning, the crystalline phase, chemical composition, thermal behavior, and particle size distribution of raw materials were determined. A X-ray fluorescence spectrometer (EDXRF Spectro IQ II) was used for determining the chemical composition. Phase analysis of the powders was done by an X-ray diffractometer (Bruker D2 Phaser) with a Cu tube of 1.54184 Å at a voltage of 30 kV. Particle size distributions of raw materials were measured by the Malvern Mastersizer 3000. Thermogravimetric analysis (TGA) of the raw materials was carried out under a nitrogen atmosphere at a heating rate of 10°C/min using the TA Instruments TGA-SDT Q600 equipment.

2.2. Preparation of samples with different percentage coal powder addition

PCH-35 composition was prepared and mixed with ground coal in different weight percentages (10% to 50%) for the production of porous anorthite ceramic products. Firstly, the slurry of PCH-35 composition was prepared and dried at 100°C for 24 hours. Then it was crushed to obtain it in powder form. The powder mixture was mixed separately with different percentages (10% to 50%) of ground coal by weight. Powders of mixtures were pressed at 25 MPa pressure, and samples were formed with a 20 mm diameter. Samples were dried at 100°C for 24 hours before sintering.

2.3. Characterization of fired samples

The pressed samples were sintered at 1300°C for 2 hours. They were characterized for present crystalline phases (XRD), physical properties (apparent porosity, bulk density water absorption), thermal conductivity, mechanical properties (compressive strength) and microstructural properties. The physical properties of sintered samples were determined by the liquid displacement method (ASTM C20). Compressive strength was measured by a Shimadzu 100 kN testing machine at a crosshead speed of 0.5 mm/min. Thermal conductivity was studied with C-Therm equipment. The microstructural properties of fired samples were determined by SEM-Quanta 250 FEG. A micro-CT test was applied to the sample that had the highest apparent porosity value.

3. Results and Discussion

3.1. Characterization of raw materials

The chemical composition of chamotte includes about 57.9% SiO_2, 38.8% Al_2O_3, 1.1% Fe_2O_3, 0.9% K_2O, 0.3% Na_2O compounds, and 0.3% loss on ignition. The chemical analysis of PFC waste contains mainly 44.25% CaO, 3.24% MgO, 3.28% Na_2O, 0.33% Al_2O_3, and 48% loss on ignition. In figure 1, the XRD pattern of chamotte indicates the presence of mainly crystalline phases like mullite, quartz, and cristobalite as well as an amorphous phase. The PFC waste has a mainly magnesium-calcium carbonate phase. Coal was used as a pore-forming material in the ceramic body. Carbon, FeO, quartz, and sodium aluminum silicate phases were determined in XRD analysis. Coal was fired for one hour at 1000 °C, and coal ash was obtained. After firing, quartz, cristobalite, sodium aluminum silicate, and FeO phases were identified in the sample (Figure1(d)).

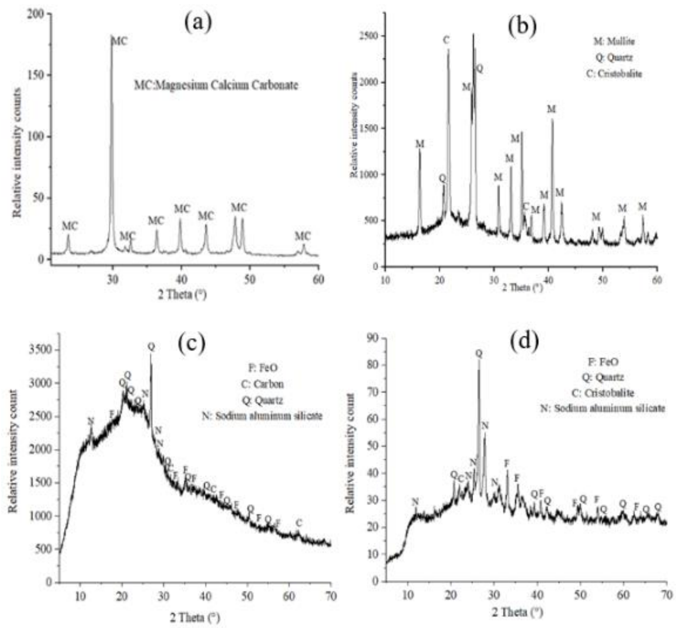

Figure 1. Phase analysis; PFC (a), chamotte (b), coal (c), coal fired at 1000°C (d)

PFC shows a bi-modal particle size distribution curve, and D_v (10), D_v (50), D_v (90) values of PFC are determined as: 3.83µm, 13.6µm and 125µm, respectively. (Figure 2a). The chamotte particle size was measured less than 139µm and the average particle size was 38 µm (Fig 2b). D_v (10), D_v (50), D_v (90) values of the used coal powder are found as follows: 6.19µm, 51.3µm and 245µm, respectively.

Figure 3 shows the TGA results for the raw materials. According to the TGA curve, the total weight loss of PFC is 52.78%. It also indicates that 34.73% carbonate decomposition occurs between 600°C and 800°C. The removal of organic compounds in the waste happens at 300–500°C, and the weight loss appears to be 18%. The TGA curve of coal demonstrates 58.83% weight loss (Figure 3b).

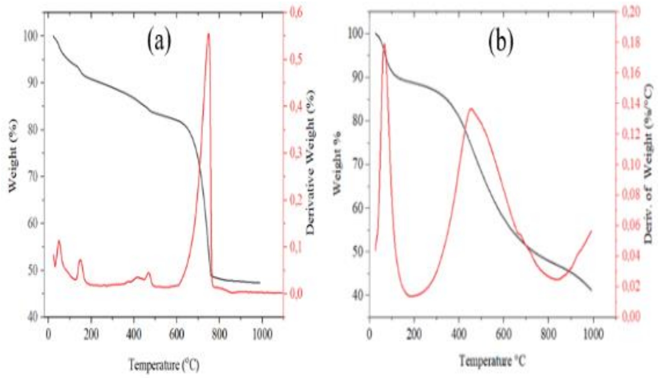

Figure 3. Thermogravimetric analysis; PFC (a) and coal (b)

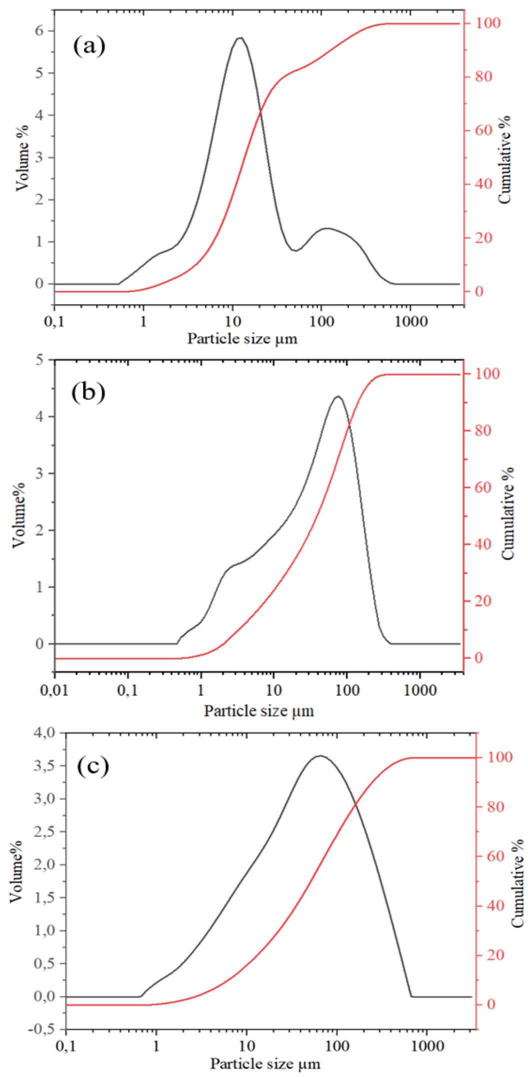

Figure 2. Particle size distribution of the raw materials; PFC (a), chamotte (b) and coal (c)

3.2. Characterization of fired samples

Figure 4 shows the image of the fired samples. Figure 5 indicates the XRD phase analysis of sintered samples. The basic crystalline phase of each fired sample is anorthite, and there is no considerable difference among all the compositions.

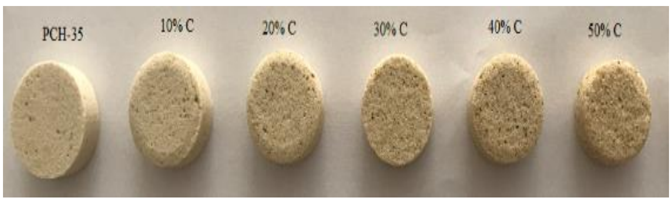

Figure 4. Fired samples

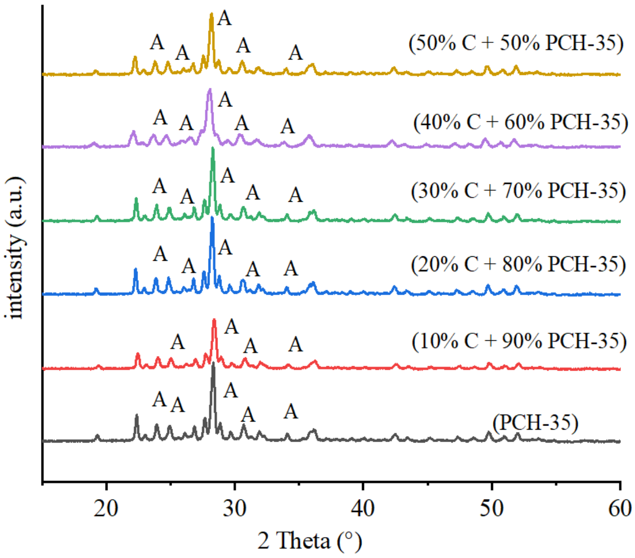

Figure 5. XRD phase analysis of fired samples

The physical properties and compression strength values of the samples were determined using three samples for each composition. The physical properties of the samples sintered at 1300°C are listed in Table 1.

When the percentage of coal increased, firing shrinkage, water absorption, and apparent porosity percentage values increased. On the other hand, bulk density decreased from 1.38 to 0.81 g/cm³. The highest linear firing shrinkage percentage was calculated at 50% ground coal content. This situation is not preferable to fabricate because high firing shrinkage may cause firing cracks in the ceramic body. The fired sample of 40% C + 60% PCH-35 composition showed under 10% firing shrinkage. Also, the apparent porosity percentage was 67%, and the bulk density was calculated at 0.88 g/cm³. According to the results of the physical tests, the 40% C + 60% PCH-35 composition was analyzed by micro-CT. In Figure 6, dark blue areas show the structure of the fired sample. Total porosity was calculated at 60.17%, and the closed porosity percentage was 0.03%.

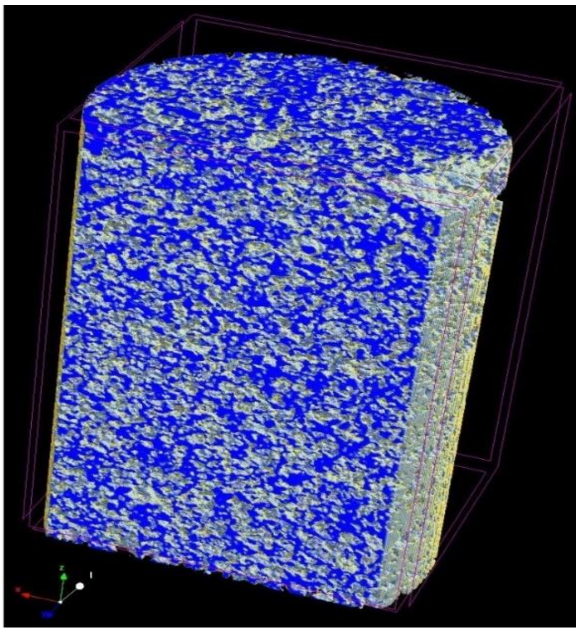

Figure 6. Micro-CT image of fired sample with 40%C + 60% PCH-35 composition

Dilatometric analysis was done for PCH-35, 20% coal-added PCH-35, and 40% coal-added PCH-35 samples fired at 1300°C. They were analyzed for their thermal expansion percentages. The shrinkage (–) and expansion (+) behaviors of fired samples are shown in Figure 7. All samples began to shrink after about 1150°C. The total thermal expansion of the samples up to 1100°C was about 0.05%. The final thermal expansion values of whole samples at 1100°C were measured at 0.05%, and this value showed the stability of fired samples up to 1100°C. The shrinkage measurements of reheated samples with 0, 20%, and 40% coal additive were determined to be 0.05%, 0.2%, and 0.3%, respectively, at 500°C during the cooling process. All samples were thermally and dimensionally stable at temperatures up to 1100°C. The samples that were produced in this study can be safely used up to 1100°C.

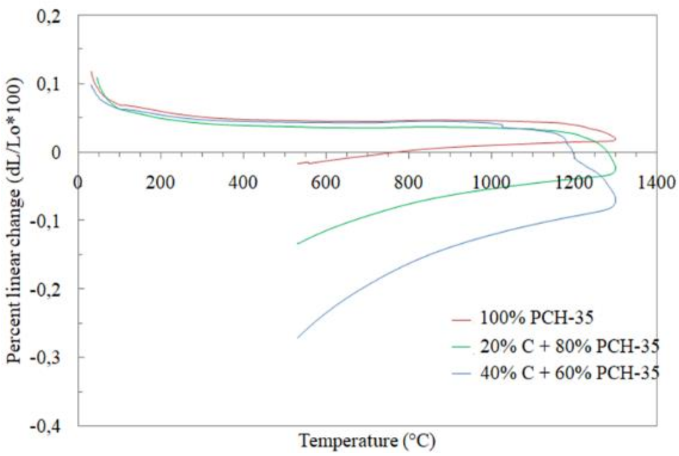

Figure 7. Percent linear changes of the samples during firing

In addition, the initial and final lengths of the samples used in dilatometric analysis were measured. The change in length values was calculated and showed shrinkage of 0.06%, 0.9%, and 1.3% for PCH-35, 20% coal added to PCH-35, and 40% coal added to PCH-35, respectively. As a consequence of the reduction in bulk density with an increase in coal percentage, the compressive strength values of fired samples decreased from 18.78 to 1.73 N/mm², as seen in Figure 8. Wu et al., stated that Synthesized anorthite grains and replenishing the intergranular spaces with liquid phases are effective for high-strength results [20]. Also, polyurethane sponge was used as a template to prepare porous ceramic, and metallurgical steel slag and kaolin were used as raw materials. The compressive strength was measured at 1.5 MPa, and the porosity was 85.3% [21].

The thermal conductivity values for each composition were measured. As seen in Figure 9, the thermal conductivity values of samples fired at 1300°C range from 0.11 to 0.06 W/mK. By increasing the percentage of coal powder, the thermal conductivity values show a decreasing trend. The development of an amorphous phase in the grain boundaries, a large number of nano-regions in anorthite grains, and high porosity accomplish low thermal conductivity in the ceramic body [20].

the fired samples, and a porous structure remained while increasing the content of coal powder.

Table 1. Physical properties of the fired samples.

Composition	Firing shrinkage %	Water absorption%	Apparent porosity %	Bulk density g/cm³
PCH-35	-3.26±0.01	35.85±0.4	49.30±0.3	1.38±0.01
10% C + 90% PCH-35	-1.07±0.03	46.17±0.5	55.57±0.2	1.20±0.01
20% C + 80% PCH-35	2.52±0.09	57.28±0.03	60.83±0.05	1.06±0.01
30% C + 70% PCH-35	5.35±0.2	68.20±0.5	64.86±0.2	0.95±0.01
40% C + 60% PCH-35	7.5±0.2	76.81±0.2	67.58±0.2	0.88±0.01
50% C + 50% PCH-35	10.71±0.1	86.50±0.5	70.19±0.04	0.81±0.01

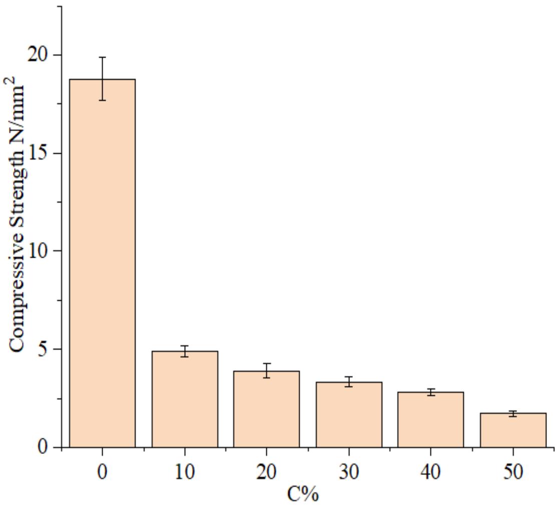

Figure 8. Compressive strength values of the samples with coal addition

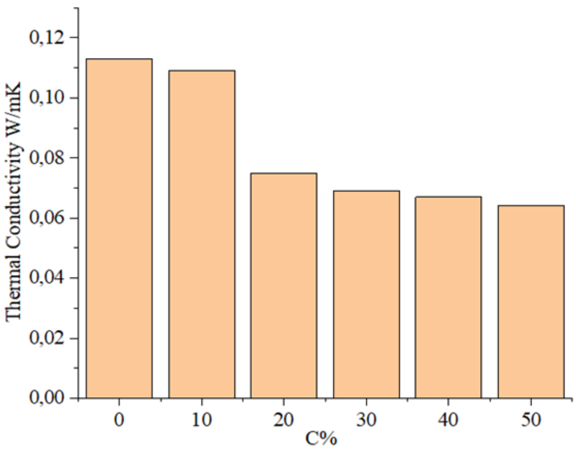

Figure 9. Thermal conductivity values of samples

The microstructural properties of fired samples were determined by SEM-Quanta 250 FEG. Samples were broken, and microstructures were investigated on the broken surfaces. Many open and closed pores were examined using SEM images. This situation ensured low thermal conductivity and high porosity properties in the final product [7]. Figure 10 shows the anorthite crystals formed in the fired ceramic body as well as the effects of the structures formed by the coal dust used as a pore-forming material in production. Anorthite crystals have been observed in the fired samples, and a porous structure remained while increasing the content of coal powder.

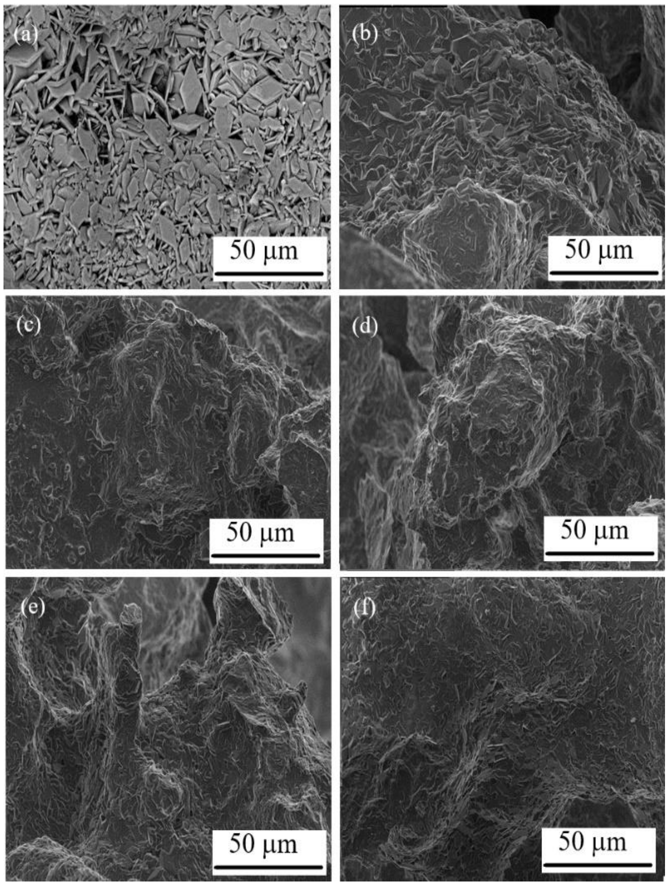

Figure 10. SEM images taken at 2500X magnification of fired samples with different compositions: PCH-35 (a), 10% C + 90% PCH-35 (b), 20% C + 80% PCH-35 (c), 30% C + 70% PCH-35 (d), 40% C + 60% PCH-35 (e), 50% C + 50% PCH-35 (f)

4. Conclusions

PCH-35 (35% press filter cake, 65% chamotte) composition was mixed with ground coal in different percentages (from 10% to 50%) for the production of porous anorthite ceramic in different apparent porosity percentages. The whole fired sample showed a major anorthite phase. Firing shrinkage values were run up after adding more than 40% coal to the PCH-35 composition. 50% of the coal added to the PCH-35 showed 10.7% firing shrinkage. In addition, cracks were observed on the surfaces of 50% of the coal added to the PCH-35 samples. Apparent porosity percentages also increased from 49.3% to 70.19% while increasing the amount of coal powder because of the combustion of coal powder. In addition, bulk density was reduced from 1.38 to 0.81 g/cm³. As a result of decreasing bulk density, a reduction was observed in compressive strength values from 18.78 to 1.73 N/mm². Thermal conductivity values also supported the apparent porosity results. Thermal conductivity results are reduced with increasing coal powder percentages. According to dilatometric analysis, the final thermal expansion values of whole samples at 1100°C were measured 0.05%, and this value indicated that fired samples were preserving their stability up to 1100°C. The shrinkage measurements of reheated samples with 0, 20%, and 40% coal additive were determined 0.05%, 0.2%, and 0.3% at 500°C during the cooling process. MicroCT analysis of the 40% coal-added PCH-35 composition showed 60.17% total porosity. The microstructural properties of fired samples determined the effect of coal percentages on the structure. A porous structure was procured while increasing the content of coal powder. Fine anorthite crystals were observed in the fired samples.

According to all test results obtained, the PCH-35 sample with 40% coal addition exhibited the best result in terms of thermal insulation. It is thermally and dimensionally stable at firing temperatures up to 1100°C. The thermal conductivity value of the samples with this composition was measured as 0.067 W/mK, and the bulk density and apparent porosity values were 0.88 g/cm³ and 68%, respectively. When compared to previous studies in the literature, it is understood that the thermal conductivity values obtained in this study are lower. In a study, the thermal conductivity value of samples produced by adding 30% wood sawdust as a pore former to the paper waste/clay mixture was found to be 0.13 W/mK [9]. Additionally, the thermal conductivity value of the samples produced by adding 30% expanded polystyrene granules to the chamotte/eggshell waste mixture was found to be 0.11 W/mK [22]. In another study, the thermal conductivity value of samples produced by mixing 30% sawdust into the rice husk ash/diatomaceous earth mixture was measured as 0.09 W/mK [23]. As a result, it was concluded that sugar process solid waste, which is an industrial waste, and coal dust additives can be used as alternative calcium sources and pore formers in the production of anorthite bricks for thermal insulation purposes.

Ethics committee approval and conflict of interest statement

There is no need for an ethics committee approval in the current article. There is no conflict of interest with any person/institution in the current article.

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Author Contribution Statement

Vacide Selin Kaya: conceptualization, experimental design, data collection, conducting analysis, literature review, writing; Mücahit Sütçü: conceptualization, experimental design, supervision, conducting analysis, writing, critical review and editing; Emre Yalamaç: conceptualization, experimental design, conducting analysis, writing, critical review and editing; Onur Ertuğrul: conceptualization, experimental design, conducting analysis, writing, critical review and editing.

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