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Effect of Three Tier Treatments on Palm Oil Fuel Ash to the Properties of Ceramic Body

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Abstract

Palm oil fuel ash (POFA) frequently harbours an elevated concentration of silica, a pivotal constituent in the fabrication of porcelain. The aim of this study is to determine the effect of three-tier treatments of POFA on the physico-mechanical properties of porcelain. POFA underwent 3 days of ground process using a ball mill machine at 250 rpm and was sieved with a 50 µm micro-sieve. POFA was subjected to heat treatment at 800°C for 1.3 hours. POFA was treated with 2M (HCl) and subjected to second heat treatment also at 800°C for 1.3 hours. Samples are produced through mixing, pelleting, and sintering processes by varying POFA and quartz compositions (0-25 wt.%). The sintering temperature is 1150 °C with heating and cooling rate at 2 °C and 5 ° C with 1.5 hours of soaking times. The samples were measured the mass loss, volume shrinkage, porosity, bulk density, water absorption and compressive strength. The microstructure's study was done by employing X-Ray Diffraction (XRD) and Secondary Electron Microscopy (SEM). The three-tier treatment surpasses both the untreated and two-tier treatments, highlighting its superior outcomes. The composition of 15 wt.% of POFA showcases the utmost potential for application in porcelain. A significant mass loss of 22.18%, coupled with the most prominent volume shrinkage at 29.49 wt.% during HT2 for 15 wt.% of POFA. The minimum recorded porosity stands at 1.06%, and the pinnacle of bulk density, observed at 0.00248 g/mm^3, aligns with HT2. Notably, the highest compressive strength recorded reaches 320.68 MPa. To validate these results, the research investigates the crystalline properties of POFA by analysing (XRD) patterns and employs (SEM) for the microstructure analysis of POFA. The findings suggest that POFA holds promise as a high-strength material for porcelain tiles with diverse applications in the industry.

1. Introduction

Porcelain is a traditional ceramic widely employed in residential, scientific, and engineering applications [1]. Typically glazed and crafted from clay fired at high temperatures, porcelains are commonly known as triaxial whiteware bodies, comprising clay, feldspar, and quartz, serving as the cornerstone of the ceramic industry [2], [3]. The conventional hard porcelain composition consists of 50% fine-grained clay, 25% fluxes (usually feldspar), and 25% flint (quartz) by weight, maturing at temperatures ranging from 1350 °C to 1450 °C [4]. Porcelain

stoneware tiles, introduced in the 1980s find widespread use in industrial and commercial flooring, offering limited color options and manufactured in compact dimensions [5].

Palm Oil Fuel Ash (POFA) is a by product derived from burning palm oil husks and shells, necessitating the removal of impurities for its application [6]. The ash, categorized based on layers determined by particle size and color, contains significant amounts of silica, presenting potential for use as a cement and ceramic alternative. To advance sustainable practices and add value to porcelain properties, further investigation into the effects of POFA in ceramics is essential. While it is commonly utilized in concrete for enhanced workability and reduced environmental impact, its potential in ceramics remains underexplored. There is a need for further research to comprehend its impact on ceramic properties, processing techniques, and incorporation methods to uncover sustainable solutions. The disposal of POFA in landfills poses environmental challenges, prompting the exploration of alternatives for a cleaner environment, reduced production costs, and enhanced porcelain properties. Academic research suggests that creating composite materials using waste from the palm oil industry, including POFA, could contribute to addressing the global waste issue.

In recent years, porcelain has undergone significant transformations, prompting detailed research into its intricate nature, emphasizing the importance of understanding raw materials, processing science, and phase and microstructural evolution [5]. With an increasing demand for porcelain tiles, researchers are actively working on developing products with superior mechanical and physical properties suitable for both household and decorative purposes [3], [7]. In response to the growing focus on sustainability in the industrial sector, a new environmentally friendly process design adhering to standards and regulations has been implemented. This includes the three-tier treatment of Palm Oil Fuel Ash (POFA), recognized for its potential as a filler to enhance the strength of concrete mixtures [8], [9]. Studies indicate that POFA, especially with high fineness, serves as an excellent pozzolanic material, substituting Portland cement and enhancing the durability of concrete in large structures [10], [11]. Additionally, POFA could serve as a promising filler for polymer concrete [12]. Ground and treated POFA can be used as a suitable pozzolanic material in large concrete structures to mitigate volume changes and micro-cracks caused by thermal stresses [13].

The goal of this research is assessing the impact of the three-tier treatments on POFA and formulating an inventive porcelain composition by incorporating POFA as an additive with traditional porcelain materials. Following that, an evaluation of both the physical and mechanical properties will be conducted to pinpoint the ideal sintering temperature for the production of top-notch porcelain materials, simultaneously analyzing the microstructure of the ceramic resulting from the three-tier treatments. This approach has the potential to reduce manufacturing costs in porcelain tile production. The potential of POFA as a ceramic additive is hindered by impurities and compositional variations. These criteria have been existing in POFA which pose challenges in its effective use in ceramics, impacting material quality and performance. Inconsistent chemical makeup can lead to unpredictable outcomes in manufacturing. To unlock POFA's full potential in ceramic production, it is crucial to address and mitigate these challenges through research and refinement efforts. This will ensure POFA is a valuable and reliable ceramic additive. The study emphasizes the potential of integrating POFA as a sustainable and cost-effective additive in porcelain production, highlighting the need for optimizing three-tier treatments, addressing impurities and compositional variations, and undertaking research and refinement efforts for performance in ceramic production.

2. Materials and Methods

POFA underwent initial processing, including ground which aims to remove impurities and enhance the purity of POFA. It took approximately three days of working time. Next, the sieving process employed a 50 µm micro-sieve to optimize the particle size distribution of POFA and promote better homogeneity in the composite. The process continued with the first heat treatment of POFA. Using the Electronic Box Furnace Protherm PLF 110-130, POFA was heated at a rate of 2 °C/min, and the cooling rate was 5 °C/min. The heating treatment occurred at 800 ºC with a soaking time of 1.5 hours. A portion of POFA powder was utilized to create samples, which were divided into three distinct groups based on variations in heat and acid treatments. Sample Group 1 consisted of untreated POFA pellets that underwent the initial heat treatment at 800 °C for 1.5 hours. Sample Group 2 included pellets subjected to the first heat treatment followed by an acid treatment using hydrochloric acid (HCl) after the sintering process. Sample Group 3 involved pellets undergoing both the first and second heat treatments, along with the acid treatment, the pellets were initially fired at 800 °C for 1.5 hours and then treated with HCl. The treated powder was then blended into a mixture ranging from 0 wt.% to 25 wt.%, as specified in Table 1. Mixing of the composition was carried out for 1 minute utilizing the Kakuhunter Planetary Centrifugal Mixer SK-350TII. The resulting mixed powder was pressed into pellets using a Hydraulic pressure machine (CARVER 3851) for 1 minute at a pressure of 3 tons. Sintering of the pellets took place at 1150 °C, with heating and cooling rates set at 2 °C/min and 5 °C/min, respectively, and a holding time of 1.3 hours. Subsequent to these processes, an evaluation of the physical and mechanical properties is conducted. The Mettler Toledo density kit XS64 conducted a comprehensive analysis, delving into essential parameters crucial for a nuanced comprehension of the material's traits included the percentage of mass loss, volume of shrinkage, porosity, bulk density, and water absorption. Furthermore, the

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evaluation extended to the compressive strength of the specimens, a pivotal mechanical property was conducted with exacting rigor using a state-of-the-art Universal Testing Machine (Testometric), boasting a formidable maximum load capacity of 1000 kN.

Table 1 *Composition of porcelain materials and the percentage of POFA replacement sintered at 1150 °C*

Clay (wt.%)	Feldspar (wt.%)	Quartz (wt.%)	POFA (wt.%)	Percentage of replacement (%)
50	25	25	0	0
		20	5	20
		15	10	40
		10	15	60
		5	20	80
		-	25	100

3. Results and Discussion

3.1 Heat and Acidic Treatment

Following the initial heat and acidic treatment, there was a noticeable alteration in the color of the POFA, albeit with a slight change. It is anticipated that the untreated palm oil fuel ash (POFA) powder will exhibit variations in its physical characteristics prior to the grinding and sieving processes [14]. Moreover, distinctions in the condition of the powder are expected before and after undergoing treatment procedures. Fig. 1 illustrates samples of Palm Oil Fuel Ash (POFA) at different stages: before treatment (a), after the initial heat treatment (b), subsequent to acidic treatment (c), and post-second heat treatment (d). The visual representation in the figure clearly indicates discernible distinctions among these four stages. However, the total weight of the POFA decreased by approximately 3% and 30% after the first heat and acidic treatments, respectively. This indicates the successful removal of impurities, particularly metals bound with silicate, from both ground powders due to the toxic and corrosive nature of HCl. Subsequent to the second heat treatment, a significant transformation in the colour of the POFA was observed compared to the untreated sample. Additionally, the ultra-fined POFA exhibited a reduction in the percentage of unburned carbon content, leading to an enhancement in its pozzolanic characteristics. The increase in silicate-based elements within the material also has a direct impact on the compressive strength of the sample. Some residual black ashes were observed, suggesting the presence of a few remaining carbon residues. When employing a low temperature burning process, the formation of amorphous silicon oxides (SiO_2) occurs instead of crystalline SiO_2 [15]. After grinding the POFA, the weight loss percentage was measured, indicating the removal of about 80% of unwanted elements. Subsequently, the POFA underwent a first heat treatment using the same acid as before to further reduce residual carbon before undergoing the second heat treatment.

(a) **(b)** **(c)** **(d)**

Fig. 1 *Samples of Palm Oil Fuel Ash (POFA) at different stages: before treatment (a),after the initial heat treatment (b), subsequent to acidic treatment (c), and post-second heat treatment (d)*

3.2 Physical Properties

This study encompassed the measurement of five distinct physical properties, namely mass loss, percentage of volume shrinkage, porosity, bulk density, and percentage of water absorption. Fig. 2 illustrates the graph depicting the relationship between mass loss and the composition of POFA. It is evident that the overall mass loss increases proportionally with the addition of POFA to the sample. At 0-25 wt.%, the mass loss increased across all treatment and untreated with constant sintering temperatures 1150 °C, indicating significance in altering the mass loss

percentage when the samples are exclusively occupied by POFA. Notably, the sample with 5 wt.% POFA content recorded the lowest mass loss at 3.11%, attributed to the untreated POFA, preventing sufficient time for particles of POFA to fill larger pores [16]. Conversely, the highest composition in this study resulted in the maximum mass loss at 22.18%. This may be indicative of rapid densification at very high temperatures, leading to a decrease in mass. Additionally, the small particle size of POFA is associated with a pozzolanic reaction, enhancing the packing effect during sintering, and improving the arrangement of small particles by occupying voids and air bubbles [9]. However, as the quantity of POFA in the sample increases, so does the number of pores [17]. Furthermore, the elevated unburnt carbon content in POFA could potentially influence changes in the mass loss value [18].

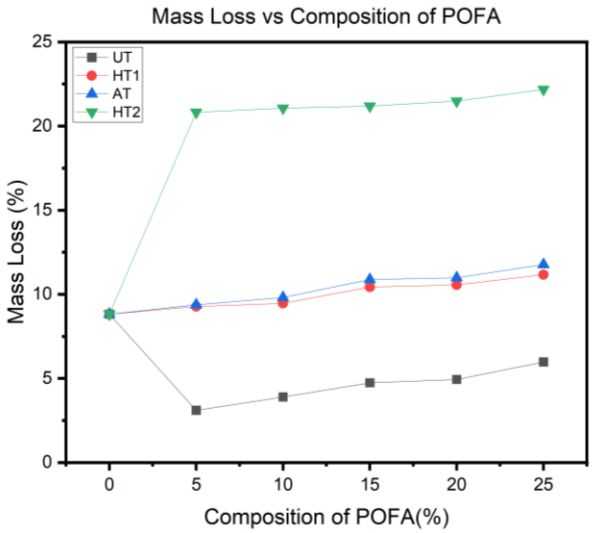

Fig. 2 *Mass Loss vs Composition of POFA for untreated (UT), first heat treatment (HT1), acidic treatment (AT) and second heat treatment (HT2)*

Fig. 3 depicts volume shrinkage variations at different POFA compositions during sintering at 1150 °C. The percentage of volume shrinkage consistently increases until 15 wt.% of POFA, declining at 20 and 25 wt.%. The peak shrinkage occurs at 29.49 wt.% during the second heat treatment, indicating an attempt to fill voids and eliminate trapped air. Notably, volume shrinkage decreases with POFA addition compared to untreated and treated samples (27.40 wt.%, 27.72 wt.%, and 28.83 wt.%) respectively, emphasizing the efficacy of the second heat treatment for specific waste materials. The trend aligns with higher bulk density, suggesting a more compact particle arrangement and limiting volumetric changes. This increased compactness results in reduced volume shrinkage during sintering. Interestingly, pelleted sample volume decreases as POFA and quartz composition rises, especially during the second heat treatment, possibly linked to trapped gas expansion forming pores during heating [19].

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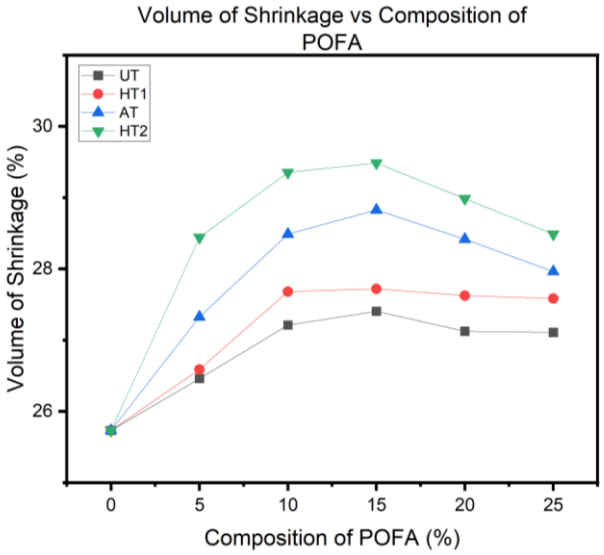

Fig. 3 *Volume of shrinkage vs Composition of POFA for untreated (UT), first heat treatment (HT1), acidic treatment (AT) and second heat treatment (HT2)*

The relationship between bulk density and porosity is evident in denser samples, which tend to have fewer pores. In Fig. 4, the porosity of porcelain samples sintered at a constant temperature of 1150 °C is depicted across various compositions. The graph shows a decrease in porosity as the proportion of (POFA) increase, but only until 15 wt.% of POFA due to the addition of POFA to the porcelain mixture induces volume shrinkage [20], resulting in a reduction in porosity. Furthermore, the incorporation of POFA increases the bulk density of the porcelain material [21], attributed to the heightened mobility of glass particles. The observed decline in porosity for 20 and 25 wt.% POFA is attributed to the introduction of POFA, even with the presence of a pozzolanic reaction, possibly due to the higher concentration of unburnt carbon in POFA relative to amorphous silica, leading to diminished reactivity. Additionally, the particle shape in POFA may contribute to heightened inter-particle friction. The recorded minimum porosity, identified at 15 wt.% of POFA, reaches 27.41 % for samples. This emphasizes that the interplay between POFA composition and sintering temperature significantly influences the porosity of the porcelain samples, offering insights into the intricate dynamics of these materials during the sintering process. This finding aligns with the general concept that an increase in bulk density in ceramic materials, including those with POFA, typically corresponds to a decrease in porosity, underscoring the importance of understanding these interconnected material properties.

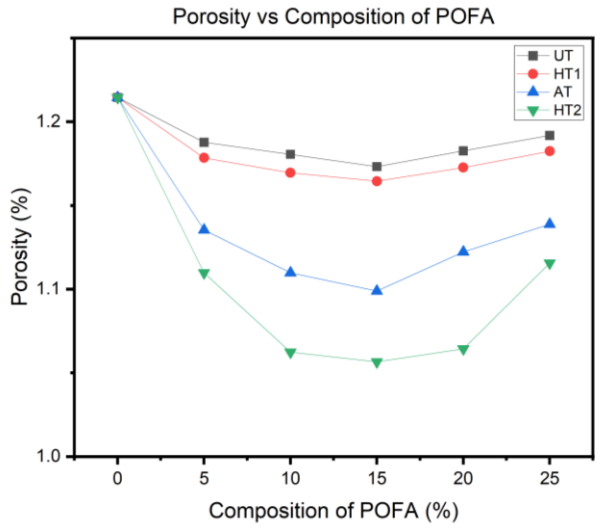

Fig. 4 *Porosity vs Composition of POFA for untreated (UT), first heat treatment (HT1), acidic treatment (AT) and second heat treatment (HT2)*

Bulk density is typically inversely related to porosity, noting that denser samples tend to have fewer pore spaces. POFA has a bulk density of 2.424 g/cm^3 [22]. Fig. 5 illustrates the Bulk density vs Composition of POFA for untreated (UT), first heat treatment (HT1), acidic treatment (AT) and second heat treatment (HT2). The peak of bulk density occurs at 0.00248 g/mm^3 during the second heat treatment, indicating an attempt to fill voids and eliminate trapped air. Notably, bulk density increases with POFA addition compared to untreated and treated samples which are 0.00241, 0.00242 and 0.246 (g/mm^3) respectively at 1150 °C, emphasizing the efficacy of the second heat treatment for specific waste materials. The discussion of wetting of crystalline phases, the dissolution of quartz, and the contribution of SiO_2 content aligns with the expected changes in the material's structure during sintering. The observation of an increasing pattern in bulk density with rising proportions of POFA up to 15 wt.% and a subsequent decreasing pattern at 20-25 wt.% is consistent with the complex interplay of factors influencing densification. An increase in bulk density generally leads to a stronger and more consolidated glassy phase in porcelain. The soaking time significantly affects the bulk density of POFA. With an increase in soaking time, there is a corresponding rise in the mobility of glass particles, resulting in higher bulk density [21]. The composition of POFA plays a pivotal role in determining its bulk density, with the growing amount of disposed palm oil fuel ash from palm oil production impacting the properties and morphology of the composite material [21]. The bulk density of POFA is also influenced by its particle size and structure; larger particle size and more porous structures lead to lower bulk density [23].

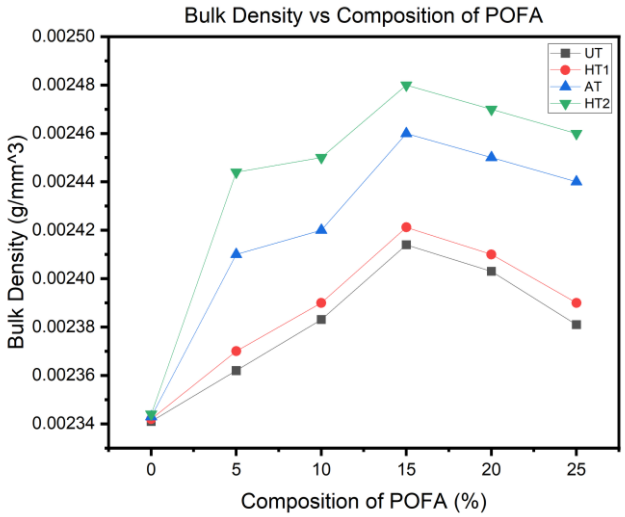

Fig. 5 *Bulk density vs Composition of POFA for untreated (UT), first heat treatment (HT1), acidic treatment (AT) and second heat treatment (HT2)*

Fig. 6 illustrates the trends in water absorption in porcelain samples with varying additions of POFA at different treatment temperatures. Notably, during the second heat treatment, water absorption percentages decrease, particularly for samples sintered at 1150 °C. The highest water absorption is observed at 0 wt.% untreated POFA, with a value of 1.75%. As the composition of POFA increases, water absorption fluctuates, indicating potential incomplete densification during sintering. Samples show water absorption percentages above 1.61 %, with the lowest recorded at 25 wt.% second heat treatment POFA. The introduction of POFA leads to the creation of pores and voids in the porcelain [24], ultimately decreasing water absorption as POFA particles act as a filler material, reducing overall porosity [25]. Furthermore, the addition of POFA aids in optimizing the shrinkage of triaxial porcelain [24], resulting in reduced water absorption. Despite notable water absorption in these samples, their density remains relatively low, suggesting the possibility of bloated and cracked surfaces resulting from over sintering, wherein trapped gas expands during sintering. It is emphasized that the sintering temperature significantly influences water absorption, while the replacement of waste material has a limited impact [26].

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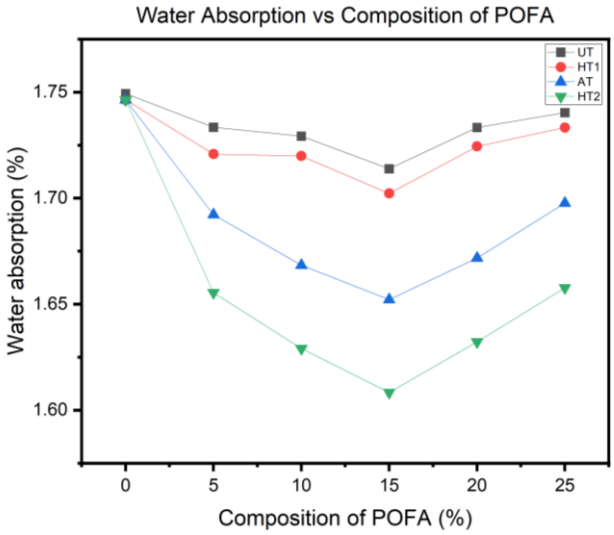

Fig. 6 *Water absorption vs Composition of POFA for untreated (UT), first heat treatment (HT1), acidic treatment (AT) and second heat treatment (HT2)*

3.3 Compressive Strength

Fig. 7 illustrates the compressive strength graph for sintering temperatures at 1150 °C with varying compositions of POFA (0 wt.%, 5 wt.%, 10 wt.%, 15 wt.%, 20 wt.%, and 25 wt.%). Indicating that beyond 15 wt.%, the increase in the composition of POFA to the porcelain composition induces microstructural changes [27]. This is ultimately attributed to the rich alumina (Al_2O_3) content in POFA [22], a crucial element in forming robust bonds within porcelain, aiding the sintering process and further enhancing compressive strength. Moreover, the compressive strength is influenced by the soaking time emphasizing the impact of correct soaking duration on porcelain strength [21]. The graphs also display samples exhibit fluctuations within the range of 20 wt.% to 25 wt.% POFA added, the addition of POFA limits the maximum loads and, consequently, the strength of the samples. This limitation could be attributed to the presence of unburnt carbons in POFA, weakening the effect of crystalline and amorphous silica. Among the three-tier treatments, samples subjected to the second heat treatment record the highest compressive strength at 320.68 MPa, while the lowest is observed at 25 wt.% POFA with 216.62 MPa also under second heat treatment. POFA's elements like calcium oxide (CaO) and K_2O may contribute to pore formation, reducing compressive strength [28]. Despite small water absorption and density variations around 2% from the standard value for samples, the compressive strength is notably high, suggesting the potential for producing lightweight porcelain tiles with standard density and resilience to high impact or loads using waste materials.

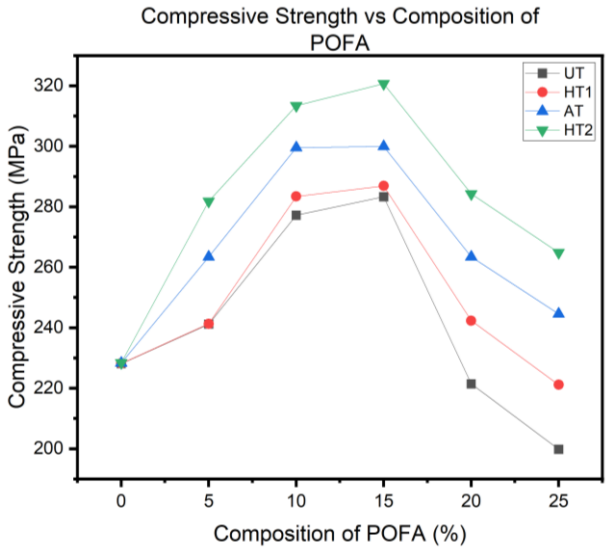

Fig. 7 *Compressive strength vs Composition of POFA for untreated (UT), first heat treatment (HT1), acidic treatment (AT) and second heat treatment (HT2)*

3.4 Crystallographic analysis

The crystallographic analysis of the specimen was conducted using X-ray Diffraction (XRD). The investigation focused on varying treatment of POFA replacement comprise of untreated POFA subjected to heat treatment at 800°C for 1.3 hours. After that, it was treated with 2M hydrochloric acid (HCl) and received a second heating at same temperature and soaking time as the first treatment. Fig. 8 illustrates XRD profile of four samples, namely UT, HT1, AT and HT2.

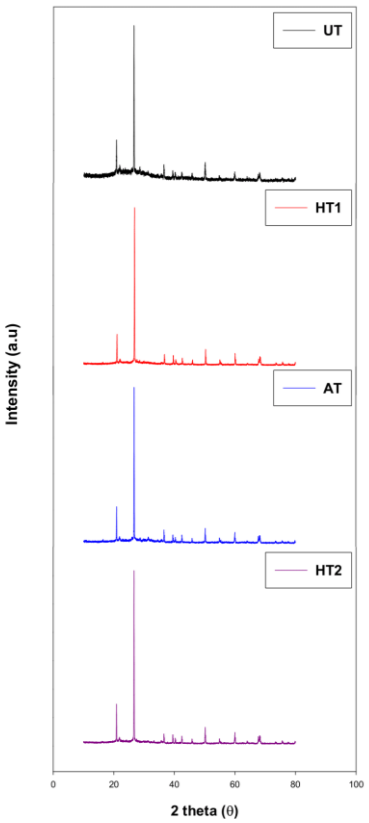

Fig. 8 *XRD profile of four samples, namely UT, HT1, AT and HT2.*

The crystalline characteristics of POFA can be deduced by analysing its X-ray diffraction (XRD) patterns. Sharp and well-defined peaks observed in the XRD pattern of POFA would imply a highly organized crystalline structure, potentially indicating the presence of crystalline phases like quartz, calcite, or other minerals. The number of peaks discernible in the XRD pattern serves as an indicator of the complexity of the crystalline structure, with the presence of multiple sharp peaks suggesting a mixture of different crystalline phases in POFA.

3.5 Surface Morphology

The microstructure analysis of the specimen was conducted using (SEM). The investigation focused on samples sintered at 1150 °C, with varying percentages of (POFA) replacement, namely 0%, 5%, 10%, 15%, 20%, and 25%. Fig. 9 illustrates SEM micrographs of three samples: (a) HT1 representing 100% POFA at 1150 °C, (b) AT representing 100% POFA at 1150 °C, and (c) HT2 representing 100% POFA at 1150 °C.

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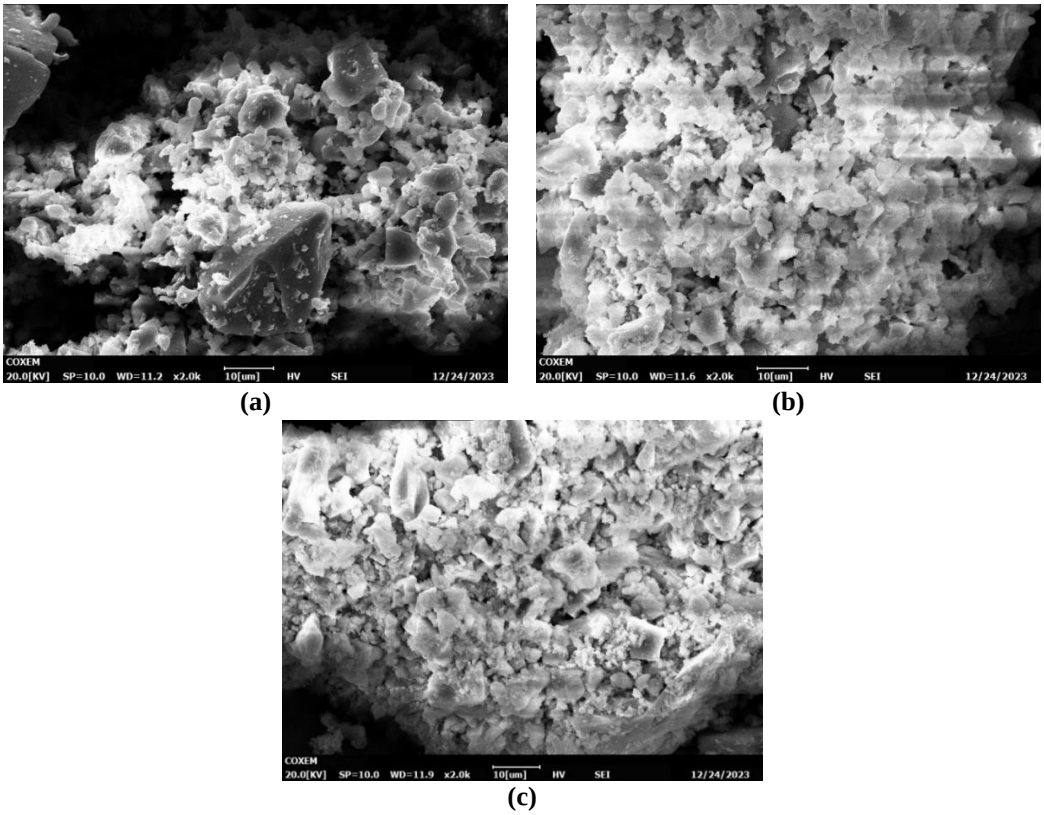

Fig. 9 *SEM micrographs of three samples: (a) HT1 representing 100% POFA at 1150 °C, (b) AT representing 100% POFA at 1150 °C, and (c) HT2 representing 100% POFA at 1150 °C.*

The microstructure analysis depicted in Fig. 9 reveals that (c) exhibits the smallest pores and particles compared to (a) and (b). This difference may be attributed to the dual heat treatment at 800 °C and the sintering temperature of 1150 °C, resulting in low porosity that promotes sample densification through rapid sintering neck connections, thereby enhancing compressive strength. Additionally, in this sample, a reduction in particle size and fewer pores is evident. The incorporation of 1150 °C POFA during sintering appears to contribute to decreased porosity.

4. Conclusion

This study establishes that the three-tier treatment outperforms both the untreated and two-tier treatments, emphasizing its superior results by suggesting that a composition of 15 wt.% of POFA exhibits the highest potential for application in porcelain. Moreover, it underscores that a composition of 15 wt.% of POFA exhibits the highest potential for application in porcelain. In addition, the study reveals a significant mass loss of 22.18%, with the most notable volume shrinkage occurring at 29.49 wt.% during HT2 recorded for 15 wt.% of POFA. The lowest recorded porosity was 1.06%, while the peak bulk density, observed at 0.00248 g/mm^3, also coincided with HT2. Notably, the highest compressive strength recorded is 320.68 MPa. Indeed, the future holds tremendous promise for ceramics, as advancements in technology and material science continue to unlock new possibilities and applications, especially using three-tier treatments.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design, data collection, methodology, analysis and interpretation of results:*** *Nuralya Athirah Emran and Mohamad Zaky Noh. All authors reviewed the results and approved the final version of the manuscript.*

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