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PRODUCTION AND CHARACTERIZATION OF WALL TILE CERAMICS WITH THE ADDITION OF AMORPHOUS BORON

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Abstract

This study investigates the effects of amorphous boron (Fluka Boron) on the mechanical and thermal properties of ceramic wall tiles. Samples with boron concentrations of 0%, 1%, 3%, 5%, 7%, and 9% were sintered at 1000°C. Results showed that moderate amorphous boron additions (3%–5%) significantly improved bulk density and compressive strength due to enhanced densification and reduced porosity. These effects are attributed to boron's fluxing action, which promotes particle bonding during sintering. Amorphous boron additions of up to 5% were observed to enhance mechanical properties and thermal conductivity, with optimal performance at this concentration. However, amorphous boron levels exceeding 5% led to diminished mechanical strength and thermal conductivity due to the formation of a glassy phase and structural heterogeneity, despite reduced apparent porosity. This study on wall tile ceramics highlights the critical role of amorphous boron concentration in balancing densification, phase composition, and microstructure to enhance compressive strength and thermal conductivity performance. By highlighting the interplay between boron content and material performance, the research contributes valuable knowledge toward the development of sustainable, high-performance ceramic materials.

Keywords: Ceramic wall tiles, Amorphous boron addition, Powder metallurgy, Materials characterization, Mechanical and thermal performance

AMORF BOR KATKILI DUVAR KAROSU SERAMİKLERİNİN ÜRETİMİ VE KARAKTERİZASYONU

Özet

Bu çalışma, amorf borun (Fluka Boron) seramik duvar karolarının mekanik ve termal özellikleri üzerindeki etkilerini araştırmaktadır. Bor konsantrasyonları %0, %1, %3, %5, %7 ve %9 olan numuneler 1000°C'de sinterlenmiştir. Sonuçlar, orta düzeyde amorf bor ilavelerinin (%3-%5), artan yoğunlaşma ve azalan gözeneklilik nedeniyle yığın yoğunluğunu ve basınç dayanımını önemli ölçüde artırdığını göstermiştir. Bu etkiler, borun sinterleme sırasında partikül bağlanmasını teşvik eden akışkanlaştırma etkisine bağlanmaktadır. %5'e kadar amorf bor ilavelerinin mekanik özellikleri ve termal iletkenliği artırdığı ve bu konsantrasyonda optimum performans gösterdiği görülmüştür. Bununla birlikte, %5'i aşan amorf bor seviyeleri, görünür gözenekliliğin azalmasına rağmen camsı faz oluşumu ve yapısal heterojenlik nedeniyle mekanik mukavemet ve termal iletkenliğin azalmasına yol açmıştır. Duvar karosu seramikleri üzerine yapılan bu çalışma, basınç dayanımı ve termal iletkenlik performansını artırmak için yoğunlaştırma, faz bileşimi ve mikro yapıyı dengelemede amorf bor konsantrasyonunun kritik rolünü vurgulamaktadır. Bor içeriği ve malzeme performansı arasındaki etkileşimi vurgulayarak, araştırma sürdürülebilir, yüksek performanslı seramik malzemelerin geliştirilmesine yönelik değerli bilgilere katkıda bulunmaktadır.

Anahtar Kelimeler: Seramik duvar karoları, Amorf bor katkısı, Toz metalurjisi, Malzeme karakterizasyonu, Mekanik ve termal performans

Cite

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1. Introduction

Wall tiles are an indispensable element of contemporary architectural and interior design, fulfilling both functional and aesthetic roles. Wall tiles are usually made of ceramic or porcelain. They are known for their durability, low maintenance, and versatility. These properties make them popular in various applications, including those in the residential, commercial, and industrial sectors.[1].

The ease of cleaning, durability, and mechanical strength of ceramic tiles contribute to their widespread use in diverse settings, including walls, countertops, and panels [2]. Ceramic tiles are renowned for their low porosity and high resistance to staining and wear, rendering them eminently suitable for high-areas and wet areas such as kitchens and bathrooms[3].

Wall tiles are produced using natural materials, including clay, quartz, feldspar, porcelain, and glass. The selection of these materials directly influences the physical and mechanical properties of the tiles[4]. Through different formulations and controlled manufacturing processes, each type of wall tile offers unique advantages for specific applications. The distinct performance characteristics of these tiles are tailored to specific needs.[1].

The production of wall tiles is a complex process that encompasses a number of key stages [5]. These include the preparation of raw materials, shaping, drying, glazing and sintering at high temperatures. Sintering is a crucial step in the production process, as it enables the attainment of the requisite hardness, water resistance, and structural integrity, which are essential for the tiles to exhibit long-lasting performance. The advent of advanced manufacturing technologies has facilitated the creation of bespoke tiles with enhanced functionalities, including antibacterial surfaces, thermal insulation, and self-cleaning properties. These innovations have facilitated the application of wall tiles in a broader range of settings, including public facilities, healthcare environments, and sustainable residential spaces [1].

In response to the evolving demands of the industry, there has been a notable shift in focus towards the improvement of the physical and mechanical properties of wall tiles through the implementation of material modifications. Additives, including titanium dioxide (TiO_2), alumina, and boron compounds, have been incorporated into the manufacturing process to improve the mechanical strength, chemical resistance, and thermal stability of the tiles. Concurrently, the incorporation of industrial waste materials, including fly ash, marble dust, and recycled glass, has gained prominence as a sustainable practice[4]. This dual emphasis on performance enhancement and sustainability is aligned with global efforts to promote eco-friendly building practices and reduce energy consumption[6].

In this investigation, a range of amorphous boron powder ratios were incorporated into ceramic wall tiles to assess their impact on the tiles' mechanical and thermal properties. Despite the extensive research conducted on conventional ceramic additives, the applicability of amorphous boron as a addition, agent in wall tiles has yet to be thoroughly investigated. This research aims to address this gap in the literature by assessing the effects of amorphous boron on the densification, strength, and thermal conductivity of ceramic tiles, thereby contributing novel insights into enhancing both performance and sustainability within the domain of ceramic manufacturing.

2. Experimental Procedures

The wall tile mix used in this study was supplied by a local ceramic factory in Izmir, Turkey. Amorphous Boron (Fluka Boron, 95–97% purity) is a commercially available additive. Firstly, the raw material was classified by sieve analysis to obtain powder samples with particle sizes suitable for wall tile production. The sub-100-micron wall tile mixture was used in the study. Then, approximately 8 g mixtures were prepared with Amorphous Boron powder. The prepared mixtures contain 0%, 1%, 3%, 5%, 7%, 9% by weight of amorphous boron additive.

Table 1. Designation and classification of samples for production

	Percent	Wall tile ceramic	Amorphous boron addition
M		8 g	-
M1B	%1	7.92	0.08
M3B	%3	7.76	0.24
M5B	%5	760	0.40
M7B	%7	7.44	0.56
M9B	%9	7.28	0.72

After, the mixtures were mixed in a mixer at 250 rpm for approximately 15 min. The mixtures were compacted with a hydraulic press at a pressure of 30 bar to produce ceramic wall tile samples with dimensions of 20 mm in diameter and 20 mm in height. The molded samples were kept at ambient temperature for 24 h and then oven dried at 45°C for 10 h and 110°C for 24 h, respectively. The samples were then gradually heated to temperatures of 1000°C using an electric type of furnace (Protherm-PLF12/25).

Figure 1. Prior to the sintering process

Figure 2. After the sintering process

After the sintering process, the produced wall tile samples were left in the furnace to cool to room temperature.

X-ray diffraction (XRD) analysis was performed utilizing a Bruker D2 Phaser to ascertain the phase composition of both the raw materials and all sintered specimens. The microstructural morphology of the manufactured samples was analyzed using scanning electron microscopy (SEM, Carl Zeiss 300VP), while energy-dispersive spectroscopy (SEM-EDS) was employed to investigate the detailed distribution of the additive within the structure. The method of measuring physical properties was carried out in accordance with the ASTM C20-00 standard. Compressive strength tests were conducted to evaluate the mechanical performance of the samples, and their thermal conductivity was measured using a C-Therm TCi. Three samples were used in the analyses.

3. Result and Discussion

The XRD in Figure 3. demonstrates the phase compositions and structural transformations of ceramic samples with increasing levels of Amorphous Boron additive (M to M9B). The sharp peaks observed in the wall tile sample (M) are attributed to quartz (01-089-1961) and kaolinite (01-089-1460), which are primary constituents of ceramic materials. This indicates that the structure of the sample is well-crystallized, as reported by An et al.[7]. As boron is incorporated, the peak intensities for these crystalline phases diminish slightly, and new peaks associated with boron-related phases (e.g., boron: 00-011-0618) begin to emerge. This suggests that boron contributes to the formation of secondary phases, enhancing sintering activity and promoting phase transformations [8]. Research has also demonstrated that boron can enhance the formation of liquid phases during sintering, which aids in the diffusion processes necessary for achieving high-density ceramics[9].

It is noteworthy that at elevated boron levels (M7B and M9B), the peak intensities broaden and diminish in sharpness, indicative of the inception of a glassy phase. The presence of boron as a addition, material has been observed to reduce the sintering temperature, thereby facilitating the process of localized melting and the formation of an amorphous matrix [10]. Furthermore, the observed shifts in the peak positions can be attributed to lattice distortions caused by the incorporation of boron, which is consistent with the findings of previous studies on boron-addition, ceramics[11], [12]. This phase evolution, characterized by a reduction in crystallinity and an increase in amorphous content, is in accordance with the anticipated behaviour of boron additives in ceramic systems, thereby underscoring their influence on thermal, mechanical, and structural properties.

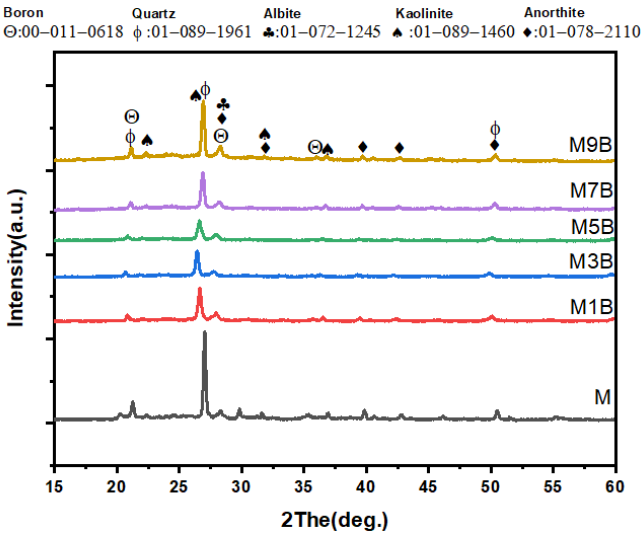

Figure 3. XRD analysis of ceramic samples (M–M9B) with increasing Amorphous Boron

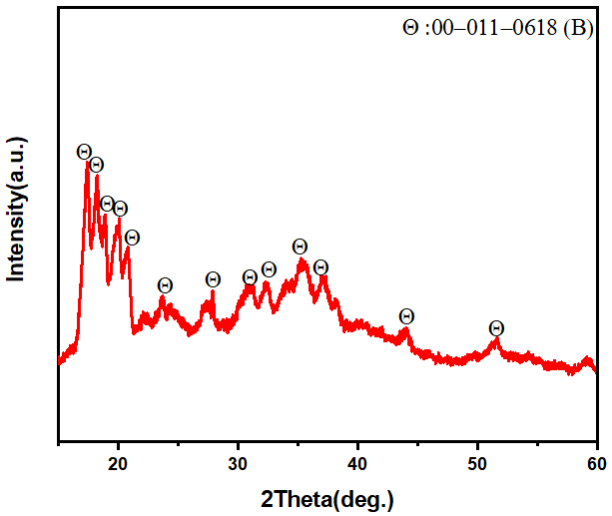

Figure 4. XRD analysis of Amorphous Boron powder

In Figure 5., SEM analysis of samples with increasing amorphous boron content shows significant changes in microstructure that are directly related to the amount of additive incorporated into the ceramic matrix. At a lower concentration of boron M1B, the surface morphology appears relatively smooth and compact with minimal porosity, indicating that the structural integrity of the tile remains largely intact. This low porosity is indicative of increased densification, which is known to improve mechanical strength and reduce

water absorption in ceramic materials[13]. As the boron content is increased to M3B and M5B, the microstructure begins to exhibit more pronounced pore formation and voids, a common phenomenon when excessive additives disrupt the homogeneity of the ceramic matrix. The much larger pores at these concentrations can adversely affect both mechanical and thermal properties, as the voids act as stress concentration points, reducing tensile strength and increasing thermal resistance[14]. This trend is even more evident at higher boron contents of M7B and M9B, where SEM images show extensive fragmentation and significant porosity across the surface. These defects are probably due to the excessive boron oxide interfering with the sintering process, resulting in failure to achieve full densification and subsequent loss of structural cohesion[15]. Studies have shown that high porosity in ceramic materials can lead to a drastic reduction in mechanical performance as well as increased thermal conductivity, which could limit the use of the tile in environments requiring high strength or insulation[16]. This finding is consistent with previous research, which has highlighted the importance of optimizing additive levels to improve ceramic performance without introducing detrimental porosity or defects.

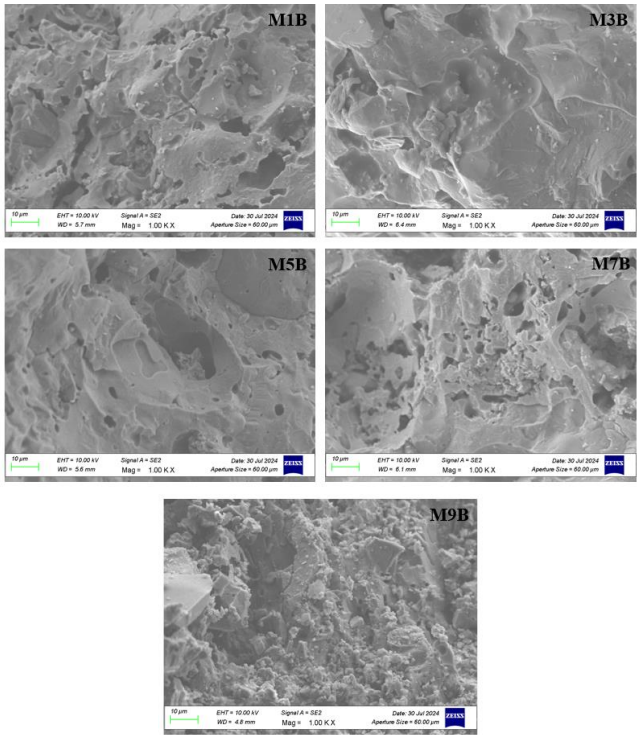

Figure 5. Morphological image of the produced samples

In Figure 6., the Energy Dispersive Spectroscopy (EDS) analysis carried out on the ceramic samples with different amounts of amorphous boron (1%, 5% and 9%) provides crucial insights into the elemental distribution and its influence on the microstructure. In the M1B sample, the boron distribution is sparse, with small and uniformly distributed clusters, indicating that the low boron content does not significantly disrupt the ceramic matrix. This distribution suggests improved mechanical properties due to the homogeneous structure and low porosity. The abundant presence of silicon (Si), oxygen (O) and aluminium (Al) in this sample indicates the dominance of silicate and mullite phases, both of which are critical for maintaining structural integrity in ceramics[17]. Calcium (Ca) and magnesium (Mg) are also moderately distributed and contribute to the vitreous phase.

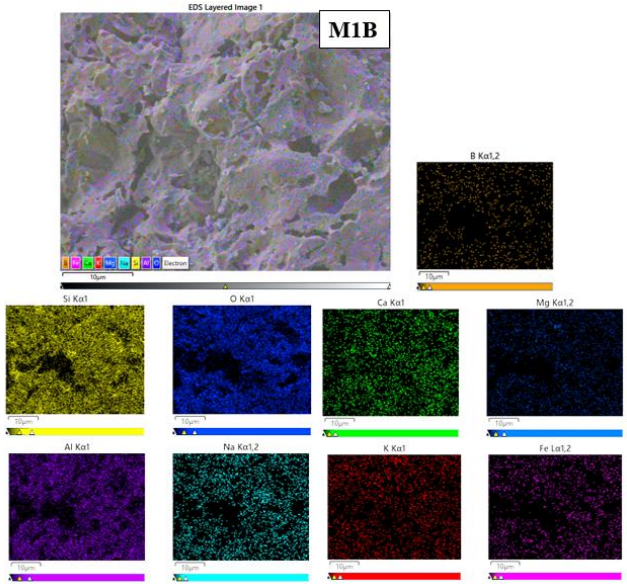

Figure 6. SEM-EDS analysis of the produced sample(M1B)

In contrast, in Figure 7. sample M5B shows a more pronounced boron distribution with larger and more frequent clusters. This indicates a higher incorporation of boron into the matrix, which could lead to improved thermal properties due to the formation of boron oxide phases. However, the presence of larger voids and pores becomes more apparent at this concentration, potentially reducing the mechanical strength and thermal conductivity of the material. The distribution of Si, O and Al remains relatively constant, ensuring that the primary silicate matrix is maintained. Meanwhile, Ca, Mg and Fe show some changes in distribution, which may result in a more glassy phase, affecting the thermal expansion and stability of the ceramic.

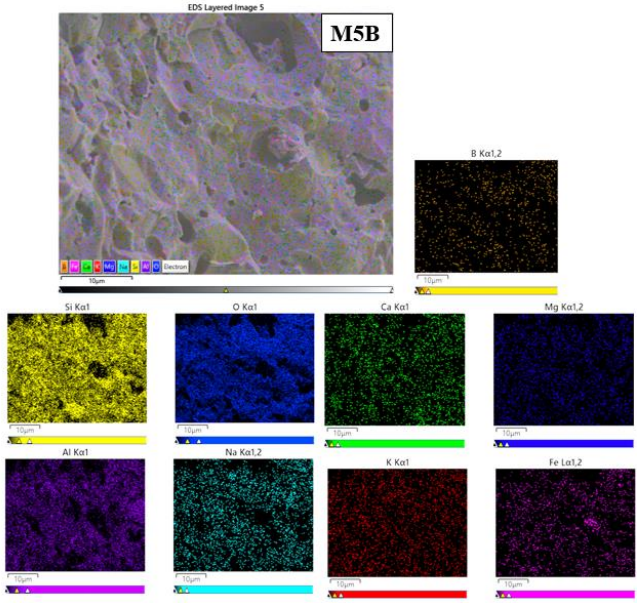

Figure 7. SEM-EDS analysis of the produced sample(M5B)

When the boron content reaches 9% (M9B) the boron distribution becomes dense and clustered, indicating a significant increase in boron throughout the sample. Whilst this may improve certain thermal properties, the EDS images show significant porosity and fragmentation which could severely affect the mechanical properties of the material[16]. The high boron content is likely to interfere with the sintering process, preventing complete densification and resulting in a more fragile structure. The uniform distribution of Si, O and Al throughout the matrix suggests that the primary silicate structure remains intact, but the increased porosity may undermine its effectiveness[18]. The presence of Ca, Mg and Fe in localized regions further suggests that phase separation may occur at this high boron concentration, leading to weakened structural regions and reduced overall strength[19].

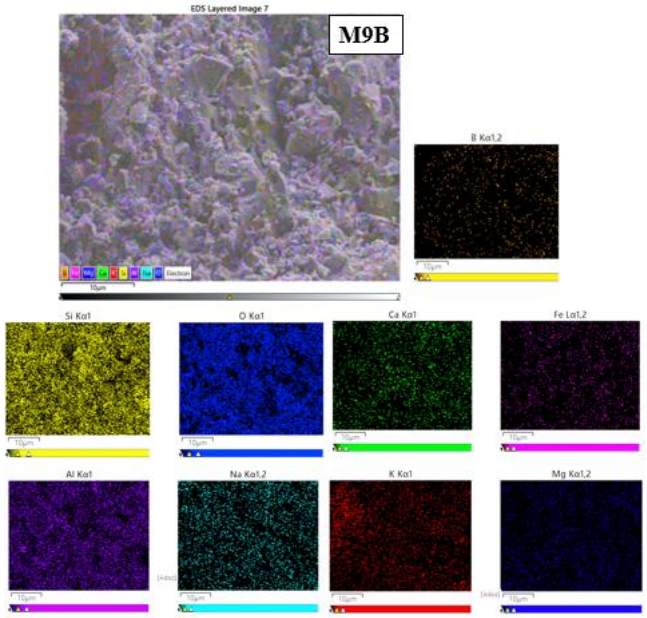

Figure 8. SEM-EDS analysis of the produced sample(M9B)

The EDS analysis confirms that while lower boron concentrations (around 1-3%) contribute to a well-distributed and dense ceramic matrix, higher concentrations (5-9%) introduce significant porosity and phase separation. These structural changes are likely to affect both the mechanical strength and thermal performance of the material. The presence of elements such as Ca, Mg, and Al plays a critical role in stabilizing the matrix and contributing to the material's overall properties[16]. However, excessive boron content disrupts this balance, leading to undesirable phase interactions and microstructural defects, as evidenced in the M9B sample [20].These findings align with previous studies that highlight the importance of optimizing boron content to enhance ceramic performance while avoiding detrimental effects associated with high porosity and structural weaknesses[10].

The bulk density trend shown in Figure 9. demonstrates the influence of amorphous boron content on the densification behavior of ceramic wall tile mixtures. The wall tile sample (M) displays a relatively low bulk density of approximately 1.8 g/cm^3, indicative of elevated porosity and inadequate densification. The incorporation of boron markedly enhances the bulk density, with values rising to 2.0 g/cm^3 M1B and subsequently to 2.1 g/cm^3 M3B. This phenomenon can be attributed to the fluxing effect of boron during sintering, which facilitates enhanced particle rearrangement and pore elimination[21]. At M5B, the peak bulk density of 2.3 g/cm^3 is observed, which is indicative of optimal densification. However, an increase in the boron content to M7B and M9B is accompanied by a decline in the bulk density, which is likely due to the excessive formation of a glassy phase. This disrupts microstructural cohesion and introduces

localized voids. This trend is consistent with previous findings in boron-addition, ceramics, which indicate that excessive additive content negatively affects densification and mechanical properties[22].

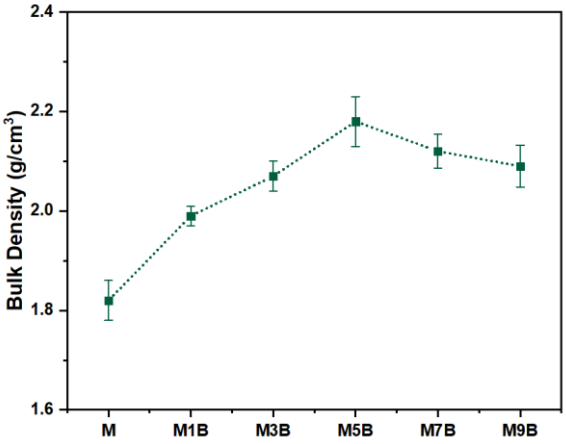

Figure 9. Bulk density of the produced samples

The physical properties of ceramic wall tiles, determined through Archimedes testing, are presented in Table 2. It emphasizes the influence of amorphous boron on their physical characteristics. As the concentrations of amorphous boron rise, Table 2 shows that the wall tile ceramic gradually becomes denser, especially up to M5B. This is because the apparent porosity and water absorption decrease. The reduction in exterior volume at higher boron concentrations (M7B and M9B) corroborates the development of a glassy phase inside the structure. SEM analysis also confirmed the presence of the glassy phase in the structure, as shown in Figure 7.

Table 2. Archimedes test results for physical properties

	Exterior volume(cm^3)	Apparent porosity (%)	Water absorption (%)
M	4.00±0.04	29.36±0.01	16.07±0.12
M1B	3.62±0.12	20.19±0.07	10.08±0.04
M3B	3.66±0.16	7.05±0.13	3.40±0.10
M5B	3.33±0.28	2.39±0.24	2.09±0.11
M7B	3.50±0.17	2.19±0.21	2.29±0.27
M9B	3.31±0.42	2.03±0.39	2.05±0.53

In Figure 10. illustrates the impact of amorphous boron addition on the mechanical performance of wall tile ceramics, as evidenced by the compressive strength results. The wall tile sample without boron (M) displays a markedly diminished compressive strength of 28.096 MPa, suggestive of a porous structure with constrained densification. Upon the addition of 1% amorphous boron (M1B), a significant increase in compressive strength was observed, reaching 210.813 MPa. This result indicates the beneficial effect of boron as a fluxing agent. This improvement is consistent with the findings of the literature, which indicate that boron enhances sintering by promoting densification and reducing porosity[5].

As the boron content increases to M3B, the compressive strength reaches its maximum at 214.794 MPa, indicating an optimal concentration where boron effectively fills voids and improves intergranular bonding. This observation is consistent with the findings of previous research, which have demonstrated that moderate boron additions can enhance both microstructural cohesion and mechanical strength [16]. Nevertheless, the addition of further boron beyond the M3B level results in a gradual decline in compressive strength. At M5B and M7B, the compressive strength shows a slight decline, reaching 203.748 MPa and 203.583 MPa, respectively. This indicates the onset of diminishing returns. This trend is likely due to the formation of glassy phases and increased porosity, as excessive boron disrupts the microstructural continuity. At the highest boron concentration of M9B, a further decrease in compressive strength is observed, reaching 197.463 MPa. This suggests a significant decline in mechanical integrity. The observed reduction in strength can be attributed to the excessive introduction of boron, which has been shown to result in microstructural defects such as pore clustering and glassy phase dominance. These defects have been demonstrated to weaken load-bearing capabilities under compressive stresses[14], [23].

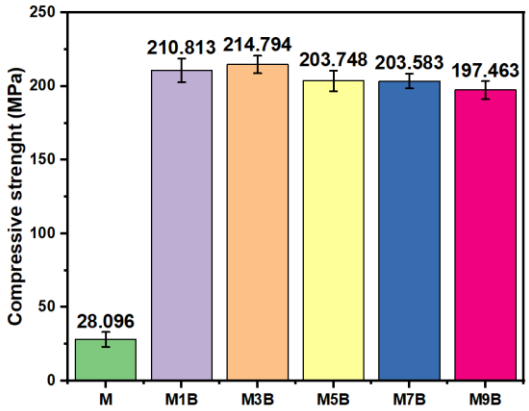

Figure 10. Compressive strength analysis of the produced samples

The thermal conductivity (k) results shown in Figure 11. illustrate the effect of increasing amorphous boron content on the ceramic wall tile mix. The wall tile (M) without boron has a thermal conductivity of 0.61 W/m.K, indicative of its high porosity and limited densification. The incorporation of boron has been observed to increase thermal conductivity, with values

reaching 0.92 W/m.K at 3% boron (M3B) and peaking at 1.02 W/m.K at 5% boron (M5B). This improvement can be attributed to the role of boron as a fluxing agent during the sintering process, which promotes densification and reduces porosity, thereby facilitating phonon transport. However, beyond 5% boron content, thermal conductivity declines sharply to 0.65 W/m.K at 7% (M7B) and 0.48 W/m.K at 9% (M9B). This reduction is likely due to the excessive formation of a glassy phase, which disrupts the crystalline structure and impedes phonon pathways, thereby diminishing heat transfer efficiency[21]. Excessive boron can also lead to increased porosity and structural heterogeneity, further reducing thermal conductivity. Therefore, while moderate boron additions enhance thermal conductivity through improved densification, higher concentrations induce structural changes that adversely affect thermal performance[24].

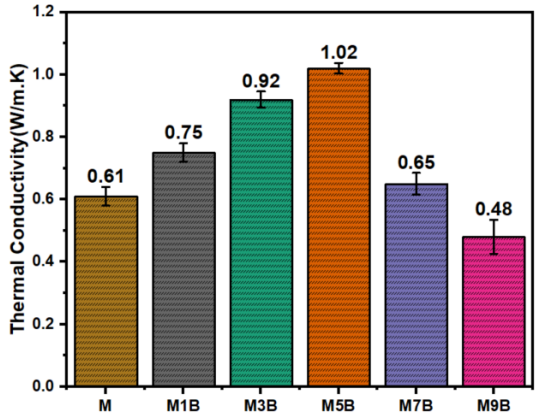

Figure 11. Thermal conductivity values of the produced samples

4. Conclusion

The addition of amorphous boron to wall tile ceramics has significantly affected the structural, mechanical, and thermal properties of the material by altering its densification behavior, phase composition, and microstructural integrity during sintering.

The amorphous boron additive functions as a flux during the sintering process, facilitating the formation of particle bonds. This results in a reduction in the porosity of the amorphous boron-doped wall tile ceramics produced. This phenomenon is particularly evident in the samples produced with 3%–5% amorphous boron added. It has been observed that elevated boron concentrations (7%–9%) facilitate the formation of a glassy phase, which has a considerable impact on the phase composition of the material. The compression test findings indicate that the compressive strength of wall tile ceramics with a 3–5% amorphous boron addition was markedly enhanced. This enhancement is ascribed to the augmented bulk density and diminished porosity. At higher concentrations (7%–9%), the glassy phase induced internal stresses and microcracks, reducing mechanical integrity. Compressive strength analysis showed that amorphous boron additions up to 5% had a beneficial effect. Moderate amorphous boron additions (%3–%5) were found to maintain acceptable thermal conductivity, while higher concentrations (%7–%9) reduced thermal performance. The formation of a glassy phase increased material heterogeneity, disrupting heat transfer pathways and lowering thermal efficiency.

5. Acknowledge

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6. References

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