Fabrication of porous anorthite ceramics using eggshell waste as a calcium source and expanded polystyrene granules

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Highlights
- Utilization of eggshell waste and EPS granules in porous anorthite ceramic production
- Determination of optimum additive rates and production temperatures
- Investigation of the physical and mechanical properties of the ceramics produced

Graphical Abstract
Highly porous and lightweight ceramics were successfully produced from the admixtures of fireclay as a source of $\text{SiO}_2$ and $\text{Al}_2\text{O}_3$ and eggshell waste as a source of CaO in compositions suitable for anorthite production alongside EPS as a pore-forming material.

Aim
The aim of this study is to produce porous anorthite ceramics by using eggshell waste, an alternative calcium source, and fireclay and pore-forming expanded polystyrene.

Design & Methodology
Phase analysis, microstructural properties, thermal conductivity, density and compression strength values of anorthite samples produced according to the determined optimum additive ratios and sintering temperatures were characterized.

Originality
Although there are numerous studies on the production of porous anorthite ceramics using various raw materials, no study has yet been reported in the literature in which eggshell waste is used in the production of anorthite-based lightweight materials.

Findings
It was concluded that the eggshell waste could be used as a favorable alternative calcium source for the production of porous anorthite ceramics due to their organic and inorganic content.

Conclusion
The thermal properties of the produced anorthite ceramics decreased by 71.8%, as expected, with a 20% reduction in density. Due to the increasing amount of pores, a remarkable decrease of 54.7% in compressive strength occurred.

Declaration of Ethical Standards
The author(s) of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.
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Araştırma Makalesi / Research Article
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Abstract

Within the scope of present work, porous and lightweight anorthite ceramics by utilizing admixtures of eggshell waste, fireclay, and expanded polystyrene (EPS) addition has been produced. First, samples of mixtures prepared with different proportions of eggshell waste were fired at different temperatures in order to determine the appropriate anorthite composition. Later, in order to produce high porosity anorthite ceramics, different amounts of EPS are added to the most suitable composition determined and porous samples were produced by firing at the certain temperature. The high degree of porous anorthite ceramics from the admixtures in the range of 0 to 30 vol.% EPS addition were satisfactorily fabricated. The physical characteristics, compressive strength and thermal conductivity were examined. Thermal conductivity of the anorthite ceramics showed a decline from 0.39 W/m·K (1.00 g/cm³) to 0.11 W/m·K (0.78 g/cm³) in proportion to the decrease in density. It has been found from the results in which these porous materials produced by the reuse of eggshell waste are suitable for use in various applications requiring elevated temperatures.

Keywords: Anorthite, lightweight ceramics, eggshell waste, porosity, thermal conductivity.

Kalsiyum Kaynağı Olarak Yumurta Kabuğu Atığı ve Genleştirmiş Polistiren Granülleri Kullanarak Gözenekli Anortit Seramik Üretimi

Öz

Mevcut çalışma kapsamında yumurta kabuğu atığı, ateş tuğlası ve genleştirmiş polistiren (EPS) karışımını kullanarak gözenekli ve hafif anortit seramikler üretimi yapılmıştır. İlk olarak, farklı oranelarda yumurta kabuğu atığı ile hazırlanmış karışım numuneleri, uygun anortit bileşimini elde etmek için farklı sıcaklıklarda pişirilmiştir. Yüksek oranlarda gözenekli anortit seramikler üretmek için belirlenen en uygun bileşime, farklı miktarlarda EPS ilavesi yapılmış ve belirli sıcaklıklarda EPS ilavesi ile elde edilen karışımın değerleri, basınç dayanımı ve ısıl iletkenlik değeri incelenmiştir. Anortit seramiklerin termal iletkenliğini ölçmek için alı konulan sap nauyruları uygun olarak 0,39 W/m·K (1,00 g/cm³) ve 0,11 W/m·K (0,78 g/cm³) değerlerine sahiptitlesi göstermiştir. Alı konulan sap nauyruların, yüksek sıcaklıklarda pişirilmiş anortit seramiklerin, yüksek sıcaklıklarda pişirilmiş anortit seramiklerin termal iletkenliğini ölçmek için alı konulan sap nauyruları uygun olarak 0,39 W/m·K (1,00 g/cm³) ve 0,11 W/m·K (0,78 g/cm³) değerlerine sahiptitlesi göstermiştir. Elde edilen sonuçlar ışıkta, yumurta kabuğu atıklarını yeniden kullanılmamıştır. Yüksek sıcaklıklarda pişirilmiş anortit seramiklerin, yüksek sıcaklıklarda pişirilmiş anortit seramiklerin termal iletkenliğini ölçmek için alı konulan sap nauyruları uygun olarak 0,39 W/m·K (1,00 g/cm³) ve 0,11 W/m·K (0,78 g/cm³) değerlerine sahiptitlesi göstermiştir. Elde edilen sonuçlar ışıkta, yumurta kabuğu atıklarını yeniden kullanılmamıştır. Yüksek sıcaklıklarda pişirilmiş anortit seramiklerin, yüksek sıcaklıklarda pişirilmiş anortit seramiklerin termal iletkenliğini ölçmek için alı konulan sap nauyruları uygun olarak 0,39 W/m·K (1,00 g/cm³) ve 0,11 W/m·K (0,78 g/cm³) değerlerine sahiptitlesi göstermiştir. Elde edilen sonuçlar ışıkta, yumurta kabuğu atıklarını yeniden kullanılmamıştır. Yüksek sıcaklıklarda pişirilmiş anortit seramiklerin, yüksek sıcaklıklarda pişirilmiş anortit seramiklerin termal iletkenliğini ölçmek için alı konulan sap nauyruları uygun olarak 0,39 W/m·K (1,00 g/cm³) ve 0,11 W/m·K (0,78 g/cm³) değerlerine sahiptitlesi göstermiştir. Elde edilen sonuçlar ışıkta, yumurta kabuğu atıklarını yeniden kullanılmamıştır.
analytical models have been suggested to explain this effect [4]. However, thermal conductivity relies not only on the amount of pore but also on factors such as the size and shape of the pore, chemical and mineralogical composition [1,5,6].

In porous ceramics, the controlled microstructure can be created by incorporating pore forming agents into the system and using different methods. Various processing techniques can be used to fabricate porous ceramics such as gel casting [7–9], freeze-drying method [10–12], direct foaming [13] and sacrificial templates method [14]. Alongside the existing methods, the use of pore-forming agents that burn during firing is most common for IFBs production. In this method, sawdust, rice husk, fine coke, starch, wheat particles, expanded perlite and vermiculite or high-molecular synthetic compounds such as foam polystyrene, polyethylene, polyvinyl are added as flammable materials to form a pore during firing [5,6,14–20].

Until now, many research studies have been carried out on anorthite-based ceramics. Anorthite ceramics can be grouped as dense and porous ceramics according to their usage fields and their engineering properties. Thanks to its outstanding physical properties such as low thermal expansion coefficient, low dielectric constant at high frequencies, and excellent thermal shock resistance, these materials with dense structures are used in low-temperature co-fired ceramic surface applications, electrical insulators, and electronic devices for mobile communications [2,21–25].

Cheng et al. also reported that anorthite ceramics can be used in tableware porcelains to increase service and quality thanks to its low relative refractive index [24]. Capoglu et al. carried out various studies to produce anorthite based on whiteware and porcelainised stoneware materials [26–28]. In addition to these, Agathopoulo et al. reported that the fluorapatite-anorthite binary system is a promising material that can be used in biomedical materials [29].

Porous anorthite-based IFBs have been the focus of interest in their use as thermal insulators thanks to their high melting point, low theoretical density, low thermal conductivity and low thermal expansion coefficient [2,4,30–35]. These materials can be produced synthetically cheaply from various raw materials. Until now, raw materials such as calcite, quartz, kaolinite, alumina have been the most frequently used materials in investigations on anorthite ceramic production [1,31]. However, in recent years, there has been also significant research on the reuse of waste materials as a new source of raw material [1,2,32–35]. Solid wastes-based porous ceramics have fascinated more and more relevancies because of their environmentally friendly and low-cost on the extensive usage of solid wastes [36]. By this time, many studies have been carried out on calcium-based alternative raw materials for CaO, one of the main components of anorthite. A patent was added to the literature by Brosnan on the usage of recycled paper waste in the manufacturing of low density ceramics [32]. Sutcu et al. generated a sort of porous anorthite ceramic from recycled paper processing residues and clay of diverse sources at 1200–1400°C and stated that using recycled paper waste can be utilized as a convenient option raw material source to fabricate porous anorthite ceramics under favour of its organic and inorganic ingredient [1,2]. The filter cake, a by-product in the sugar beet industry, was used by El-Maghraby et al to obtain anorthite as an alternative source of calcium. They reported that anorthite could be attained as a predominant phase over 1200°C up to 1350°C [33]. Lime sludge is a product that occurs during the causticisation step in the paper production process. Qui et al. was managed to produce anorthite at 1000°C using lime sludge and fly ash released during the slakka recycling process [34]. They have produced anorthite ceramic using a beneficial alternative to contribute to waste recycling. Using steel slag wastes and high alumina containing fly ash as the main raw materials, Zone et al. has synthesized porous anorthite ceramic with a porosity of about 49% at a low temperature of 1170°C. Their work provided a new idea for the use of high alumina fly ash [35].

Evaluated as the 15th largest food industry pollution by the Environmental Protection Agency, eggshell waste is actually a quite substantial resource for calcium based ceramic compositions. When the eggshell is subjected to heat treatment at just about 900°C, it includes over 99% CaO by weight. For this reason, it has the potential to be used in many different products from food products to industrial application products as a source of calcium [37,38]. Vichaphund et al. synthesized the single-phase wollastonite at 1100°C utilizing the admixture of biosolid waste eggshell and silica by microwave-assisted solid-state reaction [39]. Finally, the low thermal conductivity values of 0.25–0.29 W/m K led them to conclude that calcium silicate produced by adding eggshell waste can easily be used as a thermal insulation material [40].

Although there are numerous studies on the fabrication of porous anorthite ceramics using various raw materials, no study has yet been reported in the literature in which eggshell waste is used for the production of anorthite-based lightweight materials. To cope with, eggshell wastes and fireclay with pore making polymer additives were utilized so as to fabricate porous anorthite insulating firebricks. Adding eggshell waste, an alternative source of CaO to the calcium silicate system, allows us to create an anorthite composition. During firing, the combustion of pore-maker expanded polystyrene (EPS) and the decomposition of calcium carbonate (CaCO₃) contribute to occur a more porous structure by creating micro and macro-sized gaps in the body.

2. EXPERIMENTAL PROCEDURE

In this study, commercially purchased fireclay (chamotte125, Esan, Turkey) as a source of alumina (Al₂O₃) and silica (SiO₂) and eggshell waste collected
from catering companies as a source of calcium was used for the production of porous anorthite ceramics. This waste also has a pore-making effect due to the degradation of calcium carbonate in the course of firing. Collected eggshells were exposed to pretreatments such as rinsed with distilled water, drying at 50°C overnight, and then grinding in mechanical disc milling to get powder form. Before starting any processing, the physical and chemical features of the raw materials were characterized in advance. Mixtures consisting of fireclay and different ratios of powder eggshell waste (between 20 and 40 wt.%) were prepared for the purpose of forming an anorthite composition. The powder mixture was suitably mixed in a mortar. In order to optimize the firing temperature and eggshell percentage, the prepared powder mixes to optimize firing temperature and eggshell percentage were fired at temperatures of 1250, 1300, and 1350°C for 2 hours in an oven (Protherm PLF 16/15). In the two-stages heating regime, it was reached from room temperature to 400°C with 5°C/min in the first stage, subsequently, from this temperature to the dwell temperatures with 10°C/min.

3. RESULTS and DISCUSSION
3.1. Characterization of Raw Materials
The chemical compositions of commercially purchased fireclay with a powder size under 75µm and the eggshell waste collected from household kitchens and grinded to sizes below 100µm are shown in Table 1. Composed of a high percentage of alumina and silica, fireclay consisted of a low percentage of sodium, potassium, and calcium. The eggshell is composed of mainly calcium oxide.

The XRD patterns of the raw materials were depicted in Fig. 1. The raw eggshell waste (Fig. 1a) is comprised actually of calcite (CaCO₃). The crystalline phases determined within raw fireclay material (Fig. 1b) were quartz (SiO₂), mullite (3Al₂O₃·2SiO₂), and cristobalite (SiO₂). Since fireclay is a thermally processed vitrified clay, the loss on ignition is close to zero. Fireclay demonstrates characteristic large humps near crystalline peaks. This is due to the melting alkali forming an amorphous structure during the heat treatment of fireclay.

![Figure 1. XRD patterns of raw eggshell waste (a) and fireclay (b).](ENGLISHTEXT)
that the release of physisorbed H$_2$O, and the organic substances decomposition occurred between 200 and 400°C with a weight loss of 5%. Full calcination occurred at temperatures between 600–840°C with a weight loss of 43%, resulting in phase change owing to the decomposition of CaCO$_3$ to CO$_2$. The total weight loss of the eggshell was observed as 48% from room temperature to 1000°C.

3.2. Characterization of Fired Samples
Mixtures being made up of fireclay and eggshell waste with different proportions (20–40 wt.%) were fired at several temperatures including 1250°C, 1300°C, and 1350°C for the purpose of finding the optimum conditions of anorthite composition. XRD patterns of fired samples involving the admixtures of fire clay and eggshell waste are shown Fig. 3. Since a high amount of anorthite phase and less amount of mullite phase were observed in XRD patterns, optimum conditions for production of anorthite were determined as 1300°C temperature and 35 wt.% eggshell addition. The photographs of porous anorthite samples with EPS addition in different proportions (0-30 % by volume) are shown in Fig. 4. The macro-pores that formed on the surfaces of the samples after firing are obviously observed due to burning of fine EPS granules.

1350°C for the purpose of finding the optimum conditions of anorthite composition. XRD patterns of fired samples involving the admixtures of fire clay and eggshell waste are shown Fig. 3. Since a high amount of anorthite phase and less amount of mullite phase were observed in XRD patterns, optimum conditions for production of anorthite were determined as 1300°C. The physical properties, thermal conductivity, and the compressive strength of the anorthite ceramics produced in the present study were measured. Experimental results of anorthite samples with EPS additive in different proportions (0-30 % by volume) are listed in Table 2. The apparent porosity of the fired samples varied between 61% and 68.1%, with the addition of EPS.
Thermal conductivity and bulk density of samples with 0% and 30% EPS additive exhibited decreases of 71.8% and 18.9%, respectively. As the bulk density decreased, the thermal conductivity also decreased. Meanwhile the lowest bulk density was 0.86 g/cm$^3$, thermal conductivity values of and 0.11 W/m·K in samples with 30% EPS that were fired at 1300°C. In addition, the bulk density of samples from weight and dimensional measurements were calculated between 1.00 g/cm$^3$ and 0.78 g/cm$^3$ according to EPS addition. From these results, it is noteworthy that the physical properties of the samples containing 30% EPS are consonant with the group of IFBs [3]. The compressive strengths of the produced samples exhibited decreasing with increasing EPS ratio.

In addition to the decomposition of calcium carbonate, the addition of EPS increased the number of pores in the structure and caused a decrease in density and conductivity values. The presence of a high amount of pores in the structure caused a significant change in the compressive strength. As can be seen from Table 2, the compressive strength of samples fired at 1300°C and added to a maximum of 30% EPS showed a decrease of about 55%, and the compressive strength was observed 2.9 MPa.

Fig. 5 shows SEM images of anorthite sample produced from mixture of fireclay and 35% eggshell waste. It has been observed that the ceramic samples have composed of fine-grained crystals with plate like form.

| Physical properties                  | 0%    | 10%   | 20%   | 30%   |
|-------------------------------------|-------|-------|-------|-------|
| Apparent porosity, %                | 61±0.1| 63±0.2| 66.1±0.9| 68.1±1.2|
| Water absorption, %                 | 57.6±0.1| 62.4±0.3| 72.5±0.6| 79.7±0.7|
| Apparent specific gravity, g/cm$^3$| 2.72±0.02| 2.74±0.01| 2.69±0.05| 2.68±0.04|
| Bulk density, g/cm$^3$              | 1.06±0.02| 1.01±0.02| 0.91±0.04| 0.86±0.06|
| Bulk density, g/cm$^3$ (ASTM C134) | 1.00±0.04| 0.97±0.02| 0.85±0.05| 0.78±0.03|
| Thermal conductivity, W/m·K         | 0.39±0.01| 0.32±0.02| 0.22±0.02| 0.11±0.03|
| Compressive strength, MPa           | 6.4±0.9 | 3.95±0.2 | 3.8±0.2 | 2.9±0.4 |

Table 2. Test results of porous anorthite samples fired at 1300°C.

Figure 5. SEM images of anorthite sample with 35% eggshell additive fired at 1300°C: (a) 1000x, (b) 5000x.

Figure 6. SEM images of the fired anorthite samples with 35% eggshell additive at 1300°C according to EPS addition of (a) 0%, (b) 20%.
The microstructures of 0% and 20% EPS added anorthite samples fired at 1300°C are shown in Figs 6a and 6b, respectively. SEM micrographs of samples reveal that the effect of EPS addition was clearly seen from the microstructures. Most of the pores occurred as a result of the removal of polystyrene besides the decomposition of CaCO₃ throughout firing. Formation of micro and macro pores were observed due to gas output and burning of fine EPS spheres. The formation of a highly porous structure also contributed to lower thermal conductivity.

4. CONCLUSION
In conclusion, we have been able to successfully produced porous anorthite ceramics, for the first time, by using fireclay, eggshell waste, and pore-forming expanded polystyrene. In the examples, it was found that a firing temperature of 1300°C and an eggshell additive of 35% would be well enough to fabricate a porous anorthite ceramic. All of the samples included anorthite as the main phase and mullite as the minor secondary phase. The physical properties of the anorthite samples obtained after adding EPS at different rates (0-30%) to the mixture to increase porosity were investigated. It was deduced that bulk densities varied from 1.00 to 0.78 g/cm³, and also thermal conductivities ranged from 0.39 to 0.11 W/m·K relying on the amount of porosity. Increasing the porosity in the structure led to a decrease in bulk density and thermal conductivity, thus increasing thermal insulation. This situation is the same for compressive strength. With the increase in porosity, the compressive strength of the samples changed between 6.4 and 2.9 MPa.

Finally, it was concluded that the eggshell waste could be utilized as a favorable alternative calcium source for fabrication of porous anorthite ceramics thanks to organic and inorganic content. It can contribute to further this work use various waste calcium sources to support recycling, and produce a more porous structure in order to obtain better results from thermal conductivity tests.

DECLARATION OF ETHICAL STANDARDS
The author(s) of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

AUTHORS' CONTRIBUTIONS
Merve TORMAN KAYALAR: Wrote the manuscript.
Gökhan ERDOĞAN: Performed the experiments and analysed the results.
Ahmet YAVAS: Supported the writing and editing of the article.
Saadet GÜLER: Supported the writing and editing of the article.

Mücahit SÜTÇÜ: Analysed the results. Guided the article writing process and checked the entire article before submitting it.

CONFLICT OF INTEREST
There is no conflict of interest in this study.

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