



# Physico-Mechanical Properties of Porcelain by Substitution of Quartz with POFA Treated with 2M Hcl Acid

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## Abstract

Palm oil fuel ash (POFA) is a by-product from thermal power plant where palm kernel, shell and fibre used as fuel to generate electricity and disposed with no economic value. POFA is used as quartz replacement in the production of porcelain. POFA was dried in an oven for 24 hours at 110 °C, ground at a speed of 250 rev/sec for 12 hrs. Some of the POFA amount was treated with 2 Molar of HCl acid and some was kept untreated. Both treated and untreated were substituted with quartz at 15 wt% and mixed with porcelain composition and dry pressed into pellets at a mould pressure of 91 MPa and sintered at 1150 °C, 1200 °C and 1250 °C for 2 hrs soaking time respectively. XRF revealed that, POFA has similar chemical composition with quartz. The highest compressive strength, bulk density and Vickers microhardness being achieved at sintering temperature of 1150 °C using treated sample with the values 169 MPa, 2.432 g/cm<sup>3</sup> and 774 HV respectively. HCl treated POFA is a good candidate for quartz replacement and 1150 °C was the best sintering temperature.

**Keywords:** Bulk Density; Compressive Strength; Palm Oil Fuel Ash (POFA); Porcelain; XRD.

## 1. Introduction

An extensive research have been carried out to find an efficient, effective and available materials that has similar chemical composition with either quartz, feldspar or clay to be used as replacement in porcelain tile production in order to reduce cost of production of porcelain. One of the promising waste materials that is rich in alumina (Al<sub>2</sub>O<sub>3</sub>) and silica (SiO<sub>2</sub>) is palm oil fuel ash (POFA). This two chemical compounds are so vital due to their fineness and porous nature that enable them to be used to improve strength, reduce sulphate attack, enable good finishing, improved permeability and durability of final product and above all reduce cost of production [1]. The uses of waste materials in ceramic and construction industries such as palm oil fuel ash (POFA), fly ash (FA) and silica fume (SF) are one of the breakthrough of this century, as this make the industries environmentally friendly and give way for sustainability [2].

Palm oil tree is a tropical tree that can be found in tropical countries like Malaysia, Nigeria Thailand and Indonesia. However, The largest world production come from three ASEAN countries due to their strategic geographic location to the equator line (oil palm market monitor, 2016). Shafiqh et al., reported that annually Malaysia produces 7 million tonnes of crude palm oil which is less than that of Indonesia and 100, 000 tonnes was produced by Thailand [3].

POFA is an agro-waste material produced from burning residues of palm oil such as palm kernel, palm oil shells and fibres in a proportion of 85 % fibres and 15 % shells at a temperature of 800 °C -1000 °C to heat the boiler and produce energy for electricity generation in the palm oil power plants. 5 % of POFA is disposed as solid waste to the environment [1], [4]. Thus, POFA is a promising material to be used as cement replacement in the

construction industries or quartz in the ceramic industries due to its abundance as agricultural waste [5]. Similar research on the abundance of POFA reported that palm oil is the most frequently used vegetable oil for domestic and industrial used such as cosmetics and oleo chemicals, thus leading to the disposal of POFA as waste material. [6].

In another research by Khankhaje et al., reported that, to increase the compressive strength and durability of concrete, the use of original palm oil fuel ash (OPOFA) as partial replacements for cements is recommended [7]. This is supported by other researchers Hassan and Abdu reported that, POFA is an agricultural waste known to acquire pozzolanic behaviour [5]. POFA from chemical analysis is reported to have high quantity of silica, thus it is expected to be used as cement or quartz replacement, this also serve as an opportunity as a cheapest source of silica to reduce cost of production [8].

There is the need to utilize the industrials waste and reduce natural consumption, thus the use of POFA as quartz replacement will contribute towards that. As the urbanization, population and industrialization increases, it is expected that the waste disposal will also increase [9]. Recently, attention has been given to the effective and efficient utilization of different forms of waste for sustainability in green construction and cost reduction in ceramic industries [10], [11]. Thus, one of the most fundamental issue of waste management in the world is the utilization of waste materials such as POFA.

Porcelains are generally vitreous ceramic materials that are baked at higher temperatures. The triaxial porcelain consists of feldspar, clays mineral and quartz which after firing developed glassy, residual quartz and mullite [12]. Due to its high mechanical strength, durability, low water absorption and translucency, porcelains apart from household they are extensively used for science and engineering applications [13]. Porcelain tiles that were

previously used for floor are now used as ornaments in houses and internal wall, thus a slightly different properties are required for these applications such as mechanical designs that are directly related to the body microstructure [14].

## 2. Methods

The removal of moisture content from POFA is necessary. Thus, POFA was dried in an oven for 24 hrs at 110 °C and grind using ball mill machine at a speed of 250 rev/sec for 12 hrs, the powder was divided into 2 and level A and B. Part A was treated with 2 Molar of HCl acid and part B is kept untreated. Treated POFA (part A) was substituted with quartz at 15 wt% and mixed with 50 % clay, 25 % feldspar and 10 % quartz making triaxial porcelain composition. Similarly, untreated POFA (part B) was substituted with treated (part A) at same weight percent and the two compositions were dry pressed into pallet at a mould pressure of 91 MPa and sintered at 1150 °C and 1200 °C and 1250 °C for 2 hrs soaking time respectively. Vickers micro hardness, Universal Testing Machine and Shimadzu HMV-2 series were used to analyse the hardness, compressive strength and bulk densities of the porcelain samples. Thus, the microstructural and phase analysis were investigated using Scanning Electron Microscopy and X-ray diffraction analysis.

## 3. Results and discussions

To identify the feasibility of using POFA as quartz replacement in the porcelain production and its chemical composition, X-ray fluorescence analysis (Bruker S4 Pioneer model) operated at 60 KVP and 50 mA was used. Table 1 below shows the chemical composition of POFA.

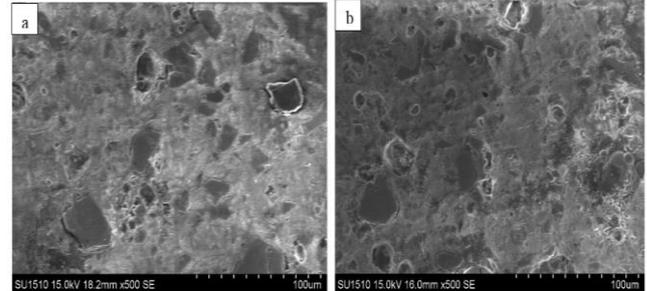
**Table 1:** Chemical composition of POFA treated with 1, 2 and 3 Molar HCl

Chemical Composition	Different Molarities of Acid (HCl) (wt%)		
	1 molar	2 molar	3 molar
SiO <sub>2</sub>	54.33	56.17	59.77
C	6.22	7.43	8.34
CaO	5.63	6.47	4.69
K <sub>2</sub> O	5.63	5.86	4.65
P <sub>2</sub> O <sub>5</sub>	6.65	5.73	3.20
Fe <sub>2</sub> O <sub>3</sub>	5.20	5.16	4.37
Al <sub>2</sub> O <sub>3</sub>	4.74	4.71	3.36
Cl	8.25	4.61	8.21
MgO	1.88	2.32	1.56
SO <sub>3</sub>	0.91	0.94	1.34
TiO <sub>2</sub>	0.30	0.29	0.24
MnO	0.06	0.08	0.05
Cr <sub>2</sub> O <sub>3</sub>	0.03	0.06	0.05
ZrO <sub>2</sub>	0.03	0.05	0.04
CuO	0.04	0.04	0.05
SrO	0.02	0.03	0.02
ZnO	0.02	0.02	0.02
Na <sub>2</sub> O	0.01	0.01	0.01
Rb <sub>2</sub> O	0.02	0.01	0.01
NiO	0.01	0.01	0.01

From Table 1, it is clear that POFA constitutes several chemical compounds with SiO<sub>2</sub> as the major composition. Similar result for the chemical composition of quartz was reported by Jamo et al., [15]. Thus, POFA is a promising candidate for replacement of quartz at certain percentage due to their similarity in chemical composition with quartz.

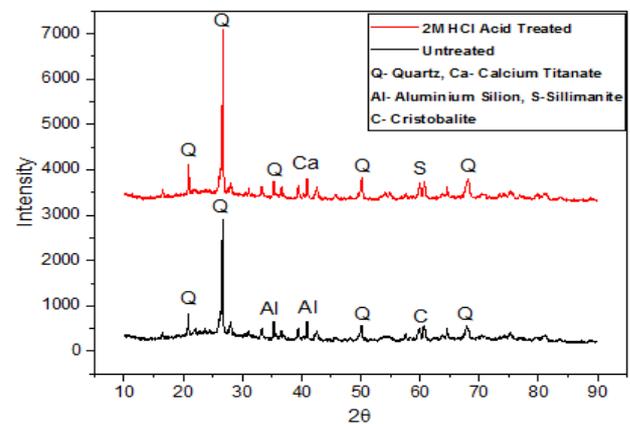
Microstructure of the sample was determined using Scanning Electron Microscopy. The micrographs of the acid treated and untreated samples as shown in Figure 1. It's clearly seen that the porosities are different for the two samples, untreated sample

appears to be more porous with hole-like on the surface that may lead to lower compressive strength, meanwhile the acid treated sample indicated a less porous and hence may have a higher strength. The result further revealed that, the acid treated sample shows an interfacial bond that is stronger than the of untreated sample, this ultimately lead to the reduction in the pore size and micro cracks. Thus, due to the reduction in the pore size and cracks, it is expected that, this yield to an increased compressive strength and bulk density [9].



**Fig. 1:** SEM for (a) 2M HCl acid treated and (b) Untreated Sample

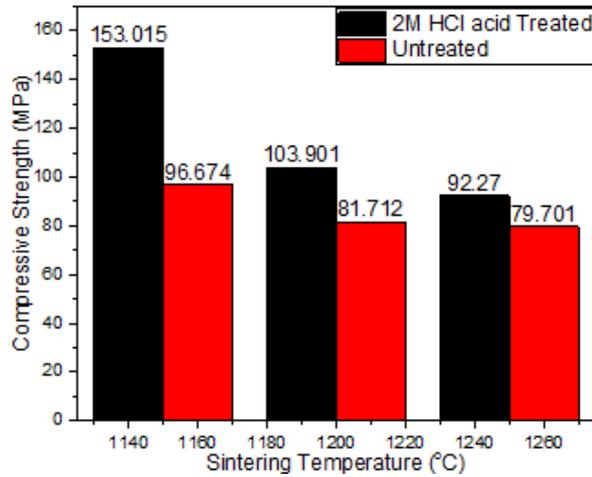
X-ray diffraction analysis was used to determine the mineralogical compositions of untreated and 2M HCl acid treated and presented in Figure 2. Cu- $\alpha$  radiation at diffraction angle of  $10^\circ \geq 2\theta \geq 90^\circ$  was used to analyse primary and secondary wave at scanning rate of 0.05°. The diffractograms disclose that, acid treated and untreated samples shows a similar pattern of peaks with predominantly quartz as major peak. Cristobalite, calcium, aluminum and sillimanite as minor, it further revealed that the acid treated shows higher peak of quartz at  $2\theta$  value of 26.652°, it can be observed that, the results are very much consistent due to the pattern of the peaks [16] [17]. Furthermore, this figure demonstrated that, after acid treatment the peak crystallizes and became clearer and more visible whereby some of the noise disappeared.



**Fig. 2:** XRD of 2M HCl acid treated and untreated sample.

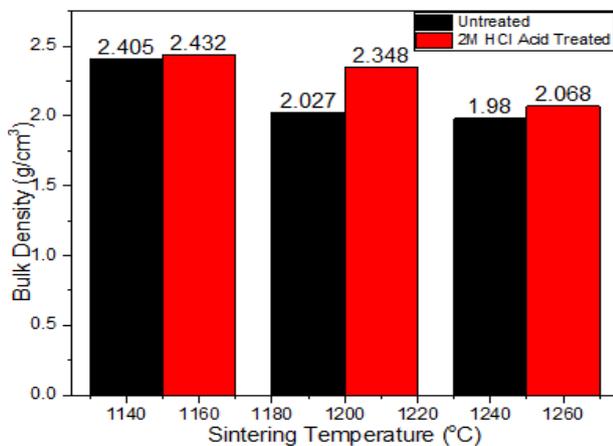
Due the economic advantage of mechanical strength of porcelain tile in the ceramic industries, previous researchers have studied it extensively such as [18]. The strength of a porcelain is measured as a function of temperature; hence, Figure 3 shows the compressive strength of porcelain samples made from untreated and treated POFA at different sintering temperatures. It is obvious that, the maximum compressive strength was achieved at the sintering temperature of 1150 °C that is 169 MPa. Thereby, as the sintering temperature increases to 1200 °C the value drop to 155 MPa, it further drastically drop again as the temperature reached 1250 °C to the lowest value of 153 MPa, these changes in the compressive strength is related to the porosity development of the body [18]. Thus, it is pronounced that, as the sintering temperature reach 1200 °C, the compressive strength starts to decrease which

is due to closed porosity development and liquid phase that invariably decrease affect the microstructure. To obtain the maximum strength of porcelain, porosity has to be zero, as the heating continues, the value of compressive strength continue to decrease.



**Fig. 3:** Compressive strength porcelain made by substitution of quartz with POFA at 15 wt% for acid treated POFA and untreated POFA for different sintering temperature.

As the strength of porcelain tile depends on microstructure, it is established that bulk density is related to the compressive strength of porcelain [19]. Lerdprom et al., stated that, for standard porcelain bulk density development depends on temperature and thus, densification of porcelain starts at temperature as low as 800 °C [12]. The bulk density of sample made from untreated, acid treated at different sintering temperature of 1150 °C, 1200 °C, and 1250 °C is presented in Figure 4.

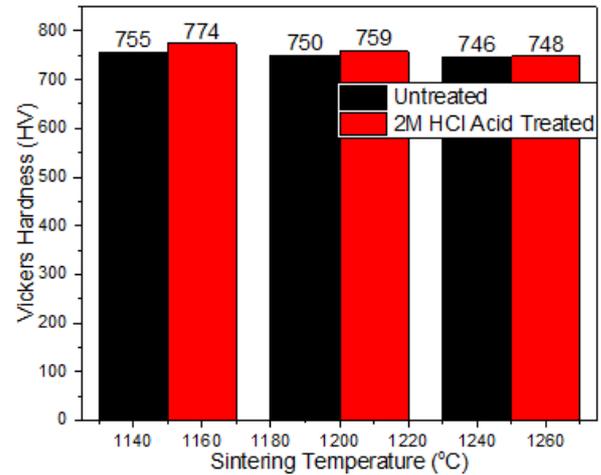


**Fig. 4:** Bulk density of sample made from untreated and acid treated POFA at sintering temperature of 1150 °C, 1200 °C and 1250 °C.

Therefore, based on the foregoing pattern of compressive strength, Figure 4 above revealed that, 2.432 g/cm<sup>3</sup> is the highest bulk density achieved at sintering temperature of 1150 °C for the 2 M acid treated sample. The result exhibit an increase in bulk density until the sintering temperature reaches 1200 °C, the value start dropping to 2.348 g/cm<sup>3</sup> and then later to 2.068 g/cm<sup>3</sup> as the temperature reached 1250 °C. Similar result was reported by Lerdprom et al., which conclude that, bloating leads to this significant decrease in the bulk density if samples were sintered beyond the optimal temperature, in this case the effect of bloating is higher than the heating rate [13]. Eren et al., reported that, it is a common relationship for porcelain tile as the sintering temperature increases the bulk density also increases after which upon

reaching maximum value as the heating continues it start to decrease [20].

To measure the mechanical properties of porcelain tile, Vickers micro hardness is probably one of the easiest way. To measure the micro hardness of porcelain, a rod tip is pressed on top of the material thus creating a deformation which is measured by the dimension of the indenter formed [19]. Figure 5 shows the micro hardness graphs against sintering temperature for heat-treated and untreated samples using 3 different sintering temperatures.



**Fig. 5:** Vickers micro hardness for 2 M HCl acid treated POFA and untreated POFA at 1150 °C, 1200 °C and 1250 °C sintering temperature.

From Figure 5, it can be interpreted that, 2 M HCl acid treated sample has the maximum value of hardness at a temperature of 1150 °C as 774 HV, as the sintering temperature increases to 1200 °C the value drops to 759 HV and then to 748 HV by 1250 °C. Likewise, similar trend was noticed for untreated sample as 755 HV, 750 HV and 746 HV for 1150 °C, 1200 °C and 1250 °C respectively. This is in agreement with other finding, which concludes that, the result of Vickers micro hardness serves as the screening test to the compressive strength result. Domenico and Tasonne reported that, the dimension of the indentation produced determine the hardness of the material [21]. Thus, mechanical strength of microstructure of the material such as micro cracks and defects greatly affect the micro hardness of the sample [22].

#### 4. Conclusion

The aim of this research is to investigate the feasibility of using palm oil fuel ash (POFA) as quartz partial replacement and to determine the effect of HCl acid treatment and sintering temperature on the mechanical properties of porcelain tile. Based on the finding of this research it is concluded that, POFA has similar chemical composition with quartz and thus, conclusion have been drawn that it can be used as partial replacement of quartz to certain weight percent (15wt%). It believed that, HCl acid treated sample shows an improved physical and mechanical properties such compressive strength, bulk density and Vickers micro hardness respectively.

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