INVESTIGATION OF BORON WASTE FOR POTENTIAL USE IN THE SYNTHESIS OF CORDIERITE CERAMICS AT LOWER TEMPERATURES

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Keywords

Cordierite, Boron waste, Phase evolution, Sintering

Abstract

The utilization of industrial by-products in ceramic production has become an essential strategy for enhancing both the sustainability and competitiveness of the ceramics industry. This study investigates the valorization of boron waste as a sustainable alternative raw material and sintering additive in the production of cordierite ceramics ($2MgO.2Al_2O_3.5SiO_2$). Cordierite ceramics are essential for high-temperature applications due to their low coefficient of thermal expansion and excellent thermal shock resistance; however, their synthesis typically requires high temperatures and narrow sintering ranges, leading to high energy consumption. In this work, cordierite bodies were formulated using magnesite, kaolin, and quartz, with boron waste additions ranging from 0 to 1 wt.% to evaluate its potential as a cost-effective fluxing agent. The samples were produced via the slip casting method and sintered at 1260°C and 1320°C for 2 hours. Phase analysis, performed using X-ray diffraction (XRD) and Rietveld refinement. Additionally, the bulk densities of the sintered specimens were determined by the Archimedes method, while their flexural strengths were measured using three-point bending tests. The results, quantified via Rietveld refinement, indicate that the cordierite phase content increased with the addition of 1 wt.% boron waste at 1320°C. Furthermore, a significant improvement in mechanical properties was observed; the flexural strength rose to 30.36 MPa. This study demonstrates that the incorporation of boron waste not only promotes the synthesis of high-performance cordierite ceramics at lower temperatures but also offers a viable pathway for environmental protection and resource management through the effective recycling of industrial wastes.

KORDİYERİT SERAMİKLERİNİN DÜŞÜK SICAKLIKTAKİ SENTEZİNDE BOR ATIKLARININ KULLANABİLİRLİĞİNİN ARAŞTIRILMASI

Anahtar Kelimeler

Kordiyerit, Bor atığı, Faz gelişimi, Sinterleme

Öz

Endüstriyel atıkların seramik üretiminde değerlendirilmesi, seramik endüstrisinin hem sürdürülebilirliğini hem de rekabet gücünü artırmak için temel bir strateji haline gelmiştir. Bu çalışma, kordiyerit seramiklerinin ($2MgO.2Al_2O_3.5SiO_2$) üretiminde bor atığının sürdürülebilir bir alternatif hammadde ve sinterleme katkı maddesi olarak kullanımını incelemektedir. Kordiyerit seramikleri, düşük ısıl genleşme katsayıları ve mükemmel ısıl şok dirençleri nedeniyle yüksek sıcaklık uygulamaları için temel malzemelerdir; ancak sentezlenmeleri genellikle yüksek sıcaklıklar ve dar sinterleme aralıkları gerektirir, bu da yüksek enerji tüketimine yol açar. Bu çalışmada, manyezit, kaolin ve kuvars kullanılarak kordiyerit bünyeler formüle edilmiş ve bor atığının maliyet etkin bir eritici olarak potansiyelini değerlendirmek amacıyla ağırlıkça %0 ile %1 arasında bor atığı ilave edilmiştir. Numuneler slip döküm yöntemiyle üretilmiş ve 1260°C ile 1320°C sıcaklıklarda 2 saat boyunca sinterlenmiştir. Faz analizi, X-ışını difraksiyonu (XRD) ve Rietveld metodu kullanılarak gerçekleştirilmiştir. Ayrıca, sinterlenen numunelerin hacimsel yoğunlukları Arşimet yöntemiyle, eğme dayanımları ise üç nokta

eğme testi uygulanarak belirlenmiştir. Rietveld yöntemi ile nicelleştirilen sonuçlar, 1320°C'de ağırlıkça %1 bor atığı ilavesiyle kordiyerit faz içeriğinin arttığını göstermektedir. Ayrıca, mekanik özelliklerde önemli bir iyileşme gözlemlenmiş; eğme dayanımı 30,36 MPa değerine yükselmiştir. Bu çalışma, bor atığı ilavesinin sadece yüksek performanslı kordiyerit seramiklerinin daha düşük sıcaklıklarda sentezlenmesini teşvik etmekle kalmadığını, aynı zamanda endüstriyel atıkların etkin geri dönüşümü yoluyla çevrenin korunması ve kaynak yönetimi için uygulanabilir bir yol sunduğunu göstermektedir.

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1. Introduction

In recent years, cordierite ceramics ($2MgO.2Al_2O_3.5SiO_2$) have gained wide application areas in line with the advancements in materials science, especially within the ceramics industry. Cordierite ceramics, with their low thermal expansion coefficient ($1–2 \times 10^{-6}$/K between 20–800 °C), excellent thermal shock resistance, high chemical durability, and high refractoriness, have a wide range of applications, including catalytic converter substrates, multilayer electronic circuit substrates, solid–liquid separation filters, furnace construction materials, thermal and acoustic insulation components, and materials requiring high thermal shock resistance (Camerucci, Cavalieri, Moreno 1998; Chen et al., 2022; Rodrigues and Moreno 2008). In thermal power plants, filters are used to capture solid particles released into the environment. Filters intended for use at temperatures between 800–1000 °C must exhibit excellent thermal shock resistance and chemical stability. Due to their high thermal shock and corrosion resistance, cordierite ceramics are employed as filter materials. Furthermore, because of their low thermal expansion coefficient and dielectric constant, cordierite is also preferred in the field of electrical and electronic applications, particularly in the form of cordierite porcelains widely used for electrical insulation (Buchanan, 2004).

The high-temperature form of cordierite, known as indialite (α-cordierite), possesses a hexagonal structure and can be synthesized via the solid-state reaction of mixed powders at 1300–1460 °C. Mixtures with a high cordierite content exhibit a narrow sintering range; below 1300 °C, the formation of the cordierite phase is limited, while at 1450 °C, cordierite decomposes into forsterite ($2MgO·SiO_2$) and mullite ($2Al_2O_3·SiO_2$) (Çelik, 2008; Sha et al, 2026). Due to the narrow sintering range and high sintering temperature of cordierite, the control of the composition during sintering and the synthesis of single-phase cordierite is inherently difficult. Minor differences in compositions and temperatures result in formation of intermediate or undesired phases (Camerucci et al., 1998, Cameruccia, Urretavizcaya, Cavalieri, 2003).

Various methods have been reported in the literature for the synthesis of cordierite. Conventional approaches include the solid-state reaction of magnesium, aluminum, and silicon oxides; crystallization from glass precursors; and liquid-phase sintering of raw materials containing magnesium, aluminum, and silicon (Gören, Özgür and Göçmez, 2006). In addition, a number of studies have focused on the incorporation of various additives, including mullite, cerium oxide, nickel oxide, zirconia, B_2O_3, P_2O_5, Bi_2O_3, fly ash, and rice husk ash, with the aim of enhancing the properties of cordierite and broadening its potential applications (Çelik, 2008; Naskar and Chatterjee, 2004; Oliveira and Fernandes, 2002) Furthermore, the addition of B_2O_3 tended to cause the formation of spherulitic dendrites with thin dendritic arms, which promoted the formation of μ-cordierite, either from crystallization of the residual glass or from transformation of μ-cordierite (Demirci, and Günay, 2011).

In this study, the production of cordierite ceramics using boron wastes, which are considered industrial by-products and remain underutilized, was aimed. The investigation focuses on whether the use of boron waste leads to the formation of the cordierite phase in the final material and on evaluating the quality of the resulting ceramics. Although there are many studies on the utilization of boron wastes, to the best of the authors' knowledge, information regarding their use in the synthesis of cordierite is rather limited. For instance, (Akpınar and Tıkız, 2019) examined the effects of various boron oxide additives on electrical properties of cordierite ceramics. They used colemanite, anhydrous borax and boron wastes (1, 2, and 3% by wt) then sintered in the temperature range of 1000-1300 °C and the electrical properties of the sintered samples were characterized. As a result of the sintering processes, it was determined that similar dielectric properties to those of cordierite were obtained at 1200 °C, and therefore, it can be concluded that a reduction of about

100–150 °C in the sintering temperature can be achieved with boron oxide–containing additives without compromising the dielectric properties of cordierite.

Thus, the aim is the utilization of waste-derived materials has attracted significant attention as a sustainable approach to resource management and environmental protection. The valorization of waste materials in ceramic production has become an essential strategy for enhancing both the sustainability and competitiveness of the ceramics industry. Another reason for selecting a boron compound as a waste material in the synthesis of cordierite ceramics in this study is that, according to the literature, it has been reported to accelerate the formation of α-cordierite (indialite) and to decrease the glass transition temperature, crystallization temperature, and sintering temperature of cordierite. In this study, boron-containing waste was used as sintering additive to facilitate the sintering of cordierite ceramics at lower temperatures. For this purpose, boron waste was added at concentrations of 0–1 wt.% to the cordierite stoichiometry formulated by using magnesite, kaolin, and quartz raw materials. The samples were then sintered at different temperatures (1260 and 1320°C) and for 2 hours and the microstructural, phase, and mechanical properties were investigated in detail.

2. Materials and method

In this study, research and publication ethics were compiled with.

Different compositions were prepared using the main raw materials, taking into account the stoichiometric ratio of the cordierite phase, approximately as follows: quartz (55–70 wt.%), alumina (25–35 wt.%), magnesite (10–25 wt.%), boron waste (0,5–1 wt.%).

In this study, tincal debris (a boron derivative known as boron waste) obtained from industrial discharge was utilized. For the purpose of providing a standardized chemical reference in the formulations, this debris is represented by its main constituent, borax ($Na_2B_4O_7{\cdot}10H_2O$). This waste was specifically chosen due to its high B_2O_3 content, which serves as a potent fluxing agent while addressing industrial waste management concerns. Boron waste material was used in different weight ratios. The chemical compositions of the raw materials were determined using an XRF spectrometer (Rigaku ZSX Primus II). The raw materials used in the composition have particle sizes smaller than 100 μm and weighed according to the compositions. The mixture was then milled in a ball mill with the addition of 35–40% water for 2 hours at 400 rpm and then dried in an oven at 105°C for 24 hours. The prepared samples were formed using the slip casting method (Fig 1).

Figure 1. Representative samples formed by the slip casting method.

The samples were sintered at 1260°C and 1320°C for 2 hours at heating rate of 6°C/min**.** After sintering, the density and porosity of the samples were measured using the Archimedes principle with an analytical balance. according to the following formula:

$$\rho(bulk) = \frac{W1}{W3-W2}\ x\ \rho water \quad (1)$$

where;
W1: Dry weight of the sample,
W2: Suspended weight in water,
W3: Saturated weight in air,
P_{bulk} : Bulk density,
ρ_{water} : Density of water

The phase analyses were accomplished by using X-Ray diffractometer (Rigaku Rint2200 series). Rietveld refinement quantifications were performed from the diffraction pattern collected in the 0–80° (2Θ) range in order to determine the amount of the formed cordierite phase. The flexural strength of samples was tested in a Shimadzu AG-IS 250kN machine at 1 mm/min (Fig 2). The formula for flexural strength, σ, under a three-point load was used as;

$$\sigma = 3FL/2bd^2 \quad (2)$$

where;
F = force at fracture point,
L = support span length,
b = width of sample,
d = thickness of the sample.

The experimental samples were used in the size of 25×15×250 mm. Flexural tests were performed using five different specimens for each composition, and the average results were reported.

Figure 2. A universal testing machine equipped with a three-point bending fixture.

3. Results

The sintering conditions of samples containing different percentages of boron waste and the experimental results are summarized in Table 1. The theoretical density of cordierite is 2.52 g·cm^{-3} (Guo et al., 2024). The measured densities increased as the sintering temperature increased. In addition, it was observed that the density increased with the increasing amount of boron waste for both sintering temperature. The highest density value was obtained in the sample with the highest boron waste content sintered at 1320°C for 120 min (Fig 5).

Table 1. Experimental results of boron waste-added cordierite samples sintered at different temperatures for 120 min.

Boron waste (wt.%)	Sintering Temp	Time (min)	Cordierite (%)	Flexural Strength (MPa)
0	1260	120	65,04	13,07
0	1320	120	72,54	17,76
0.5	1260	120	73,30	19,79
0.5	1320	120	86,58	24,68
1	1260	120	76,16	21,85
1	1320	120	92,14	30,36

Phase analysis of the composites sintered at both temperatures revealed cordierite, indiallite (α-cordierite), and enstatite ($MgSiO_3$) peaks across all samples. Fig. 3 illustrates a representative XRD pattern for the 1 wt.% boron waste-added sample sintered at 1260°C. The XRD results were also confirmed by Rietveld analysis. The results are given in Table 1. Based on the Rietveld refinement results, comparing the samples sintered at the same temperature, for example for 1260°C, the cordierite content rose with increasing boron waste addition. A similar trend was observed for the samples sintered at 1320°C. This enhancement can be attributed to the role of boron as a fluxing agent, which promotes the formation of a liquid phase during sintering. This liquid phase may facilitate the diffusion of chemical species and accelerate the reaction kinetics, thereby leading to a more efficient crystallization of the cordierite phase. Furthermore, the presence of this liquid phase not only improved phase formation but also contributed to higher densification by filling the inter-granular pores, which is consistent with the measured increase in bulk density. Comparing the two different sintering temperatures for samples with the same boron waste contents, the amount of cordierite phase increases with increasing sintering temperature. While the cordierite content was determined to be approximately 76.16% (Table 1 and Fig 3) for the sample with the highest boron waste addition sintered at 1260°C, it increased to around 92.14% for the same composite sintered at 1320°C. The significant increase in cordierite content from app. 76.16% to 92.14% as the temperature rose to 1320°C suggests that the boron waste may play a crucial role as a fluxing agent. At higher temperatures, the boron-rich liquid phase likely reduces the viscosity of the system and enhances the diffusion rates of MgO, Al_2O_3 and SiO_2 species. This liquid phase formation may promote the reaction kinetics for cordierite crystallization, leading to a more complete phase transformation in the composite with the highest boron waste addition.

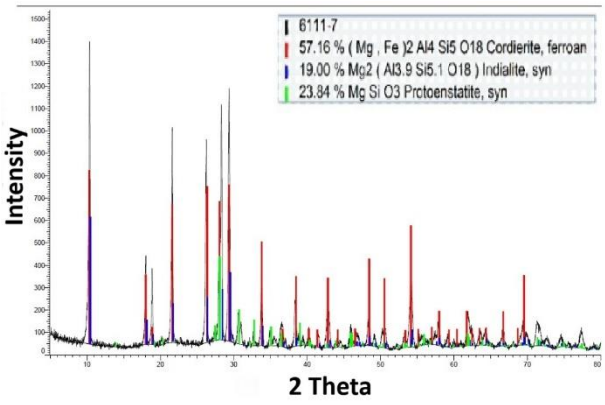

Figure 3. XRD pattern of 1 wt% boron waste-added cordierite samples sintered at 1260°C for 120 min.

Following the characterization studies, the sintered samples were subjected to flexural strength tests. The

mechanical properties of the sintered samples are summarized in Table 1. Fig. 4 illustrates a representative flexural test result for the 1 wt.% boron waste-added sample sintered at 1320°C. It was observed that both the sintering temperature and the boron waste content have a significant impact on the flexural strength. For instance, the flexural strength of the 1 wt.% boron waste-added sample increased from 21.85MPa to 30,36MPa as the sintering temperature. rose from 1260°C to 1320°C. This improvement is consistent with the increase in bulk density and the higher cordierite phase percentage. The results indicate that the liquid phase formed by the boron waste may not only promote densification but also enhance the mechanical integrity of the cordierite-based composites.

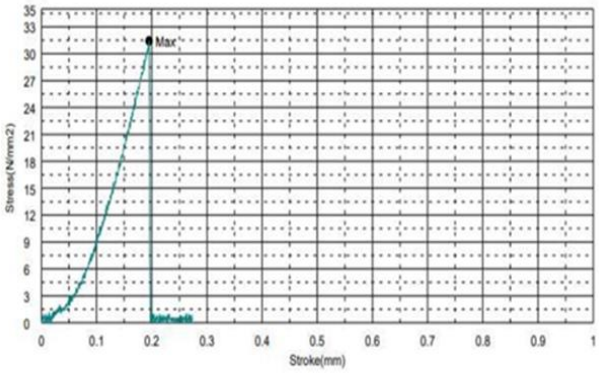

Figure 4. Flexural stress-stroke diagram of 1 wt% boron waste-added cordierite samples sintered at 1320°C for 120 min.

The graphical representation of the density and flexural strength for the samples sintered at 1320°C is shown in Figure 5.

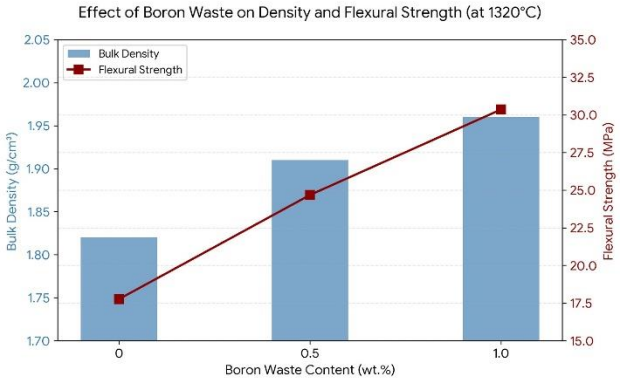

Figure 5. Influence of boron waste content (wt. %) on the bulk density and flexural strength of the cordierite ceramics sintered at 1320°C for 120 min.

When compared to the literature, the observed reduction in sintering temperature (approx. 100–150°C) is consistent with previous findings. For instance, Akpinar and Tıkız (2019), who reported that boron-based additives enhance dielectric properties while facilitating lower thermal processing. The improvement in flexural strength also aligns with the the literature. Demirci and Günay (2011) reported that B_2O_3 additions promoted the crystallization of μ-cordierite into the more stable α-cordierite (indialite), thereby reinforcing the ceramic matrix.

4. Discussion

In this study, the effect of boron waste addition on the phase evolution, densification, and mechanical properties of cordierite ceramics was systematically investigated. The addition of boron waste facilitated the formation of the cordierite phase at lower sintering temperatures. The highest cordierite content, comprising both cordierite and indiallite (α-cordierite) phases, was reached in the sample containing 1 wt.% boron waste sintered at 1320°C. It is considered that the boron waste acted as an effective fluxing agent, promoting the formation of a liquid phase during sintering. This liquid phase may have enhanced the reaction kinetics and diffusion rates of MgO, Al_2O_3 and SiO_2 leading to a more complete crystallization of cordierite compared to boron-free samples. Hence, the bulk density of the composites increased proportionally with both the sintering temperature and the boron waste content. The maximum density was achieved in the sample containing 1 wt.% boron waste sintered at 1320°C, which can be attributed to the liquid phase filling the inter-granular pores. Same correlation was observed between the cordierite phase amount, densification, and mechanical strength. The flexural strength was improved as the boron waste addition increased. These findings demonstrate that boron-containing industrial wastes can be successfully utilized as alternative raw materials to reduce sintering temperatures and enhance the properties of cordierite-based ceramics, contributing to more sustainable ceramic production processes.

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Author Contributions

Ö.K. Yılmaz contributed to the investigation, data curation, and literature review; B.Y. Islak was responsible for the formal analysis, methodology, interpretation of results, and writing of the original paper. İ. Tatar contributed to the conceptualization and supervision.

Conflict of Interest

No conflict of interest has been declared by the authors.

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