

ORIGINAL RESEARCH ARTICLE

Effect of evaporation boat waste particle size on the characterization of crucible specimens

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ABSTRACT

The demand for high-performance crucibles in metal casting and smelting industries has led to an increasing interest in alternative materials due to the limitations of traditional graphite-based crucibles. Evaporation boat waste, primarily composed of boron nitride (BN) and titanium diboride (TiB₂), offers a sustainable alternative but has not been fully explored for crucible production. Additionally, the influence of particle size on material properties such as density, porosity, and macrostructure remains under-researched. This study aims to address this gap by examining the effects of particle size and sodium silicate binder on the physical properties of crucibles made from evaporation boat waste. Crucibles were prepared with varying particle sizes (100, 120, 140, 170, and 200 mesh) and a 15% sodium silicate binder. Density, porosity, and macrostructure were analyzed, showing that smaller particle sizes led to higher density and lower porosity. Optical macrostructure analysis indicated a more homogeneous and compact structure for finer particles. The results showed that the 200 mesh particle size achieved the highest density of 2.138 g/cm³, representing a 5.1% increase over the lowest density of 2.0343 g/cm³. The 200 mesh sample also exhibited the lowest porosity of 1.14%, a 2.7% decrease from the largest particle size.

Keywords: evaporation boat waste; crucible; boron nitride; titanium diboride; particle size; sodium silicate; density; porosity

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1. Introduction

The swift growth of high-temperature industrial sectors, particularly metal casting and smelting, has heightened demand for superior refractory materials with reliable thermal stability. Crucibles are essential for these operations, as they withstand extreme heat. Despite its prevalent use as a traditional crucible material, graphite's durability and service life are constrained under specific operational conditions. This constraint has spurred the development of new crucible materials that combine enhanced performance, cost-effectiveness, and environmental sustainability.

Evaporation boat waste presents significant promise as an alternative raw material for crucible production. This waste mostly comprises boron nitride (BN) and titanium diboride (TiB₂), recognized for their high-temperature stability, refractory properties, and oxidation resistance^[1]. Notwithstanding these beneficial characteristics, utilized evaporation boats are frequently classified as industrial trash post-

service. Transforming this debris into crucible specimens offers a pragmatic approach for waste minimization while augmenting the value of discarded high-performance materials. Sodium silicate, commonly known as waterglass, is pertinent in this context as it serves as a stable inorganic binder in ceramic and refractory systems^[2].

Particle size is a critical characteristic that significantly impacts crucible performance. Particle refining can enhance packing behavior, increase density, and reduce porosity by improving particle interactions and facilitating a more uniform distribution of the binder. The manufacturing process, encompassing mixing, pressing, and sintering, significantly influences the quality of the final product, as these processes govern compaction, particle organization, and structural integrity^[3,4]. The behavior of evaporation boat waste cannot be presumed to be equivalent to that of traditional alumina-based ceramics. BN/TiB₂-based materials possess unique chemical compositions, sintering behaviors, and interactions with sodium silicate binders, necessitating direct assessment.

The integration of evaporation boat waste and sodium silicate binder is crucial for the production of crucibles that are both technically efficient and environmentally sustainable^[5]. Prior research has shown that reduced particle size can improve mechanical properties, such as hardness and impact resistance. Simultaneously, meticulous regulation of particle size and binder content can enhance density and porosity, which are essential for sustaining crucible performance under high-temperature conditions^[1,6].

This study investigates the influence of particle size on the production of sodium-silicate-bonded crucibles originating from evaporation boat waste. The inquiry highlights three primary physical attributes: density, apparent porosity, and macrostructure. This study aims to identify optimal processing parameters for manufacturing crucibles with enhanced structural and physical properties by utilizing various waste powder fractions and sodium silicate as the binder. The findings are expected to facilitate the development of more efficient, sustainable refractory materials derived from the reutilization of industrial waste.

This study builds upon previous investigations of particle-size influences in crucible materials by examining a waste-derived BN/TiB₂ combination integrated with an inorganic binder. It provides a detailed understanding of the impact of particle size on the ultimate properties of crucible specimens and may serve as a basis for advancing refractory material technology^[7,8]. The study advances sustainable materials engineering by facilitating the recycling of industrial waste and enhancing the design of materials for high-temperature applications.

While the utilization of evaporation boat waste has been examined in ceramic applications^[9], its potential as a primary raw material for crucible manufacturing has not been adequately addressed. Similarly, although the impact of particle size on density and porosity is well recognized^[10,11], there has been scant focus on sodium-silicate-bonded crucibles derived from evaporation boat waste. This study's originality is in assessing 100–200 mesh evaporation boat waste powder fractions as the primary variable in crucible production, with density, apparent porosity, and macrostructure analyzed via particle-packing and binder-distribution mechanisms^[12].

2. Materials and methods

This study utilized evaporation boat waste as the principal raw material. The waste primarily comprised boron nitride (BN) and titanium diboride (TiB₂), each constituting 50% of the total composition ^[1]. These compounds are widely used in the metalized plastic sector due to their ability to withstand elevated-temperature operating conditions. The waste material examined in this study was obtained from an industrial facility operated by PT X in Sidoarjo, East Java, Indonesia. Sodium silicate (**Figure 1**), often known as waterglass, was chosen as the binder for its durability and bonding efficacy in refractory material systems. Its integration

is linked to improved composite structural integrity and increased resilience to thermal stress. Sodium silicate is deemed suitable to produce high-performance crucibles^[13,14].



Figure 1. Sodium silicate.

Before additional processing, the evaporation boat waste was pulverized using a hammer mill. The resultant powder was then subjected to mechanical sieving to obtain controlled particle-size fractions (**Figure 2**). The material was categorized into five mesh sizes: 100, 120, 140, 170, and 200 mesh^[3]. These fractions approximately correspond to nominal sieve apertures of 150, 125, 106, 90, and 75 μm , respectively^[6]. **Table 1** displays the particle size characterization values of the evaporation boat waste powder utilized as a primary material in this study. A dry sieving method was employed to reduce particle agglomeration induced by moisture before mixing with sodium silicate. Regulating the particle size distribution is crucial as it affects the ultimate properties of the crucible specimens, including its density, porosity, mechanical strength, and heat resistance^[11,15].



Figure 2. Sieving process.

Table 1. Mesh size and nominal sieve opening of evaporation boat waste powder.

Mesh size	Approximate nominal sieve opening (μm)	Role in this study
100	150	Coarsest fraction
120	125	Intermediate coarse fraction
140	106	Intermediate fraction
170	90	Intermediate fine fraction
200	75	Finest fraction

The waste powder from the evaporation boat was combined with a sodium silicate binder via a dry-mixing procedure. The binder percentage was set at 15 wt% relative to the waste particles, in accordance with optimization results from prior research on sodium-silicate-bonded ceramics and crucible systems formed from

evaporation boat waste^[6]. After mixing, the resultant slurry was placed into crucible molds and compacted at 40 MPa using a hydraulic press as illustrated in **Figure 3**^[9]. The pressing stage aimed to facilitate uniform particle arrangement and sufficient initial densification. Previous studies have shown that binder concentration and compaction pressure are essential processing factors for producing refractory materials with dependable structural integrity^[16].



Figure 3. Hydraulic press machine.

The molded specimens were then cured at 100 °C for 2 hours to facilitate the hardening of the sodium silicate binder^[12]. The curing settings were established based on guidelines from previous research on sodium-silicate-bonded ceramic systems^[17,18]. After curing, the crucibles were heat-treated in a furnace at 1150 °C for 2 hours^[13]. The chosen temperature indicates a moderate sintering state aimed at consolidating the sodium-silicate-bonded BN/TiB₂ waste body, rather than achieving full densification of the TiB₂ ceramics. Full densification of TiB₂ generally necessitates significantly elevated processing temperatures or the incorporation of certain sintering additives^[15,19].

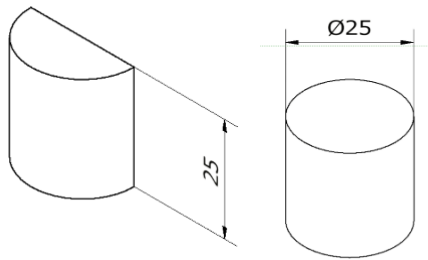


Figure 4. Specimen geometry (mm).

The assessment of bulk density and apparent porosity was conducted using a water-immersion method. Before testing, the specimens (**Figure 4**) were desiccated and weighed to ascertain their dry mass. They were subsequently submerged in water for 24 hours to facilitate water infiltration into the open pores. Following immersion, the specimens were extracted and meticulously cleaned to remove excess surface water while preserving pore saturation. The mass of each item was subsequently documented under saturated surface-dry conditions. The saturated specimens were weighed while submerged in water to determine the immersed mass required to calculate bulk density and apparent porosity. The bulk density was calculated using Equation (1):

$$\rho_b = [M_d / (M_{sat} - M_{sub})] \times \rho_w \quad (1)$$

where ρ_b is the bulk density (g/cm^3), M_d is the dry mass of the specimen in air (g), M_{sat} is the saturated surface-dry mass in air (g), M_{sub} is the saturated mass while suspended in water (g), and ρ_w is the density of water (g/cm^3). The apparent porosity was calculated using Equation (2):

$$P_a = [(M_{\text{sat}} - M_d) / (M_{\text{sat}} - M_{\text{sub}})] \times 100\% \quad (2)$$

where P_a is the apparent porosity (%), and the mass terms are as defined above. The equations were rewritten to eliminate the previous undefined variables and to distinguish dry, saturated, and submerged weights clearly. This study conducted three times of density and porosity testing, and the mean value for every measurement was determined. The surface macrostructure of the specimens was examined using an optical microscope with $200\times$ magnification. The observations were performed at the middle, bottom, and top sections of each specimen to assess possible positional differences in surface uniformity^[20].

3. Results

The findings demonstrate that a smaller particle size improved the physical performance of the crucible specimens by increasing density and decreasing apparent porosity. **Figure 5** illustrates a density increase from $2.0343 \text{ g}/\text{cm}^3$ for the 100-mesh fraction to $2.1380 \text{ g}/\text{cm}^3$ for the 200-mesh fraction, reflecting an enhancement of roughly 5.10%. Consistent with this finding, **Figure 6** demonstrates that apparent porosity decreased from 3.85% to 1.14% as particle size diminished.

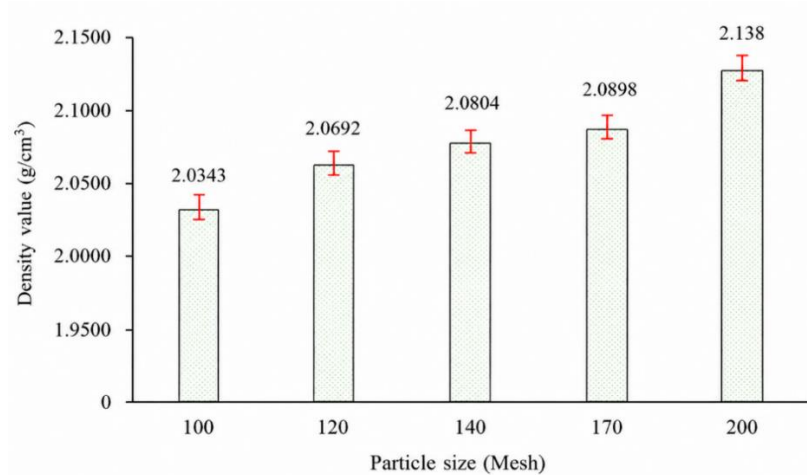


Figure 5. Bulk density of crucible specimens as a function of evaporation boat waste particle size.

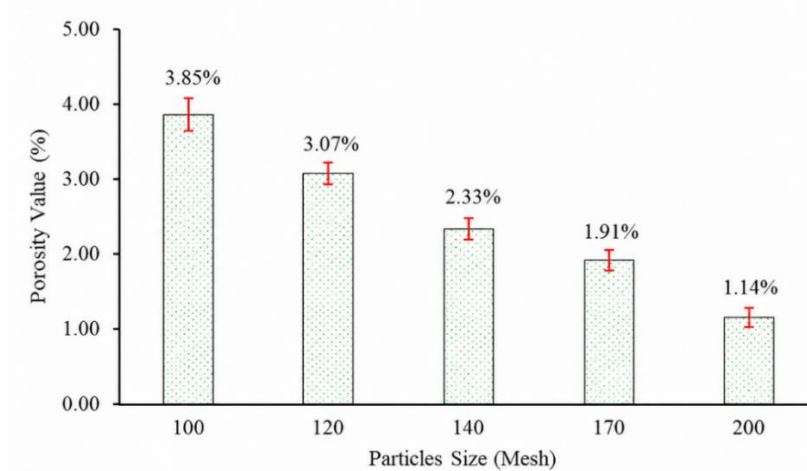


Figure 6. Apparent porosity of crucible specimens as a function of evaporation boat waste particle size.

The examination of the optical macrostructure revealed that variations in particle size significantly influenced the surface appearance of the crucible specimens. **Figure 7** illustrates that the 100-mesh specimen had a rather coarse surface, uneven particle distribution, and prominent dark regions that may indicate open pores or interstitial spaces among particles. In contrast, the 120-mesh specimen depicted in **Figure 8** exhibited a more refined surface texture, characterized by a denser arrangement of smaller particles. Enhanced improvement was observed in the 140-mesh specimen, as illustrated in **Figure 9**, where the surface was more uniform and structurally cohesive.

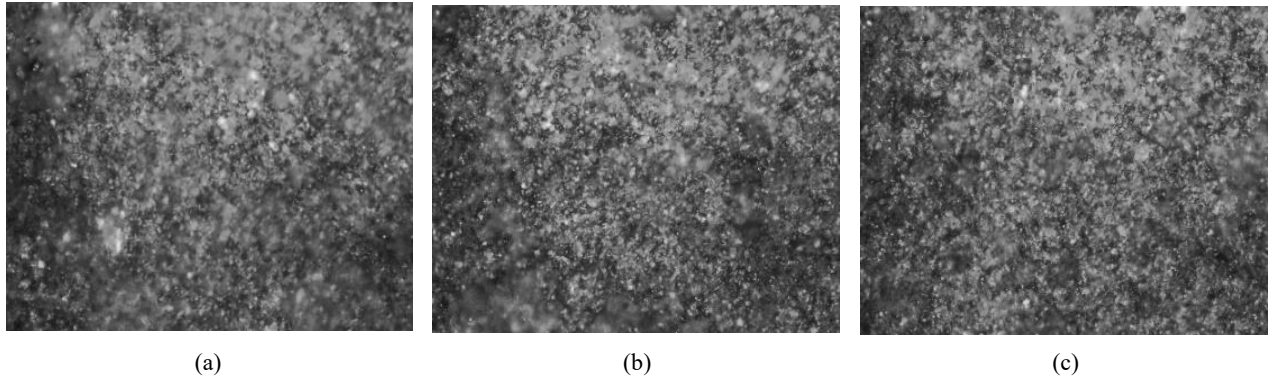


Figure 7. Optical macrostructure of the 100-mesh specimen at 200 $\times$ magnification: (a) middle view, (b) bottom view, and (c) top view.

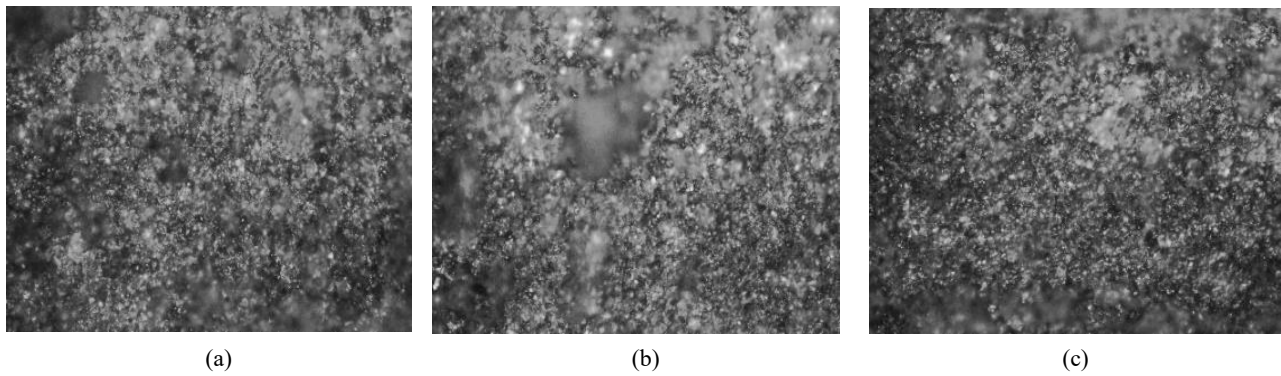


Figure 8. Optical macrostructure of the 120-mesh specimen at 200 $\times$ magnification: (a) middle view, (b) bottom view, and (c) top view.

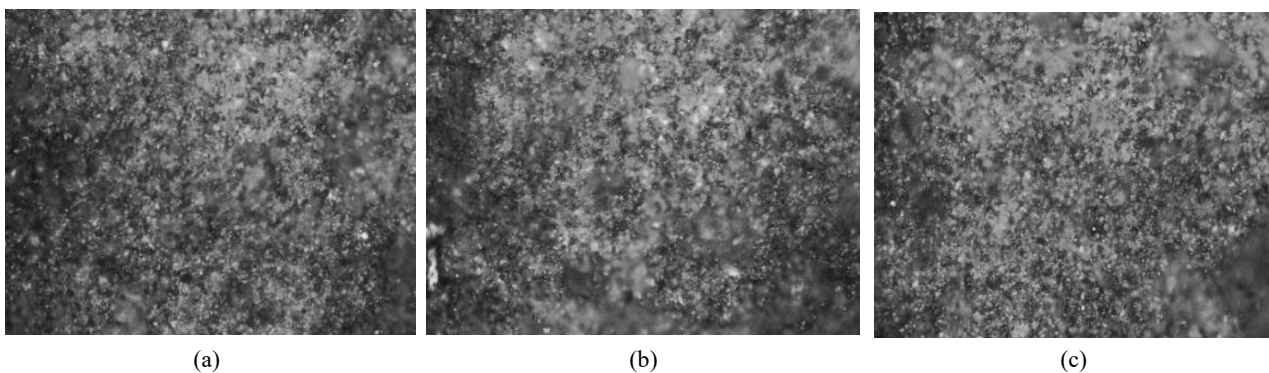


Figure 9. Optical macrostructure of the 140-mesh specimen at 200 $\times$ magnification: (a) middle view, (b) bottom view, and (c) top view.

The 170-mesh specimen, illustrated in **Figure 10**, exhibited a denser particle configuration and a smoother surface compared to specimens made with lower mesh sizes. The most polished surface was noted in the 200-mesh specimen depicted in **Figure 11**. This specimen demonstrated the highest optical density and smoothness, with intergranular gaps nearly imperceptible. Consequently, it yielded the most uniform surface appearance among all specimens. The results indicate that increasing the mesh number improved the surface morphology of the crucible specimens. Smaller particle fractions enhanced packing efficiency, minimized observable

interparticle voids, and produced a more consistent texture. The upper, central, and lower portions were analyzed to evaluate positional variations within each specimen. Coarser particle fractions exhibited greater variability in surface roughness and local void distribution across these locations, indicating less efficient compaction and binder distribution. Conversely, finer particle fractions exhibited more uniform surface characteristics across all examined locations, indicating more regular particle packing and a more uniform distribution of the sodium silicate binder.

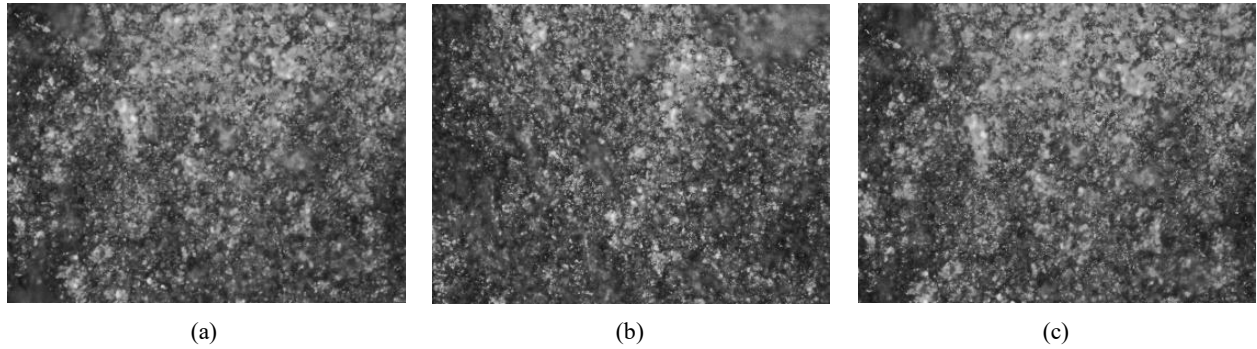


Figure 10. Optical macrostructure of the 170-mesh specimen at 200× magnification: (a) middle view, (b) bottom view, and (c) top view.

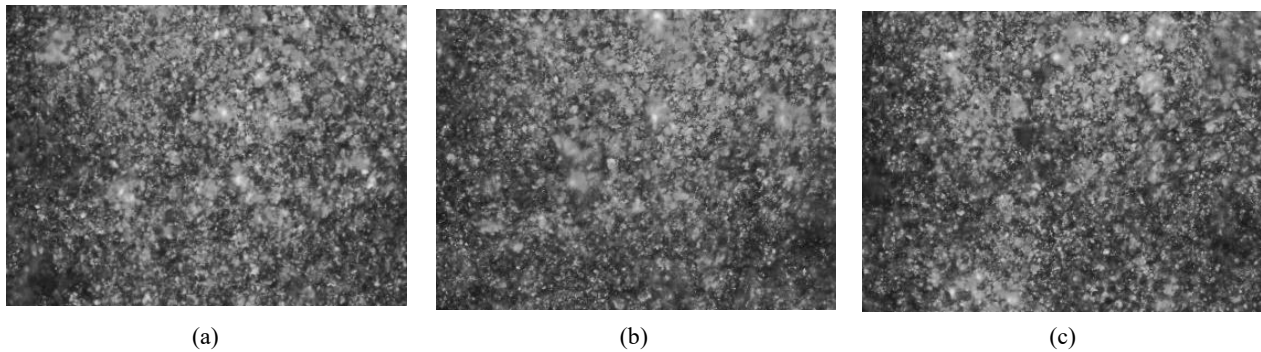


Figure 11. Optical macrostructure of the 200-mesh specimen at 200× magnification: (a) middle view, (b) bottom view, and (c) top.

4. Discussion

The results corroborate earlier research demonstrating that particle size and binder content are critical determinants of ceramic material properties. Generally, finer particles increase density and boost mechanical performance by improving interparticle interaction and minimizing internal voids^[2,3]. The observed trend indicates that reducing the particle size of the evaporation boat waste improved the density of the crucible specimens. This phenomenon can be elucidated by powder-packing principles, in which finer particles occupy smaller voids and establish more contact points within a given volume. During compression, these particles can more effectively reorganize into interparticle voids, thus diminishing linked pore channels. As a result, a more compact green body is formed before heat treatment, leading to increased bulk density and decreased apparent porosity. The density increased from 2.0343 to 2.1380 g/cm³, a significant change in the context of crucible manufacturing from recovered trash. The enhancement was attained with a constant sodium silicate concentration and a moderate sintering temperature, suggesting that particle refinement alone facilitated improved consolidation. The associated reduction in apparent porosity further substantiates that the finer powder generated a more cohesive solid structure. In porous ceramic materials, porosity influences several functional properties, including strength, permeability, thermal conductivity, and resistance to molten-metal infiltration. Consequently, minimizing open porosity is essential for crucible efficacy, since interconnected pores can promote liquid infiltration, chemical deterioration, and crack development after successive hot exposure.

Sodium silicate serves as an inorganic binder in this system, creating surface coatings and connecting bridges between particles upon curing and heat treatment. The increased surface area of finer particles offers additional bonding sites, yet it may also elevate the binder requirement for thorough coverage. The binder content was consistently maintained at 15 wt.% across all particle sizes, indicating a relationship between particle packing efficiency and binder distribution. The enhanced density of the 200-mesh specimen suggests that superior particle packing, and increased contact area were more significant than the possible disadvantage of heightened binder requirements within the examined range.

The macrostructural observations align with the observed trends in density and porosity. Coarse particle fractions yielded rougher surfaces and more pronounced discontinuities, whereas finer fractions generated smoother, denser, and more uniform surfaces. More pronounced disparities among the middle, bottom, and top regions in the 100- mesh and 120-mesh specimens indicate that coarser powders exhibited greater susceptibility to localized packing heterogeneity during compaction.

5. Conclusion

This study examined the impact of evaporation boat waste particle size on the density, apparent porosity, and optical macrostructure of crucible specimens bonded with sodium silicate. Within the examined range of 100–200 mesh, finer particle fractions improved the density of the specimens. The maximum density of 2.1380 g/cm³ was achieved with the 200-mesh specimen, whilst the minimum density of 2.0343 g/cm³ was noted for the 100-mesh specimen. This indicates an approximate density enhancement of 5.10% from the coarsest to the finest fraction. A gradual decrease in perceived porosity was noted with diminishing particle size. The porosity diminished from 3.85% in the 100-mesh specimen to 1.14% in the 200-mesh specimen. The negative relationship between density and apparent porosity indicates that smaller evaporation boat waste particles facilitated more effective packing, hence decreasing the volume of open pores available to water under the specified fabrication conditions.

The examination of the optical macrostructure at 200× magnification further corroborated this pattern. Specimens made with finer particles showed smoother, more uniform surfaces, whereas those prepared with coarser particles exhibited rougher textures and more pronounced discontinuities. These enhancements are linked to improved particle packing, increased interparticle interaction, and more efficient bonding-bridge creation by the sodium silicate binder in the finer powder fractions. Consequently, the 200-mesh fraction signifies the most advantageous particle-size condition among those analyzed for the prospective creation of crucible specimens originating from evaporation boat waste.

Author contributions

Conceptualization, Rusiyanto Rusiyanto and Rifky Ismail; methodology, Athanasius Priharyoto Bayuseno; software, Deni Fajar Fitriyana; validation, Wirawan Sumbodo, Athanasius Priharyoto Bayuseno and Rifky Ismail; formal analysis, Willy Gumilang Toh; investigation, Deni Fajar Fitriyana; resources, Rusiyanto Rusiyanto; data curation, Willy Gumilang Toh; writing—original draft preparation, Ninda Kurniadi; writing—review and editing, Ninda Kurniadi; visualization, Ninda Kurniadi; supervision, Rifky Ismail; project administration, Rifky Ismail; funding acquisition, Athanasius Priharyoto Bayuseno. All authors have read and agreed to the published version of the manuscript.

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Conflict of interest

The authors declare no conflict of interest.

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