Advanced and Sustainable Technologies
ASET
E-ISSN 2976-2294
Volume 5, No 2, September 2026 [367-375]

The Effect of Mechanical Activation Method on Kaolin-Based Geopolymer Ceramic

Khairun Atiqah Abdul Rahman[1], Romisuhani Ahmad [1,2]*, Wan Mastura Wan Ibrahim[1,2], Mohd Mustafa AlBakri Abdullah[2], Shayfull Zamree Abd Rahim[1,2], Nur Bahijah Mustapa[2], and Noor Fifinatasha Shahedan[2,3]

[1]Faculty of Mechanical Engineering & Technology, Universiti Malaysia Perlis, Pauh Putra Campus, 02600 Arau, Perlis, Malaysia.
[2]Centre of Excellence Geopolymer and Green Technology (CEGeoGTech), Universiti Malaysia Perlis, 01000 Kangar, Perlis, Malaysia.
[3]Fuel Cell Institute, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia.

Received 9 February 2026, Revised 14 April 2026, Accepted 10 May 2026

ABSTRACT

Mechanical activation has emerged as a promising alternative to enhance the reactivity of aluminosilicate materials for geopolymer ceramic production while reducing dependence on energy-intensive thermal activation. This study investigates the effect of mechanical activation on kaolin-based geopolymer ceramics, with particular emphasis on the influence of milling duration on physical, microstructural, and mechanical properties. Kaolin powder was mixed with an alkaline activator consisting of sodium hydroxide and sodium silicate at a mass ratio of (Na_2SiO_3/NaOH = 0.24), using a solid-to-liquid ratio of 1.0 to form geopolymer paste. The paste was cured at 80°C for 24 hours, followed by crushing into powder form. Mechanical activation was then carried out through ball milling at a constant rotational speed of 100 rpm using different milling durations of 30 minutes, 45 minutes and 60 minutes. The activated powders were then formed and compacted under uniaxial pressing at 5 ton pressure and sintered at 1000°C to obtain geopolymer ceramic bodies. The results revealed that milling duration significantly influenced the properties of the geopolymer ceramics, with an optimum milling time of 45 minutes producing the highest flexural strength and density, as well as the lowest water absorption. SEM analysis confirmed the formation of a dense and homogeneous microstructure at this milling duration. These findings show that mechanical activation is an effective, low-energy approach to improving the performance of kaolin-based geopolymer ceramics.

Keywords: Ceramic, Geopolymer, Geopolymer ceramics, Kaolin, Mechanical activation.

1. INTRODUCTION

Ceramics are inorganic, non-metallic materials widely used in traditional and advanced engineering applications due to their high thermal stability, hardness, and chemical resistance. Ceramics are characterized by high hardness, strength at elevated temperatures, and significant resistance to chemical [1][2]. However, producing conventional ceramics generally requires high sintering temperatures, leading to high energy consumption and increased environmental impact [3]. In response to these challenges, geopolymer ceramics have gained significant attention as a sustainable alternative because of their lower processing temperature, reduced carbon footprint, and promising mechanical properties [4].

Geopolymers are amorphous aluminosilicate materials formed through alkali activation of silica- and alumina-rich precursors [5]. Among various raw materials, kaolin is considered a suitable

*Corresponding author: romisuhani@unimap.edu.my

precursor due to its high purity, availability, and favorable chemical composition. The major challenge in using natural kaolin as a geopolymer precursor is its insufficient intrinsic reactivity, caused by its stable layered crystalline structure. Conventional approaches rely on calcination to transform kaolin into a more reactive phase; however, this process requires additional energy input and reduces the overall sustainability advantage of geopolymer technology. Therefore, developing an alternative activation method that enhances kaolin reactivity with lower energy consumption is essential. Mechanical activation has been proposed as an alternative method to enhance kaolin reactivity without high-temperature treatment [6]. Through ball milling, mechanical activation reduces particle size, increases surface area, and induces structural disorder, leading to improved dissolution of aluminosilicate species in alkaline environments.

Despite growing interest in mechanical activation as an alternative approach to enhance the reactivity and performance of aluminosilicate materials [6-8], several research gaps remain regarding the role of mechanical activation time in kaolin-based geopolymer ceramics. Existing studies generally show that prolonged mechanical activation reduces particle size and improves material reactivity; however, the optimum activation duration and its relationship to subsequent material properties remain unclear. The mechanisms responsible for the transition from beneficial particle refinement to detrimental over-activation effects also remain insufficiently understood. Furthermore, the relationship between activation time and the resulting physical, mechanical, and microstructural properties of geopolymer-derived ceramics has not been comprehensively investigated under consistent processing conditions. This study investigates how mechanical activation time affects the physical, mechanical, and microstructural properties of kaolin-based geopolymer ceramics sintered at 1000 °C. The study aims to determine the optimum activation time for improving the performance of the ceramics. The effects of activation time on particle size, densification, and microstructure are also evaluated.

2. MATERIAL AND METHODS

2.1 Raw Material

Commercial kaolin powder was obtained from Kaolin Industries Associated Sdn. Bhd., Malaysia and used as the aluminosilicate precursor. Sodium hydroxide (NaOH) pellets and sodium silicate (Na_2SiO_3) solution were supplied by South Pacific Chemicals Industries Sdn. Bhd., Malaysia were used as alkaline activators. A 12 M NaOH solution was prepared using distilled water, while the sodium silicate solution contained 30.1 wt% SiO_2, 9.4 wt% Na_2O, and 60.5 wt% H_2O.

2.2 Geopolymer Preparation

The alkaline activator solution was prepared by mixing sodium silicate and sodium hydroxide solution at a Na_2SiO_3/NaOH ratio of 0.24. Kaolin powder was then mixed with the alkaline activator at a solid-to-liquid ratio of 1.0 until a homogeneous paste was obtained. The paste was poured into molds and cured at 80°C for 24 hours to initiate geopolymerization. After curing, the hardened geopolymer samples were crushed and ground into fine powder.

2.3 Mechanical Activation

Mechanical activation of the geopolymer powder was carried out by ball milling at a constant rotational speed of 100 rpm with a ball-to-powder ratio of 10:1. Milling durations of 30, 45, and 60 minutes were used to investigate the effect of activation time on the properties of the resulting ceramics. Previous studies have reported that mechanical activation of kaolin through ball milling can significantly modify particle size distribution and surface reactivity, which subsequently affects geopolymer formation and ceramic densification [7, 9].

2.4 Pressing and Sintering

The mechanically activated powders were uniaxially pressed using a stainless-steel die at a pressure of 5 tons to form green bodies. The green samples were sintered at 1000 °C in a conventional furnace with a heating rate of 5 °C/min and a holding time of 3 hours. This sintering process was conducted to enhance densification and interparticle bonding. The selected sintering temperature of 1000 °C is consistent with previous findings that thermal treatment enhances crystallization, reduces porosity, and improves interparticle bonding in geopolymer-derived ceramics [10].

2.5 Characterization Techniques

The particle size distribution of kaolin geopolymer-based ceramics was determined using a particle size analyzer (PSA) (Malvern Instruments, Nano Z.S., Malvern Panalytical Ltd., Malvern, UK), employing deionized water as a dispersant. Flexural strength was measured using a three-point bending test at a crosshead speed of 0.3 mm/min and a support span of 30 mm, according to ASTM C1161 [11]. Bulk density was calculated from the measured mass and dimensions of the samples using the standard density formula. Volumetric shrinkage was calculated based on dimensional changes before and after sintering, while water absorption was measured following ASTM C373 [12]. Microstructural analysis of fracture surfaces was conducted using JEOL JSM-6460LA scanning electron microscopy (SEM).

3. RESULTS AND DISCUSSION

3.1 Particle Size Distribution

Figure 1 shows the size distribution of kaolin-based geopolymer ceramics at 30, 45 and 60 minutes of mechanical activation. Increasing the activation time generally leads to a reduction in particle size and a narrowing of the particle size distribution in powder metallurgy processes. At 30 minutes of mechanical activation, the distribution is relatively broad, with the main peak located at approximately the submicron region. A small amount of scattering is also observed at smaller particle sizes, indicating the presence of finer particles within the powder. This suggests that mechanical activation has initiated particle fracture and refinement, although the milling duration may not yet be sufficient to achieve a highly uniform particle distribution [13]. The 45 minutes sample showed the narrowest and most concentrated distribution. The sharper distribution indicates a more uniform particle population compared with the 30 min sample. This demonstrates effective particle refinement and improved particle-size uniformity due to prolonged milling. However, further activation to 60 minutes resulted in a shift toward a larger particle size and a broader distribution. This behavior suggests particle re-agglomeration caused by excessive milling, whereby freshly fractured particles with high surface energy tend to form secondary agglomerates [14].

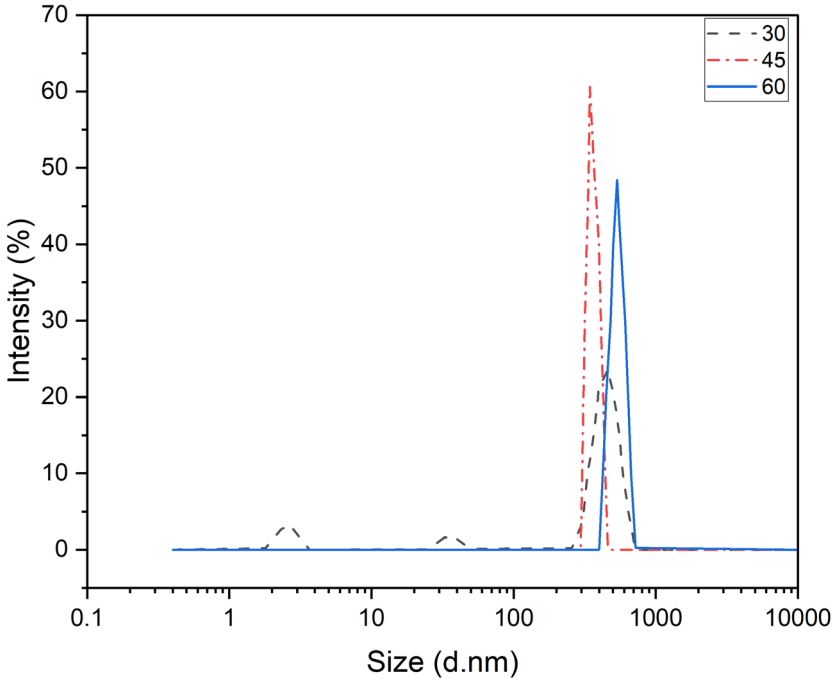

Figure 1: Particle size distribution of kaolin-based geopolymer ceramics mechanical activation of 30, 45 and 60 minutes.

3.2 Flexural Strength and Density Analysis

Figure 2 illustrates the flexural strength and density of kaolin geopolymer-based ceramics at different mechanical activation times. Both parameters demonstrate a similar trend with increasing activation time, suggesting a strong relationship between densification and mechanical performance. The sample without mechanical activation exhibited the lowest flexural strength of 33.07 MPa and density of 1.722 g/cm^3. With increasing activation time to 30 min, the flexural strength and density increased to 52.01 MPa and 2.028 g/cm^3, respectively. The improvement continued at 45 min, where the highest flexural strength (93.39 MPa) and density (2.064 g/cm^3) were achieved. The simultaneous increase in flexural strength and density suggests that mechanical activation promotes particle refinement and improves particle packing, resulting in a more densely consolidated structure during sintering. Finer particles provide a larger contact area and facilitate more efficient rearrangement and neck formation during sintering, reducing internal voids and improving load transfer within the ceramic matrix [15]. Therefore, the higher density obtained at 45 minutes is associated with improved structural integrity, which contributes to the significantly higher flexural strength.

However, prolonging the activation time to 60 minutes led to a reduction in both flexural strength and density, with values of 84.48 MPa and 1.995 g/cm^3, respectively. This reduction implies that prolonged mechanical activation may not continuously improve particle refinement. Conversely, excessive activation could facilitate re-agglomeration of the finely fractured particles, leading to less efficient particle packing and the formation of microstructural defects [16]. As a result, the flexural strength decreases in conjunction with the reduction in density. This behaviour indicates that a 45-minute duration achieves the optimal balance between particle refinement and packing efficiency, resulting in the highest densification and mechanical strength. In general, the close correlation between the density and flexural strength trends indicates that improved densification plays a crucial role in augmenting the mechanical strength.

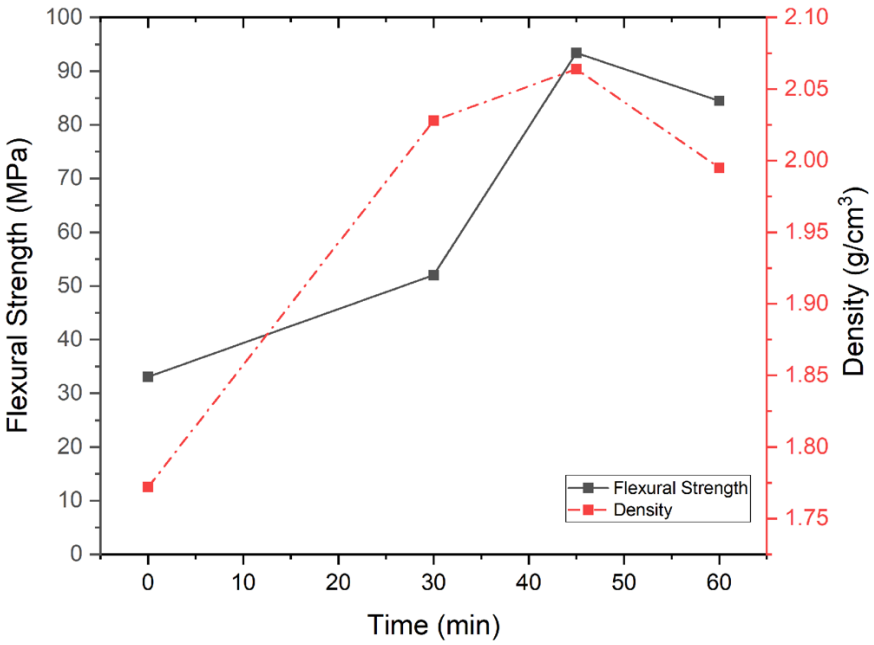

Figure 2: Flexural strength and density of kaolin-based geopolymer ceramics with and without mechanical activation.

3.3 Volumetric Shrinkage and Water Absorption Analysis

Figure 3 shows the volumetric shrinkage and water absorption of kaolin-based geopolymer ceramics at different mechanical activation times. The two physical properties showed an overall improvement with increasing mechanical activation time, particularly up to 45 minutes. The sample without mechanical activation exhibited the highest volumetric shrinkage of 14.911% and water absorption of 6.0%. With increasing activation time to 30 minutes, shrinkage decreased to 11.792%, while water absorption decreased to 4.5%. Further activation to 45 minutes resulted in a reduction in shrinkage to 9.733% and the lowest water absorption of 3.2%, indicating that finer particle size increases the specific surface area and provides more reactive sites for geopolymerization. The finer particles also promote more efficient particle rearrangement and packing during shaping and sintering, resulting in a denser and more homogeneous microstructure with reduced interconnected porosity [17]. As a result, the construction of a denser structure restricts water penetration and contributes to the reduced water absorption that is observed at 45 minutes of mechanical activation. Numerous investigations on activated geopolymers have shown that the material's structural stability is increased during sintering due to decreased porosity and contraction resulting from finer particles and a better packing arrangement [18-19].

When the activation time was extended to 60 min, the volumetric shrinkage continued to decrease to 8.235%. However, water absorption increased from 3.2% to 4.0%. The slight rise in water absorption at the time of 60 minutes can be explained by over-activation processes, particles agglomeration and creation of microstructural defects or microcracks. These flaws enhance the connectivity of the pore structure, which enables more water to flow into the material regardless of the longer activation time.

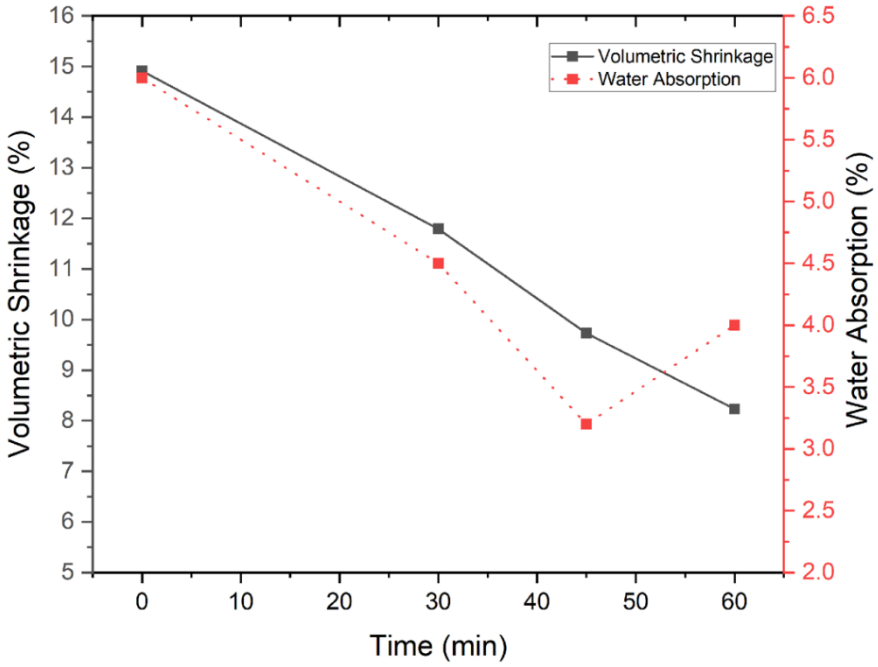

Figure 3: Shrinkage and Water Absorption of kaolin-based geopolymer ceramics as a function of mechanical activation time.

3.4 Microstructure Analysis

Figure 4 shows the SEM micrographs of the fracture surfaces of kaolin-based geopolymer ceramics subjected to different mechanical activation times (0, 30, 45, and 60 minutes) after sintering at 1000°C. The sample without mechanical activation has uneven particle morphology with distinct open pores and irregular surfaces. The particles are loosely packed, which means that there is no refinement of the particles and there is a weak bond between the particles. This porous and non-uniform microstructure justifies the lower density and flexural strength derived in this sample. At 30 minutes, the particles are finer and more evenly distributed, with partial pore closure, to form a more compact microstructure. The maximum density and homogeneity of the geopolymer matrix are best observed at 45 minutes, when the particles are tightly packed, and the pore structure is no longer visible. This demonstrates enhanced reactivity, resulting in increased densification and interparticle bonding, as well as a significant reduction in particle size. Duxson et al. (2007) found that mechanical properties are directly proportional to higher densification and lower porosity, which is consistent with the maximal flexural strength and density recorded at this activation time [20]. The literature also indicates that microstructural enhancements, such as refined particle packing and dense geopolymer gels, are associated with increased mechanical strength and reduced porosity, as demonstrated by SEM analysis of activated geopolymers [21-22].

At 60 minutes of mechanical activation, signs of particle agglomeration and microstructural defects can be observed. High mechanical activation can cause particle re-agglomeration and internal stresses in the geopolymer matrix, forming microcracks. Even though the porosity is relatively small, these defects harm the mechanical integrity of the ceramic, which leads to flexural strength loss. This confirms the idea of an optimum mechanical activation condition, since excess activation does not improve microstructural quality and can worsen material performance [20].

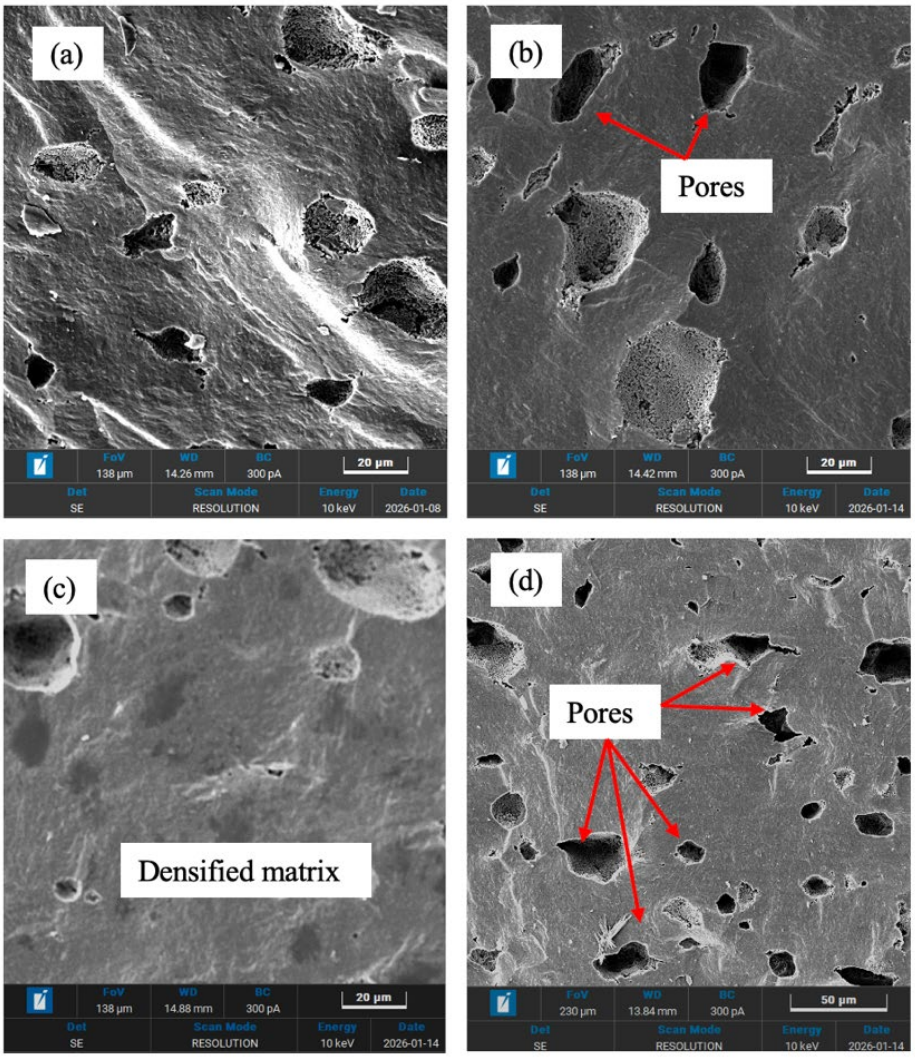

Figure 4: SEM micrographs of kaolin-based geopolymer ceramic fracture surfaces (a) without mechanical activation and with different mechanical activation times, (b) 30 minutes, (c) 45 minutes, (d) 60 minutes at 1000× magnification.

4. CONCLUSION

The study demonstrates that mechanical activation can influence the physical, mechanical and microstructural properties of kaolin-based geopolymer ceramics. The findings indicate that mechanical activation is highly effective in improving material performance by refining particles and increasing the packing and reactivity of geopolymers. Within the processing conditions investigated, 45 minutes of mechanical activation provided the most favourable balance, producing the highest flexural strength and density together with the lowest water absorption and a relatively homogeneous microstructure. The corresponding improvement is attributed to enhanced particle refinement and packing, which promote densification and interparticle bonding during subsequent sintering. Nevertheless, mechanical performance declined when the activation time was extended to 60 minutes, suggesting that excessive milling may cause particle re-agglomeration and microstructural defects. Therefore, these studies demonstrate that mechanical activation must be carefully controlled to obtain a balance of particle refinement with structural integrity. Furthermore, mechanical activation appears to be a less energy-intensive method for increasing kaolin activity and producing geopolymer ceramics with acceptable mechanical properties and low water absorption for various applications. Overall, the study

provides evidence that 45 minutes of mechanical activation is a viable process condition within the limits of the parameters studied and provides a foundation for future research into developing mechanically activated kaolin-based geopolymer ceramics towards sustainable engineering ceramic products.

ACKNOWLEDGEMENTS

The authors would like to acknowledge the support provided by the laboratory staff and supervisors involved in this research.

REFERENCES

[1] Pathania, D., Katwal, R., Thakur, P. Ceramic materials: General introduction, properties, and fabrication methods. Smart Ceramics: Preparation, Properties, and Applications, (2018) pp.33-72. https://doi.org/10.1201/9781315163598-2

[2] Sudha, P. N., Sangeetha, K., Jisha Kumari, A. V., Vanisri, N., Rani, K. Corrosion of ceramic materials. Fundamental Biomaterials: Ceramics, (2018) pp.223-250. https://doi.org/10.1016/B978-0-08-102203-0.00009-3

[3] Jamil, N. H., Abdullah, M. M. A. B., Ibrahim, W. M. A. W., Rahim, R., Sandu, A. V., Vizureanu, P., Gómez-Soberón, J. M. Effect of sintering parameters on microstructural evolution of low sintered geopolymer based on kaolin and ground-granulated blast-furnace slag. Crystals, vol 12, issue 11 (2022) p.1553. https://doi.org/10.3390/cryst12111553

[4] Mohamad Zaimi, N. S., Mohd Salleh, M. A. A., Abdullah, M. M. A. B., Ahmad, R., Mostapha, M., Yoriya, S., Chaiprapa, J., Zhang, G., Harvey, D. M. Effect of kaolin geopolymer ceramic addition on the properties of Sn-3.0 Ag-0.5 Cu solder joint. http://researchonline.ljmu.ac.uk/id/eprint/13432.

[5] Zaimi, N. S. M., Salleh, M. A. A. M., Abdullah, M. M. A. B., Nadzri, N. I. M., Sandu, A. V., Vizureanu, P., Sandu, I. G. Effect of kaolin geopolymer ceramics addition on the microstructure and shear strength of Sn-3.0 Ag-0.5 Cu solder joints during multiple reflow. Materials, vol 15, issue 8 (2022) p.2758. https://doi.org/10.3390/ma15082758

[6] Mañosa, J., Gómez-Carrera, A. M., Svobodova-Sedlackova, A., Maldonado-Alameda, A., Fernández-Jiménez, A., Chimenos, J. M. Potential reactivity assessment of mechanically activated kaolin as alternative cement precursor. Applied Clay Science, vol 228 (2022) p.106648. https://doi.org/10.1016/j.clay.2022.106648

[7] Szabó, R., Kristály, F., Dolgos, F., Ambrus, M., Mucsi, G. The combined influence of classification and mechanical activation of kaolin on the structure and properties of geopolymer. Case Studies in Construction Materials, vol 22 (2025) p.e04876. https://doi.org/10.1016/j.cscm.2025.e04876

[8] Mañosa, J., Alvarez-Coscojuela, A., Maldonado-Alameda, A., Chimenos, J. M. A lab-scale evaluation of parameters influencing the mechanical activation of kaolin using the Design of Experiments. Materials, vol 17, issue 18 (2024) p.4651. https://doi.org/10.3390/ma17184651

[9] Wang, Q., Li, X., Ma, S., Yang, H., Shi, W., Chang, Q., Jin, H. Mechanical activation of metakaolin through milling: Impact on the geopolymerization process. Journal of Non-Crystalline Solids, vol 650 (2025) p.123373. https://doi.org/10.1016/j.jnoncrysol.2024.123373

[10] Derouiche, R., Baklouti, S. Phosphoric acid based geopolymerization: Effect of the mechanochemical and the thermal activation of the kaolin. Ceramics International, vol 47, issue 10 (2021) pp.13446-13456. https://doi.org/10.1016/j.ceramint.2021.01.203

[11] ASTM Committee C-28 on Advanced Ceramics. Standard test method for flexural strength of advanced ceramics at ambient temperature. ASTM International, (2013).

[12] Vieira, A. W., Innocentini, M. D. D. M., Mendes, E., Gomes, T., Demarch, A., Montedo, O. R. K., Angioletto, E. Comparison of methods for determining the water absorption of glazed

porcelain stoneware ceramic tiles. Materials Research, vol 20, issue Suppl 2 (2017) pp.637-643. https://doi.org/10.1590/1980-5373-mr-2017-0089
[13] Ionescu, A. D., Voicu, G., Constantin, G. A., Ipate, G., Ştefan, E. M., Begea, M., Iordache, S. D. Research on the influence of grinding time on the particle size distribution when obtaining rosehips powder. INMATEH-Agricultural Engineering, vol 76, issue 2 (2025) p.923. https://doi.org/10.35633/inmateh-76-79
[14] Abdulmenova, E., Vaulina, O., Kulkov, S. Electrochemical hydrogenation after mechanical activated near equiatomic Ti-Ni powder. IOP Conference Series: Materials Science and Engineering, vol 511, issue 1 (2019) p.012009. https://doi.org/10.1088/1757-899X/511/1/012009
[15] Mustapa, N. B., Ahmad, R., Ibrahim, W. M. W., Abdullah, M. M. A. B., Wattanasakulpong, N., Nemeş, O., Risdanareni, P. Effect of sintering mechanism towards crystallization of geopolymer ceramic-a review. Materials, vol 16, issue 11 (2023) p.4103. https://doi.org/10.3390/ma16114103
[16] Yang, Y. F., Zhang, J. Y. Study on effect of particle size of iron on properties of pure iron sintered. Applied Mechanics and Materials, vol 182 (2012) pp.332-335. https://doi.org/10.4028/www.scientific.net/AMM.182-183.332
[17] Xu, X., Bao, S., Zhang, Y., Luo, Y., Qin, L., Li, S. Role of particle fineness and reactive silicon-aluminum ratio in mechanical properties and microstructure of geopolymers. Construction and Building Materials, vol 313 (2021) p.125483. https://doi.org/10.1016/j.conbuildmat.2021.125483
[18] Heah, C. Y., Kamarudin, H., Al Bakri Abdullah, M. M., Binhussain, M., Musa, L., Khairul Nizar, I., Liew, Y. M. Effect of mechanical activation on kaolin-based geopolymers. Advanced Materials Research, vol 479 (2012) pp.357-361. https://doi.org/10.4028/www.scientific.net/AMR.479-481.357
[19] Mohd Mortar, N. A., Abdullah, M. M. A. B., Abdul Razak, R., Abd Rahim, S. Z., Aziz, I. H., Nabiałek, M., Ghazali, M. F. Geopolymer ceramic application: A review on mix design, properties and reinforcement enhancement. Materials, vol 15, issue 21 (2022) p.7567. https://doi.org/10.3390/ma15217567
[20] Duxson, P., Fernández-Jiménez, A., Provis, J. L., Lukey, G. C., Palomo, A., van Deventer, J. S. Geopolymer technology: The current state of the art. Journal of Materials Science, vol 42, issue 9 (2007) pp.2917-2933. https://doi.org/10.1007/s10853-006-0637-z
[21] Gao, B., Li, Y., Jang, S., Son, H., Lee, H., Bae, C. J. Effect of geopolymerization reaction on the flexural strength of kaolin-based systems. Materials, vol 17, issue 10 (2024) p.2223. https://doi.org/10.3390/ma17102223
[22] Mustapa, N. B., Ahmad, R., Al Bakri Abdullah, M. M., Ibrahim, W. M. W., Sandu, A. V., Nemes, O., Risdanareni, P. Effect of the sintering mechanism on the crystallization kinetics of geopolymer-based ceramics. Materials, vol 16, issue 17 (2023) p.5853. https://doi.org/10.3390/ma16175853

Conflict of interest statement: The authors declare no conflict of interest.

Author contributions statement: Conceptualization, R.A, M.M.A.B., W.M.W.I; Methodology, K.A., S.Z.A.R; Investigation, K.A., R.A., N.F. S; Formal Analysis, K.A.; Writing – Original Draft Preparation, K.A., R.A; Writing – Review & Editing, K.A.; R. A.

Data Availability Statement: The data that support the findings of this study are available from the corresponding author upon reasonable request.