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Influence of Mixing Time on the Hardness and Structure of Local Clay-Based Crucibles

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Abstract. Although clay crucibles are frequently utilized in regional industries, their inadequate mechanical strength often causes durability issues. This study investigates the influence of mixing duration on the Vickers hardness and macrostructure of crucibles composed of local clay, kaolin, and molasses. The composition was made up of 47.5% clay, 47.5% kaolin, and 5% molasses as a binder, with 15% water added relative to the total weight. Durations of 15, 30, and 45 minutes were evaluated to determine their impact on material qualities. The findings indicated a positive relationship between mixing duration and hardness. At 15 minutes, the mean hardness was 4.1 HV, which escalated to 8.5 HV at 30 minutes and 12.4 HV at 45 minutes. The increased hardness with extended mixing durations indicates a more homogeneous particle dispersion and enhanced bonding among the raw ingredients. The findings suggest that increasing the mixing time can elevate the quality and longevity of locally manufactured crucibles, rendering them more appropriate for small-scale metallurgical applications.

Keywords: crucible, local clay, kaolin, mixing time, binder, refractory material, Vickers hardness.

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1. Introduction

Clay is derived from the weathering of feldspathic rocks, including granite and igneous formations, and is classified under the phyllosilicate family, distinguished by hydrated aluminosilicate layers [1,2]. Clay minerals in natural soil usually exist in pure form; instead, they are typically found as intricate combinations of various mineral groups. Their fundamental structural unit comprises interconnected tetrahedral silica and octahedral alumina sheets. Due to their unique physicochemical characteristics, availability, and comparatively low cost, clays are extensively utilized as fundamental raw materials in the ceramic industry [1–4].

Since ancient times Indonesian society has widely utilized clay as a raw material for the tile industry, bricks, pottery, and ceramics. Local clay has a very high level of heat resistance that makes pottery and ceramic products not easily broken during the combustion process. Chemically clay does contain aluminum oxide (Al_2O_3) dan silica (SiO_2) as the main element making it suitable as a fire-resistant material [5]. Based on the nature of the soil, clay is suitable for use as a refractory material that can be applied in high temperatures, such as in the crucible in the metal casting industry.

A crucible is a container or cup made of ceramic or metal that is used to melt metal and alloy materials at very high temperatures in the metal casting process [6]. To be able to make a crucible that has good quality, there are several factors that influence it such as the constituent materials, particle size, binder, as well as the temperature and length of the combustion process. Generally, crucibles that are widely used in the metal casting industry in Indonesia are made from graphite. However, crucibles made from graphite have the disadvantage of being easily broken and cracked when receiving temperature spikes, and can only last for 10 times fine melting, while in rough melting it can only last 5 times [7]. Seeing the potential of local clay, it is necessary to study the manufacture of crucible made from local clay.

This study combined kaolin as an additional material with local clay in crucible manufacturing. Kaolin is characterized by low iron content, predominantly hydrous aluminum silicate, and inorganic oxides and trace amounts of heavy metal ions [8,9]. The primary oxides are SiO_2 and Al_2O_3, with secondary elements comprising Fe_2O_3, CaO, MgO, TiO_2, and Na_2O. Kaolin demonstrates minimal shrinkage, insufficient dry strength, and superior refractory characteristics, with a melting point reaching 1785 °C, rendering it appropriate for improving the heat resistance of crucibles [10–13]. This research additionally utilized molasses as an organic ingredient [14,15]. Molasses, a liquid by-product of the sugar industry, enhances homogenization, strengthens particle bonding, and prolongs binding time in the mixture.

Unfortunately, there are still some problems in the crucible manufacturing process, one of which is in the material mixing process. The mixing process is a process of combining several different materials into one to produce the desired output. Being an important process in various industries because the mixing process is a process that will determine the quality of a product [16]. Several things can affect the level of the mixture, the parameters of the mixing level procedure can be influenced by the type of machine used, mixing speed, air pressure in the machine, the order of adding ingredients, mixing temperature, and mixing time [17].

Prolonged mixing time improves the material's uniformity, density, and compactness. Nonetheless, this prolonged mixing duration leads to a decrease in the material's workability. Conversely, an inadequate mixing period results in an uneven mixture, marked by coarse particles and remaining pores, diminishing the material's homogeneity [18]. Improper mixing time and use of tools can cause the resulting dough to be uneven and can cause cracks during drying. Seeing these problems, it is necessary to have technology in the form of a twin screw extruder machine to produce a homogeneous mixture of raw materials. The extruder machine is a machine commonly used in various industries, the working principle of this machine is to enter the raw material to be processed, then the material that has been entered will undergo a mixing process and then be removed through the mould hole (die), its function is to form the material that has been processed in the extruder machine [19,20]. The current study employed local clay and kaolin in combination with molasses as a natural binder. Although clay crucibles have long been used, studies focusing on using local clay, kaolin, and molasses as natural binders with controlled mixing durations remain limited. Therefore, this study investigated the influence of mixing duration on the Vickers hardness and macrostructure of crucibles formed of local clay, kaolin, and molasses.

2. **Methods**

The crucible specimens in this study were manufactured using locally sourced clay from Kebumen, Indonesia. Subsequently, the dehydrated clay is sieved using a 100-mesh sieve. This investigation incorporates clay, kaolin, molasses, and water.

Crucible specimens were produced using a mixture of clay, kaolin, and molasses, with a percentage of 47.5 wt.%, 47.5 wt.%, and 5 wt.%, respectively. This study involved the utilization of a twin screw extruder machine for mixing the raw materials.

The twin screw extruder machine is loaded with clay and kaolin that have been prepared according to the composition. Water was incorporated into the mixture of raw materials during the mixing process, totaling up to 15% of the overall mixture. The water is gradually added to ensure a thorough mixing of the substances. After thoroughly mixing the clay, kaolin, and water, a 5 wt.% solution of molasses is added to the mixture. The mixing process is conducted by varying its duration to 15, 30, and 45 minutes. A homogeneous mixture is characterized by a particle system in which the concentration of each component is evenly distributed throughout. In optimal conditions, the spatial arrangement of particles in a binary mixture can be characterized by an equitable dispersion of one component's particles among those of the other components over all areas and orientations. This configuration becomes especially apparent when the particle sizes and amounts of the components are analogous. Homogeneous mixtures, or solutions, are defined by a consistent composition and the lack of noticeable differences among their components without agglomerates and visible color streaks.

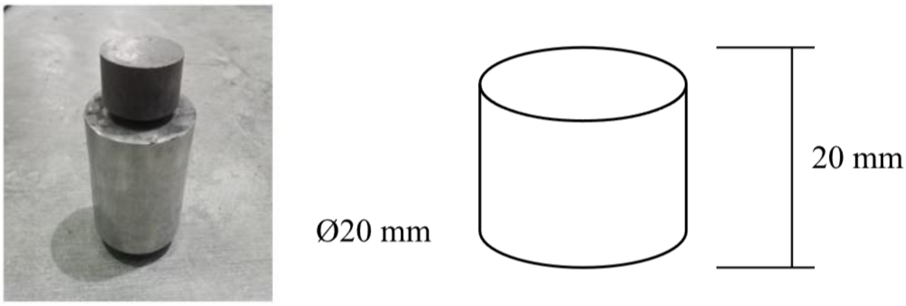

Figure 1. Mold and final dimensions of the specimen

The mixture of materials was pressed using a 20 MPa hydraulic machine in a mold (Figure 1) to create a green body. The green body obtained was dried for 16 hours in an oven maintained at a temperature of 100°C. The combustion procedure was conducted on green body specimens utilizing a furnace set at 1000°C for 3 hours. The specimens generated in this investigation were examined for their hardness and morphology to assess the impact of the length of raw material mixing on the qualities of the specimens. Vickers hardness measurements followed ISO 6507 standards, employing an indentation load of 49.03 N (5 kgf). Indentation testing was conducted in a laboratory using a Vickers hardness tester (Future-Tech Corp. FM-700, Tokyo, Japan) with a measurement error of 0.1 mm. Five repetitions were conducted for each type of specimen test, and the average data was computed. Macrostructure testing was conducted utilizing an Olympus Microscope CX41. This test was performed to ascertain the surface morphology of the specimen, including the dispersion of raw materials and the formation of pores.

3. Results and Discussion

The average Vickers hardness test results on each specimen are shown in Figure 2. The hardness test results indicate that the mixing duration significantly affects the hardness of the specimen. The findings of this investigation demonstrate a positive correlation between the period of mixing and the hardness of the material. The crucible specimens yielded average hardness values of 4.1 HV, 8.5 HV, and 12.4 HV when mixed for durations of 15 minutes, 30 minutes, and 45 minutes, respectively. The hardness value increases as the mixing time increases, as the particles that comprise the sample become more homogeneous and compressed, resulting in an increased density. Generally, increasing density results in enhanced hardness, as a denser material possesses more closely packed particles, hence providing more resilience against deformation under stress, wear, abrasion, and indentation [21–23].

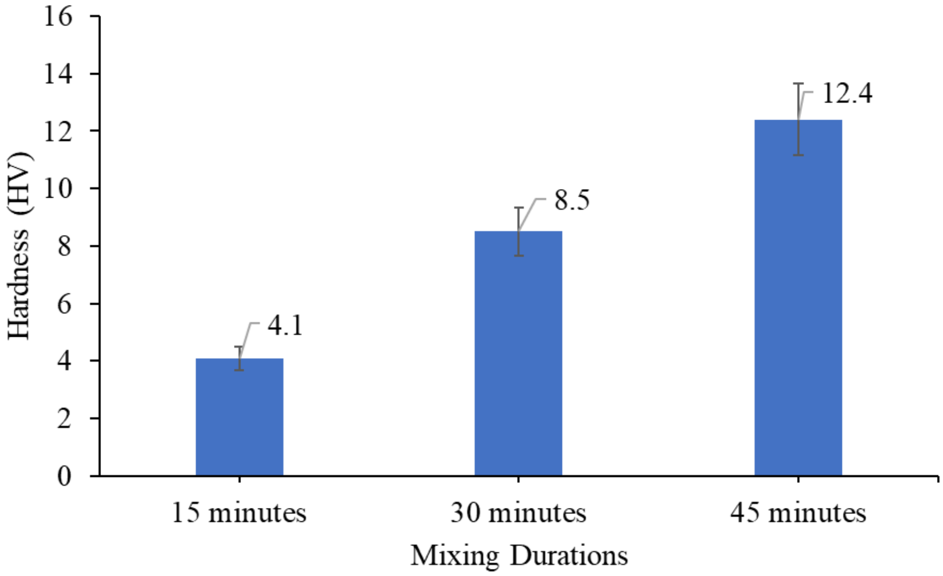

Figure 2. Hardness of local crucible specimens

The findings of this study indicate that prolonging the mixing duration leads to a higher degree of homogeneity in the raw material combination, resulting in a decrease in microstructural flaws and an increase in material hardness. Furthermore, enhanced mixing facilitates a more uniform dispersion of particles, leading to heightened density and diminished porosity, ultimately resulting in augmented hardness. Increased mixing durations facilitate more thorough chemical reactions between the different components of the raw materials, leading to the formation of more stable hard phases in the material's microstructure. In addition, extended mixing duration aids in minimizing the formation of aggregates or lumps in the material, which might potentially weaken it and decrease its hardness. The specimen with the highest hardness was created due to the mixing process lasting 45 minutes. This study's findings align with those of Speth et al. [24], who showed that an increase in mixing time enhances the homogeneity of the composite mixture, with 15 minutes identified as the best period for attaining a uniform distribution of aluminum and SiCp particles. Their investigation indicated that specimens mixed for one minute displayed an inhomogeneous mixture, characterized by apparent significant agglomerates of aluminum or SiCp particles. After five minutes of mixing, the mixture achieved greater homogeneity, although some agglomerates were retained.

The hardness of the crucible specimens in this study is lower than that reported in previous research. Rusiyanto et al. [25] found that crucibles of evaporation boat waste, kaolin, castable, and rice husk charcoal, subjected to different firing temperatures, displayed hardness values between 12.24 and 36.08 HV. The findings indicated a positive correlation between firing temperature and material hardness. The observed increase in hardness is due to enhanced densification, alterations in crystal proportions characterized by a predominance of BN and a decrease in TiB_2, and the emergence of new phases, specifically osbornite/TiN, which exhibit superior mechanical hardness. At elevated firing temperatures, atomic mobility is enhanced, strengthening the interparticle bonds and facilitating improved material densification. Widodo et al. [15] indicate that the hardness of crucible specimens composed of evaporation boat waste and molasses increases with the material's density. Materials exhibiting higher density typically possess a more compact and organized structure, resulting in increased resistance to deformation and enhanced ability to maintain their shape under external forces. Increasing the molasses content from 0% to 5% leads to a higher density, subsequently increasing Rockwell hardness. Increasing the molasses content from 5% to 10% reduces density, which subsequently leads to a decrease in Rockwell hardness. The specimens exhibited densities of 2.21 g/cm^3, 2.25 g/cm^3, and 2.23 g/cm^3 for molasses concentrations of 10%, 5%, and 0%, respectively, alongside Rockwell hardness values of 52 HRA, 62 HRA, and 58 HRA.

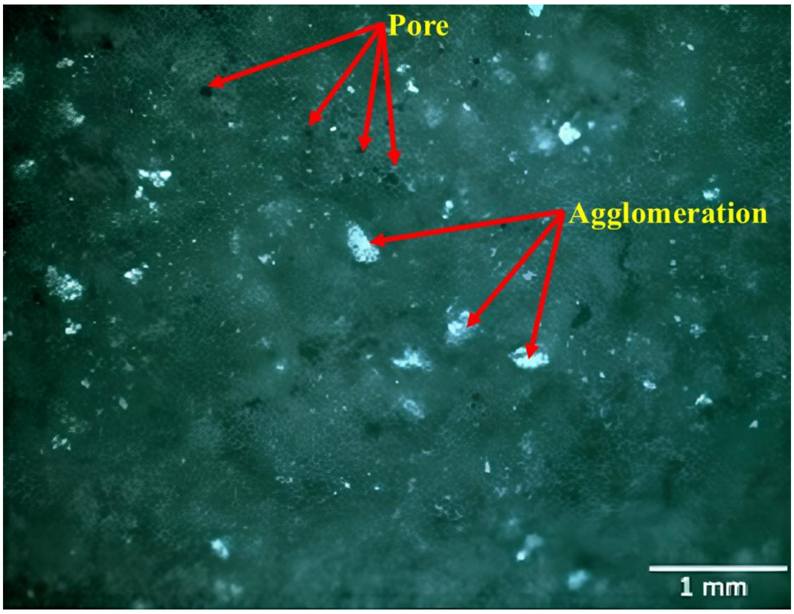

Figure 3. Macrostructure on specimens with a mixing duration of 15 minutes

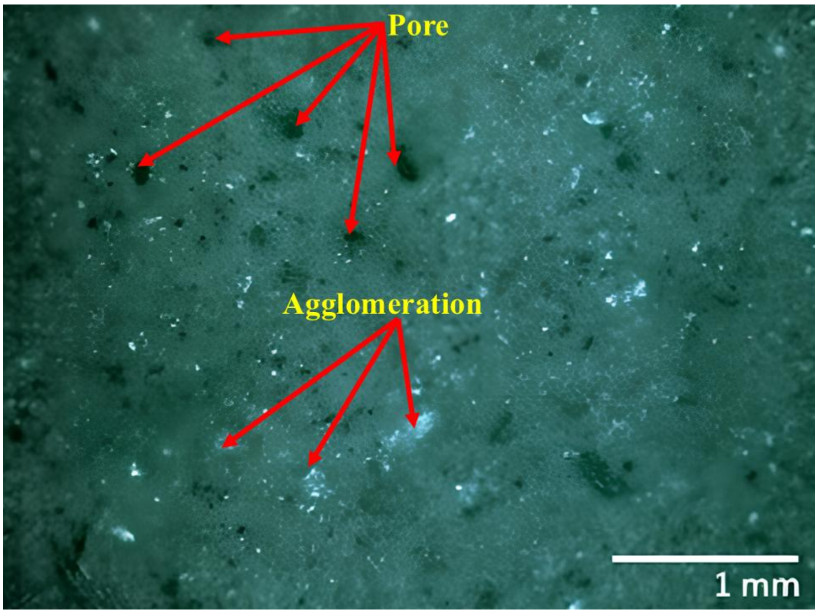

Figure 4. Macrostructure on specimens with a mixing duration of 30 minutes

Figures 3, 4, and 5 illustrate the macrostructural observations of specimens created with raw material mixing durations of 15, 30, and 45 minutes, respectively. This study performed macrostructural analysis to assess the surface shape of specimens produced from varying mixing durations. The specimens were analyzed microscopically at 100× magnification to evaluate their macrostructural properties. The findings of this study indicate that the mixing duration of the raw materials has a significant effect on the physical structure of the resulting specimens. Microscopic analysis of the photographs reveals that the substance is uniformly mixed after a 45-minute mixing period. This is evidenced by the even distribution of kaolin particles, which is more consistent than other samples. Furthermore, specimens derived from raw materials that undergo a 45-minute mixing process exhibit a reduced porosity level compared to other specimens. Therefore, the mixing process produces the best outcomes over a sufficiently extended duration. Liu et al. [26] and Ruegenberg et al. [27] demonstrate that extended mixing durations yield finer pore architectures and decreased material permeability.

Prolonged mixing has corresponded with an increase in yield stress and a reduction in viscosity, underscoring the significant impact of mixing duration on overall material performance. The results correspond with the study's findings, indicating that prolonging the mixing duration of clay, kaolin, and molasses improved particle homogeneity and directly increased the material's density, enhancing its mechanical qualities. Imran et al. [28] assert that agglomerates and pores in the composite specimens signify that the filler mixing procedure has not achieved complete homogeneity. The agglomerates and pores generate localized stress concentration areas, potentially inducing crack formation [29]. The existence of agglomerates and pores undermines the integrity of the bonds created by the filler and the epoxy matrix. As a result, the stress applied to the composite is unevenly distributed through the filler, reducing the composite specimen's tensile strength.

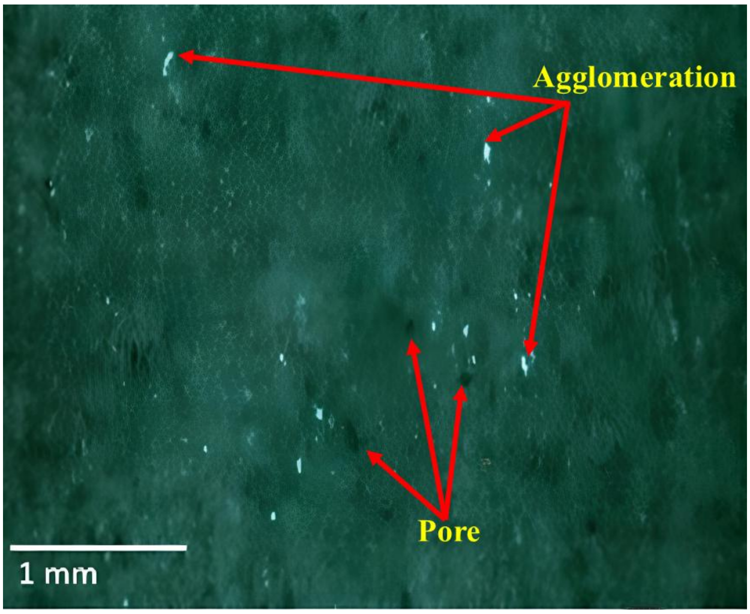

Figure 5. Macrostructure on specimens with a mixing duration of 45 minutes

4. Conclusion

This study highlights that the duration of mixing clay, kaolin, and molasses substantially influences the hardness of the crucible specimens. The Prolonged mixing period increases hardness, increasing from 4.1 HV to 12.4 HV. The best hardness value was attained with a mixing duration of 45 minutes, yielding an average hardness of 12.4 HV. Prolonged mixing durations have demonstrated the ability to produce a more uniform distribution of kaolin particles and diminish the quantity of pores on the specimen's surface. The increased uniformity and density strengthen the mechanical qualities of the crucible, specifically its resistance to pressure, wear, friction, and scratching. These discoveries are significant for companies, particularly in metal casting and refractory material production. Crucibles with enhanced hardness can augment melting process efficiency by providing superior resistance to structural damage, prolonging service life, and diminishing equipment replacement expenses. The findings of this research can inform enhancements to the crucible production process, highlighting the significance of regulating the mixing duration as a critical component. Although these discoveries significantly contribute to the field, additional testing is required to corroborate the results, especially under extreme conditions such as prolonged exposure to elevated temperatures and corrosive environments, to replicate real-world industrial operating settings accurately.

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