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# Effect of Composition and Sintering Temperature in the Production of Clay-POFA Ceramic

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Abstract: Palm oil fuel ash (POFA) has the potential to replace quartz in the composition of porcelain because of its distinct properties, such as high silica and hydroxyl group concentrations. This study investigated the effect of composition and sintering temperature under different treated POFA (TPOFA) on the morphology, physical, and mechanical properties of clay-POFA ceramic. The POFA was treated at temperatures of 800 °C, 900 °C, and 1000 °C. The TPOFA was mixed with clay ata composition of 10 wt% to 50 wt% and dry pressed into pellets at a mould pressure of 3 tonnes and sintered at 1100 °C and 1125 °C for 1 hour soaking time respectively. The sintered samples were evaluated the mass loss, volume shrinkage, bulk density, and compressive strength and characterized the surface morphology. The results showed that heat treatment and sintering temperature were crucial, affecting porcelain's physical and mechanical properties. The highest compressive strength, bulk density, and mass loss were achieved at a sintering temperature of 1125 °C using a treated sample of 900 °C of 10 wt% with the values 313 MPa, and 2.529 g/cm3, respectively. Treated POFA is a good candidate for quartz replacement, and 1125 °Cwas the best sintering temperature. These results showed that POFA could provide the basis for the development of low-cost and lightweight ceramic for tiles applications.

Keywords: Ceramic, POFA, Heat Treatment, Clay, Waste

# 1. Introduction

Ceramics are generally made from kaolin, quartz, and feldspars. They are highly resistant to abrasion due to their hardness. However, one of the drawbacks is that they are brittle, heavy, and easy to break [1]. Cracks are frequently seen around quartz grains in conventional porcelain microstructures because of the significant thermal expansion mismatch between the glassy and crystalline [2]. Few

researchers [3-6] have studied the alternative option of the replacement of either quartz, feldspar, or clay by using organic waste that has a high amount of silica oxide  $(SiO_2)$  and alumina  $(Al_2O_3)$  such as palm oil fuel ashes (POFA) [7]. These two compounds are crucial to increase pozzolanic reactivity and improving mechanical strength. A research report shows that high pre-treatment temperatures cause the crystallization of amorphous silica and the presence of SiO<sub>2</sub> due to the removal of carbon [4]. Hence, the temperature of heat treatment POFA (TPOFA) is significant to this study. This is due to different heat treatment temperatures giving the different surface area, microstructure, and specific cumulative pore volume. According to Zhong [8], specific surface area becomes smaller with the increases in heat treatment of POFA, resulting in denser ceramic and cumulative pore volume reduced to smaller size due to the removal of porous carbon during heat treatment.

Besides, POFA is organic waste that adds to environmental contamination on both the input and output sides of its operations. Thereby resulting in Malaysia's severe issue of material waste. Dumping spaces are becoming more crowded as palm oil fuel ash (POFA) is produced at an increasing rate. Environmental efforts are hampered by dangerous garbage entering the environment and accumulating industrial activities that produce excessive amounts of greenhouse gases as a byproduct [9]. Hence, the POFA can be an alternative to replace the quartz [10].

Therefore, this study focuses on the effect of physical and mechanical properties with the replacement of POFA. A study reported that sintering temperatures of ceramic improve the physical and mechanical properties of ceramic; mass loss [11], bulk density [12], volume shrinkage [13], water absorption [14], porosity [15], and compressive strength [16] is temperature-dependent. In research, temperature significantly impacts the mechanical characteristics of ceramics, such as the highest compressive strength, low porosity, and low volume shrinkage of ceramics achieved at temperatures of 900 °C [17]. Another research from Sani [3] reported that the compressive strength of porcelain samples generated from treated POFA with HCL acid is higher than untreated; the compressive strength is 169 MPa obtained at 15 wt% of POFA. Hence, the sintering temperature is a vital processing step for achieving the required levels of stiffness and stability in ceramic bulk materials' mechanical properties. Extremely high temperatures can cause inefficiency in the clay-POFA porcelain. This is supported by another study [18] concluded that the highest physical attributes of porcelain samples incorporating RHA and POFA were seen at a temperature of 1100 °C and a substitution rate of 20% by weight.

Previous investigations have only created POFA porcelain at a single heat treatment temperature. This study examined the viability of POFA as a quartz replacement in clay-POFA production, as varied POFA treatments may impact the characteristics of ceramic. The effect of 10%, 20%, 30%, 40%, and 50% POFA replacement on the physical and mechanical properties at 1100 °C and 1125 °C was investigated. In addition, the performance of treated POFA (TPOFA) ceramic was examined. The findings of this study provide valuable information for the tile sector, which can profitably employ waste materials, as well as for industries that produce waste POFA, enabling the manufacturing of environmentally benign and economically advantageous ceramic.

#### 2. Materials and Methods

#### 2.1 Materials

POFA was supplied by a crude palm oil plant in Kluang, Johor, Malaysia. The raw POFA was first reduced in particle size using a ball mill. Then, POFA was heated in a furnace at temperatures ranging from 800 to 1000 °C for 2 hours and 34 minutes at a heating rate of 5 °C/minute. With increasing treatment temperatures, the color of POFA changed from black to light brown after treatment. The modified POFA was subjected to further grinding and sieving to achieve a particle size of 50  $\mu$ m or less.

#### 2.2 Methods

The treated POFA (TPOFA) was gradually integrated into the clay body from 10 wt% to 50 wt% (Table 1) with the addition of PVA as a binder agent. Using a Kakuhunter for two minutes, the composition was mixed. At a pressure of 3 tonnes, pellets were pelleted from the combined powders using hydraulic Carter pressure. All pellets were sintered at temperatures of 1100 °C and 1125 °C for soaking durations of 1 hour at a heating rate of 1 °C per minute.

Sample name	Kaolin	POFA		
Α	90	10		
В	80	20		
С	70	30		
D	60	40		
Е	50	50		

Fable 1:	The	composition	with	the	substitution	of	quartz	by	PO	FA
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The physical and mechanical properties of the pellets were determined, including mass loss, volume shrinkage, bulk density using Mettler Toledo density kit XS64, and compressive strength. X-ray diffraction (BRUKER) was used to analyze the POFA's crystalline structure, and scanning electron microscopy (COXEMEM-30AX) was used to analyze the POFA's microstructural characteristics.

#### 3. Results and Discussion

The physical, mechanical, and characterization of samples at different sintering temperatures were analyzed.

#### 3.1 Mass Loss

A decreasing trend was obtained as the treated POFA (TPOFA) increased for both graphs under every temperature; 800°C, 900 °C, and 1000 °C as shown in Figure 1.



Figure 1: Graph of Mass Loss for (a) 1100 °C and 1125 °C

At 1100 °C sintering temperature, the treated POFA at 800 °C showed the highest mass loss percentage, followed by treated POFA at 900 °C and treated POFA at 1000 °C. The graph of T900 °C and T1000 °C exhibits similar however, at 40% to 50% composition of POFA, 1000 °C TPOFA have the highest mass loss percentage while 900 °C has the highest mass loss percentage at 30 wt%. Interestingly, it could be seen that the graph at 1125 °C exhibits a similar trend to the previous graph. The highest mass loss percentage is obtained at 10 wt% of POFA, which is 14.67%, slightly higher than the 1100 °C sintering temperature, which is 14.31%. Generally, when the composition of POFA was increased from 10% to 50%, the mass loss of samples decreased for both sintering temperatures. Despite that, sintering temperature at 1125 °C has a higher mass loss rate compared to 1100 °C. This was due to partial pore shrinkage after POFA treatment.

#### 3.2 Volume Shrinkage

The results demonstrate that the shrinkage was the lowest at 10 wt % addition of treated POFA, with the highest value of volume shrinkage is 800 °C TPOFA of 30 wt% as shown in Figure 2. At 1100 °C and 1125 °C, each graph increases from 10 wt% to the maximum at 30 wt%, then decreases. The line for 900 °C and 1000 °C TPOFA at graph (c) exhibit increasing pattern however the line for 900 °C and 1000 °C at graph (d) only increased at 20 wt% then decreasing. There were some changes in the mass of sample due to the reduction of porosity.



Figure 2: Graph of Volume Shrinkage for (a) 1100 °C (b) 1125 °C

#### 3.3 Bulk Density

Each graph has the highest density at 10 wt% of 1000 °C sintered temperature, which is 2.538 g/cm<sup>3</sup> and 2.531 g/cm<sup>3</sup>, respectively as shown in Figure 3. Also, both sintered temperature samples have the lowest density at 20 wt% of 800 °C. According to Zhong [8], treated POFA at 800 °C and 900 °C showed larger macrovoids and less dense sponge-like layers resulting in high porosity of each TPOFA. Despite this, treated POFA at 1000°C containing larger particle size of POFA induces blind pores in the microstructure to produce dense sample. However, the density of samples for 1125 °C fluctuates, unlike 1100 °C. It may be due to a few samples crack that affects the average density during the sinteringprocess. From the observation, the bulk density reached the maximum density at 1000 °C TPOFA for both graphs at 10 wt% of POFA maybe be due to the neck development connecting the nearby big particles; 1000 °C TPOFA particles looked to have large particle sizes, as stated by a previous study.



Figure 3: Graph Bulk Density for (e) 1100 °C (f) 1125 °C

#### 3.4 Compressive Strength

Figure 4 shows the graph of compressive strength for 1100 °C (g) and 1125 °C (h) versus the composition of POFA, as can be seen in the graph for 800°C TPOFA and 900 °C TPOFA decreased gradually with the addition of TPOFA except for 1000 °C TPOFA.



Figure 4: Compressive Strength for (g) 1100 °C (h) 1125 °C

As expected, the compressive strength decreased with the addition of POFA except for treated POFA at 1000°C for both graphs. It was observed in Figure 3.4 that the trend for both sintering temperature graphs 1100 °C and 1125 °C are similar, where 800 °C TPOFA and 900 °C TPOFA decrease except for 1000°C TPOFA. Interestingly, the compressive strength rises only after 30 wt% of 1000 °C. Based on a previous study, the 1000 °C TPOFA gives low porosity which is 42.4%, that lead to the densification of samples because of rapid sintering neck connection induced the tensile strength that was superior to 800° C TPOFA and 900 °C TPOFA samples because of the larger grain size, which results in a more robust. These variations in compressive strength are connected to the body's porosity development.

# 3.5 Surface Morphology

The SEM micrographs of the samples: F' of 10 wt% (900 °C TPOFA at 1125 °C), F of 10 wt% (900 °C TPOFA at 1100 °C), and N' of 50 wt% (1000 °C TPOFA at 1125 °C) are presented in Fig. 5.



(a)



(b)



(c)

Figure 5: Surface Microstructure for (a) F' of 10 wt% (900 °C TPOFA at 1125 °C, (b) F of 10wt% (900 °C TPOFA at 1100 °C), and (c) N' of 50 wt% (1000 °C TPOFA at 1125 °C)

As can be observed from the microstructure of Figure 3.5, (a) has the lowest pore and small particle compared to (b) and (c). This may be due to the heat treatment of 900 °C and the sintered temperature at 1125 °C, which gives low porosity that leads to the densification of samples because ofrapid sintering neck connection induce compressive strength. At the highest TPOFA of 1000 °C, the larger particle size and more pores can be seen. The presence of 1000 °C TPOFA may increase the porosity during the sintering. RHA and POFA were seen at a temperature of 1100 °C and a substitutionrate of 20% by weight.

It was found that the 900 °C TPOFA particles were irregularly small-sized in shape, as can be seen in images (a) and (b). in contrast, graph (c) appears to be more porous with hole-like on the surface; after enduring pre-treatment temperature and the greatest sintering temperature, it is also possible to view the surface's roughness and pores with clarity. This demonstrates that sintering at varying temperatures will modify the surface morphology of the sample.

#### 3.6 X-ray Diffraction

Figure 6 shows the result of the X-Ray Diffraction (XRD) pattern and XRD analysis of the samples. The selected samples are (F) 10 wt% of 900 °C at 1100 °C, (B') 20 wt% of 800 °C at 1125 °C, (C) 30 wt % of 800 °C TPOFA at 1100 °C, (N') 40 wt% of 1000 °C at 1125 °C and (O') 50 wt% of 1000 °C at 1125 °C.

Quartz elemental peaks imply the presence of crystalline phases, as observed. Quartz phases are mainly found in XRD because of the heat treatment of POFA that eliminates carbon aftercombustion and induces silica elements in POFA [8]. The transformation of the amorphous phase intocrystalline quartz caused the quartz intensity to rise at higher treatment temperatures. Cristobalite and hematite, as minor, further revealed that the 1125 °C sintering temperature shows the higher peak of quartz at  $2\theta$ value of  $26.65^{\circ}$ .



### 4. Conclusion

This study aims to evaluate if it is possible to substitute quartz with palm oil fuel ash (POFA) and to find out how heat treatment and sintering temperature affect the microstructure, and physical, and mechanical qualities of porcelain tile. To conclude, POFA is a good option to replace quartz in porcelain. The maximum mechanical strength and bulk density of around 310 MPa and 2.529 g/cm<sup>3</sup> were attained for 900 °C TPOFA porcelain, for which strong mechanical strength and densification are two of the most important conditions for porcelain production. The maximum physical properties for porcelain samples containing 900 °C treated POFA occurred at a temperature of 1125 °C, on 10 wt% substitutions. Changes in the microstructure are necessary for an increase in the physical characteristics. Fewer pores and small particles enhance the quality of porcelain. Technically and economically, replacing quartz with palm oil fuel ashes (POFA) is thus practicable. The challenges associated with the disposal of excessive POFA can be alleviated by using this industrialwaste in the production of low-cost goods. In addition, the return of POFA, which is now regarded as abyproduct of palm oil processing companies, into the ceramic industry's value chain helps the circular economy and the achievement of a true industrial symbiosis.

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