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Effect of Iron (111) Oxide (Fe_2O_3) as an Additive and Substitution of Quartz with POFA on Physico-Mechanical Properties of Porcelain

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ABSTRACT

Porcelain tile is a translucent ceramic material developed from the mixture of clay, feldspar and quartz. Its excellent functional and technical properties such as low water absorption, low porosity, stain resistance; high bending and compressive strength make it indispensable for industrial activities. This research aimed to determine the effect of the addition of iron (111) oxide (Fe_2O_3) on physicomechanical properties of porcelain. Quartz was substituted with treated Palm Oil Fuel Ash (POFA) at 15wt% and mixed homogeneously with porcelain compositions using ball mill machine for 12 hours at a speed of 250 rev/sec and dry pressed at a moulding pressure of 91 MPa and sintered at 1150°C for 2 hours soaking time. Iron (111) oxide (Fe_2O_3) was added at 1, 2, 3, 4, 10 and 15wt% to the compositions of porcelain, dry pressed and sintered at the same temperature. The maximum value of compressive strength, bulk density and Vickers hardness were achieved by adding Fe_2O_3 at 5wt% as 138.94 MPa, 2.515 g/m^3 and 829 HV respectively. It is revealed that the addition of Fe_2O_3 at 5wt% enhanced both physical and mechanical properties of porcelain.

Keywords: Palm Oil Fuel Ash (POFA), Bulk Density, Compressive Strength, Vickers Micro-Hardness.

1. INTRODUCTION

Palm oil factories use palm fibre, empty fruit bunches and palm shells as fuel at a temperature between 800°C to 900°C to heat the boiler and generate electricity; the ash produced in the process is known as Palm Oil Fuel Ash (POFA). In Thailand alone, it is reported that annually, more than 100,000 tonnes of POFA is produced and disposed of with no any economic value, only some are utilized in concrete as cement partial replacement. Similarly, in Malaysia, 60,000 tonnes is annually produced [1]. Researchers [2] discover the potentials of high Fineness Palm Oil Fuel Ash (FPOFA) as a pozzolanic material that can be used to replace type 1 Portland cement at 30wt% of binder. By the addition of POFA to the cement, the compressive strength is improved and there is evidence of good resistance to chloride penetration [3]. Another research by [4] indicated that high fineness POFA can reduce water permeability and drying shrinkage of concrete.

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In Malaysia and other African countries, palm oil industries are one of the most important agricultural industries. The report suggested that in Malaysia alone, there is an approximate of 10 million tonnes of waste by the palm oil industries annually, which include POFA, fly ash, farm fibre, empty fruit bunches and palm oil shell [5].

POFA is supplementary cementitious material that has been studied for the past 2 to 3 decades due to its environmental, economic and engineering advantages [6]. This is because POFA is waste material that is disposed of as landfill and requires stockpile. Thus, recycling waste materials leads to a reduction in the cost of production in the ceramic industries and solve environmental imbalance. Moreover, incorporating POFA in cement or porcelain production has been proven to improve the physicomechanical properties of the final product [7].

Due to the growing threat posed by climate change, depletion of ozone layer and global warming, international authorities have imposed uncompromising legislation on the environment which also applies to palm oil industries that consistently burnt palm oil waste and produce excess energy and emission of greenhouse gasses [8].

Previously, researchers mainly utilized POFA in Ordinary Portland Cement (OPC) as replacement up to 20% without sacrificing either durability or compressive strength [9]. Looking at the quantity of POFA produced, this shows only a few amounts were utilized, as for the utilization in porcelain production, the author noticed a dearth of available research in the reviewed literature [1].

Porcelain products have been used as ceramics for tiles, sanitary ware, tableware, and many scientific and engineering applications several years ago [10]. Basic porcelain properties to be considered during productions include impermeability of water (lack of open porosity), durability, mechanical and chemical properties. Pressing and slit casting are the main two technologies adopted for producing porcelain objects, hence both technologies are deemed efficient by the ceramic industries for the high volume of production.

According to ISO 13,006, porcelain tile is grouped into Ala and Bla due to its vitreous property and low water absorption of $\leq 0.5\%$. Among the ceramic tiles, porcelain stoneware possesses several technical and exquisite properties due to its low water absorption, mechanical strength and low porosity. Two types of porcelain currently attracting attention are glazed (which received vitreous surface coating) and unglazed (also known as technical porcelain which is formulated by support layer). The support layer composed of feldspar (fluxing agent), quartz (inert raw materials), and clay [11].

Due to an increase in demand of higher quality of the finished product, production of porcelain floor and wall tiles have seemingly increased. The ceramic and construction industries have now focused their research on the incorporation of waste from palm oil industries, building, sanitary waste, metallurgy dust and several ashes into the ceramic bodies and obtain high mechanical and physical properties [12] [13] in order to reduce the cost of traditional porcelain, and also due to the large production of solid waste generated and disposed by palm oil industries.

Standard porcelain generally consist of 50% clay (plasticizer), 25% feldspar (fluxing agent) and 25% quartz (filler) [14][15][16]. Based on the above formulations, porcelain was classified as highly vitreous, translucent and dense white ceramic [17]. Porcelain has been known for its outstanding functional features and excellent technology, such as good mechanical properties that make porcelain workable for industrial and congestion areas. Low water absorption of porcelain makes it resist liquid permeability and well resistant to frost. Moreover, porcelain has proven to have good resistance to chemical attack and it is also easy to lean [17]. This research investigates the effect of the addition of iron (111) oxide on physicomechanical properties of porcelain.

2. MATERIALS AND METHOD

POFA was collected from Pamol Plantations Sdn Bhd, Kluang Johor, Malaysia. The powder is dried in an oven to remove the moisture and was ground to particle size ≤ 50μm to enhance the silica (SiO_2) production. After treating POFA with 2 molar HCl acid, quartz was substituted with POFA at 15wt%, mixed homogeneously with porcelain compositions using ball mill machine for 12 hours at a speed of 250 rev/sec. The mixed porcelain sample was divided into two and labelled as part 1 and part 2. Part 1 was dry pressed at a moulding pressure of 91 MPa and sintered at a temperature of 1150°C for 2 hours soaking time in order to determine the effect of quartz replacement with POFA. Iron (111) oxide (Fe_2O_3) was added at 1, 2, 3, 4 5, 10 and 15wt% to part 2 and the effect of its addition were studied. Both samples were dry pressed at 91 mould pressure and sintered at 1150°C for 2 hours soaking time. To investigate the effect of replacement of quartz with POFA and addition of iron (111) oxide (Fe_2O_3) on physicomechanical properties of porcelain, x-ray fluorescence analysis, X-ray Diffraction (XRD) analysis, Scanning Electron Microscopy (SEM), Vickers microhardness, compressive strength test and bulk density were conducted.

3. RESULTS AND DISCUSSION

X-ray fluorescence analysis is a proficient machine used to determine the chemical compositions of POFA. Bruker S4 Pioneer model operated at 60 KVP and 50 Ma was used and the result is presented in Table 1.

Table 1 The chemical composition of POFA

Chemical Composition	HCl acid Treated			Heat treated (wt%)	Untreated (wt%)
	1 Molar (wt%)	2 Molar (wt%)	3 Molar (wt%)		
SiO_2	41.7	43.2	36.3	40	42.2
C	0.1	0.1	0.1	0.1	0.1
CaO	8.01	7.75	8.53	8.77	8.09
K_2O	7.83	8.62	6.27	9.5	7.68
Cl	6.27	0.95	14.4	1.06	5.49
P_2O_5	3.99	3.81	4.77	4.27	4.13
MgO	3.05	2.42	1.92	2.79	2.94
Al_2O_3	2.74	2.68	3.38	3.26	2.76
Fe_2O_3	2	2.65	2.13	2.97	2.37
SO_3	1.52	1.14	1.14	1.44	1.33
TiO_2	0.21	0.27	0.24	0.27	0.25

POFA mainly consists of silica (SiO_2) and other oxides in a smaller percentage. From Table 1, it can be presumed that treatment plays a vital role in altering the chemical compositions. It is clear that 2 molar acid treatment is selected to be the best method for POFA treatment due to the higher silica content, this indicates that POFA is a good candidate as quartz replacement and as pozzolanic materials that react with calcium hydroxide at room temperature and form cementitious material for construction industries [18]. A similar result was obtained by [19][20].

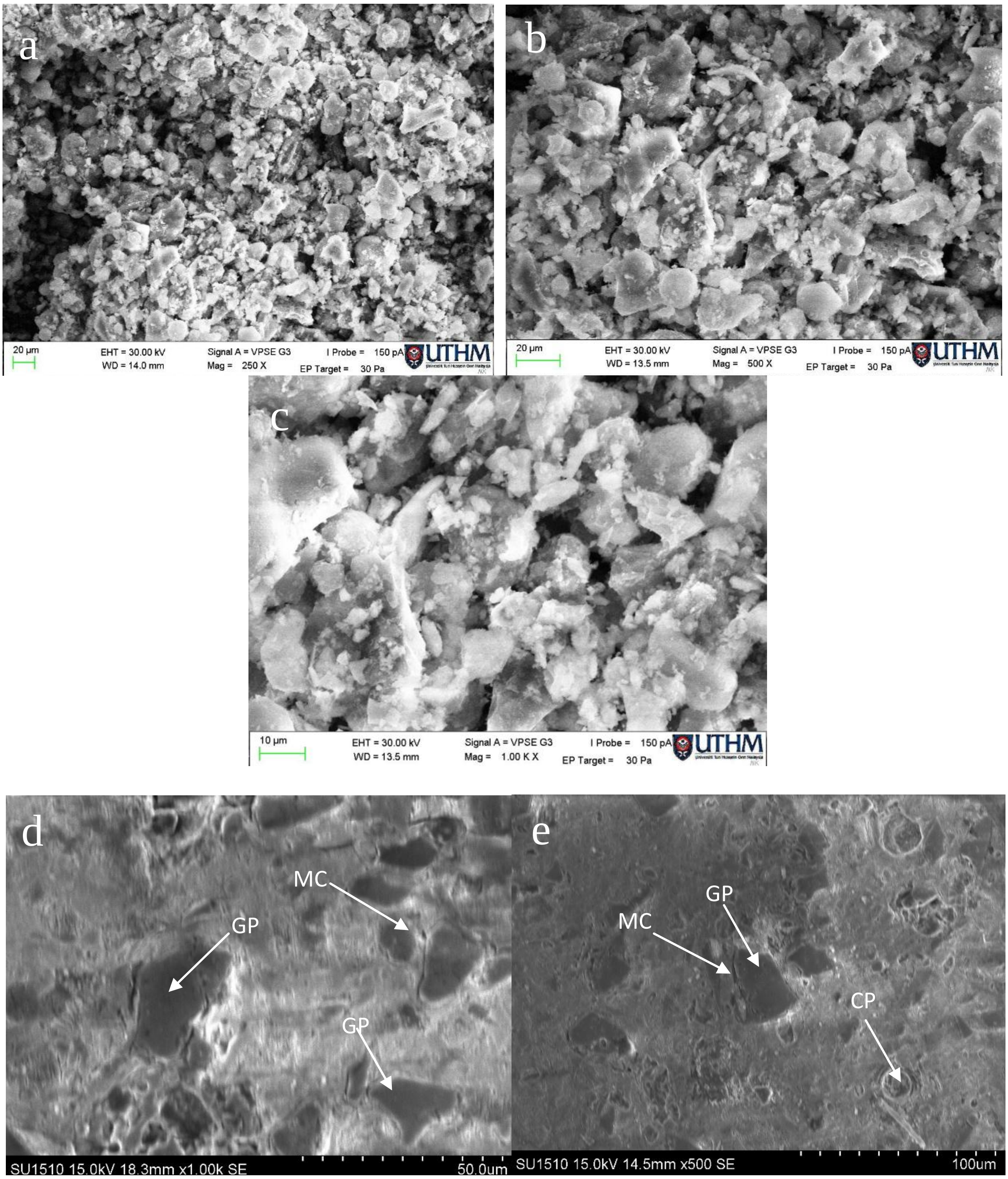

Figure 1. SEM of POFA powder (a,b and c) and porcelain without addition of ferum Fe_2O_3 (d) and after addition (e). [MC = micro crack, CP = closed porosity, GP = Glassy phase] (d) and after addition (e). [MC = micro crack, CP = closed porosity, GP = Glassy phase].

The morphology and microstructural analysis were conducted using SEM (JOEL-JSM 6380 model) operate at 15 KV. Figure 1 shows the SEM of POFA powder and porcelain powder.

The SEM micrograph in Figure 1 (a, b and c) revealed that POFA powder is very porous that possess spongy structure and amorphous in nature. After acid treatment, the powder particles agglomerates and compacts together.

It is good to note that, the properties and microstructural behaviour of porcelain are directly affected by the homogeneity of the powder during processing. The type of powder used, the distribution of the particles and its size are the determinant factors of the physicomechanical properties of porcelain [10]. The microcracks present in Figure 1 (d and e) is due to the crystalline quartz and glassy matrix mismatch as a result of thermal expansion; this was supported by [21]. Another research reports that cracks are normally generated within and around quartz particles that have a natural effect on the mechanical performance of the final product [22][23]. Figure 1(d) clearly portrays a typical characteristic of porcelain which is the presence of low porosity. It is also observed that after the addition of Fe_2O_3 there is evidence of closed porosity and complete densification and crystallization as shown in Figure 1 (e).

In order to obtain the intensities of the x-ray as a function of angle between the incident and the diffracted beams, X-ray Diffraction (XRD) analysis machine was used, Cu-Kα radiation with a scanning rate of 0.05° per second 40KV/40mA at an angle of 10° ≥ 2θ ≤ 90° was adopted and the result is presented in Figure 2 below.

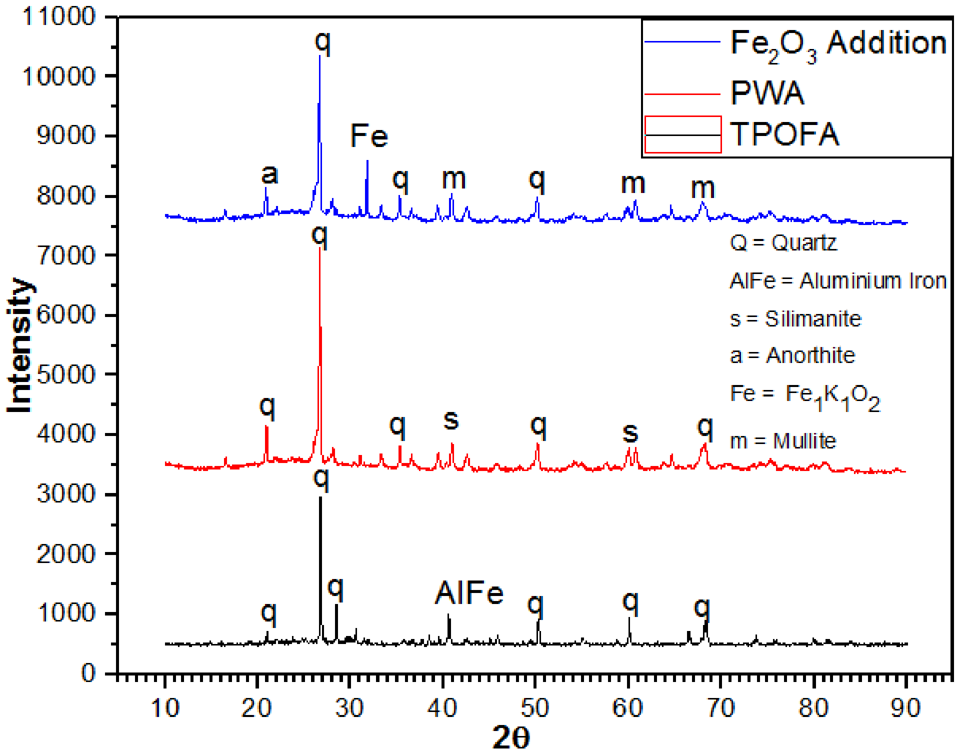

Figure 2. XRD pattern of treated POFA (TPOFA), porcelain without the addition of Fe_2O_3 (PWA) and porcelain with the addition of Fe_2O_3.

Figure 2 shows the XRD pattern for all the samples with quartz as major composition and a few other compounds as a minor phase. The quantitative analysis of the peaks reveals that for porcelain, without the addition of Fe_2O_3 there is 46.5% quartz, 35.4% aluminosilicate, 5.1% calcium, and 13.1% sulfuric acid [24][25]. Whereas, after addition of Fe_2O_3, the quantity changes to 42% quartz, 32% mullite, 21% anorthite, and 5% iron which indicates the reaction of iron (111) oxide with other chemical compounds. From Figure 2, it can be deduced that the peaks of porcelain made with treated POFA are clear and crystallized with little impurities such as AlFe. Whereas, for the peaks of porcelain without the addition of Fe_2O_3 (PWA), the broadened peaks that make it difficult to indicate the actual peaks which ultimately affects the strength of the final products. Hence, by introducing Fe_2O_3, some peaks crystallize and the presence of mullite and anorthite lead to better densification and improved mechanical properties of porcelain ceramic.

It is obvious that, after the addition of Fe_2O_3 to the composition of porcelain, the second largest phase is Fe which greatly influences the drastic change in the bulk density and strength. Further investigation revealed that, although there is no significant change in the structure of the peaks, it is clear that for the porcelain sample with the substitution of POFA at 15wt%, the peak crystallizes and there are no impurities. Likewise, for the porcelain with the addition of Fe_2O_3, a decrease of peak intensity was observed and a presence of Fe as the second major peak was also discovered [17].

Universal Testing Machine (UTM) was used to measure the compressive strength of the porcelain tile formed with and without the addition of Fe_2O_3. A sample was placed between two level plates and an axial compressive load was applied to the sample surface until a certain load is reached or the sample is broken. Fe_2O_3 was added to the composition of porcelain at 1, 2, 3, 4, 5, 10 and 15wt% to determine its effect on the mechanical strength of the sample and the result is presented in Figure 3.

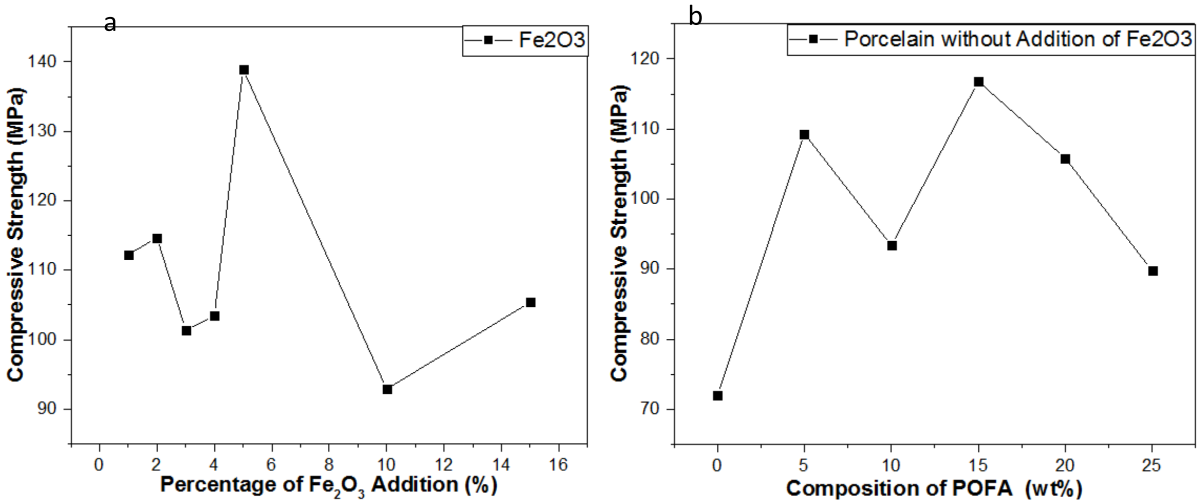

Figure 3. The compressive strength of porcelain with and without the addition of Fe_2O_3.

From Figure 3, it is sufficiently clear that porcelain formed with the substitution of quartz with treated POFA at 15wt% exhibit a compressive strength of 116.77 MPa. Whereas, after adding 1wt% of Fe_2O_3, the value is 112.25 MPa. As the percentage increased, the compressive strength also increased, except at 3wt%. The maximum value was achieved at 5wt% as 138.94 MPa, thereby after reaching maximum the value drop to 89.76 and 100.98 MPa at 10 and 15wt% respectively. A similar result was obtained by previous researchers for the addition of POFA to concrete at a certain weight percentage [26–29]. Thus, the highest compressive strength obtained corresponds to the highest bulk density is at 5wt% addition of Fe_2O_3 and sintering temperature of 1150°C, this could be due to the mullitization and increase in the liquid phase that leads to complete densification process and dissolution of quartz as suggested by [30]. Furthermore, for porcelain that consists of clays and feldspar, higher mullite formation is possible because as the temperature increases, the mechanical strength of the body also improves. Nevertheless, as the temperature reaches its climax, the mullite crystals became coarse, thereby decreasing the mechanical strength [31]. The compressive strength of porcelain was found to increase due to the addition of Fe_2O_3 owing to its dissolution with other crystals in the melt to form a solution, leading to enhanced mullitisation and formation of glassy phase and thus increase in compressive strength [32]. Therefore, based on the above mechanical strength pattern as a result of the addition of Fe_2O_3 at 5wt% of POFA, it is conclusively pronounced that Fe_2O_3 plays a vital role in the mechanical strength of porcelain at a certain weight percentage, after which by reaching the maximum percentage, it has a negative effect on the compressive strength.

Archimedes' principle of water displacement was used to determine the bulk density of the samples using Mettler Toledo XS64 model. The adopted technique is by weighing the sample in air and then immersed in water and weigh again. The density is calculated using the ratio of suspended mass in the air to the exterior volume of the sample, this is the difference between saturated mass as suggested by [17] and the result is presented in Figure 4 below.

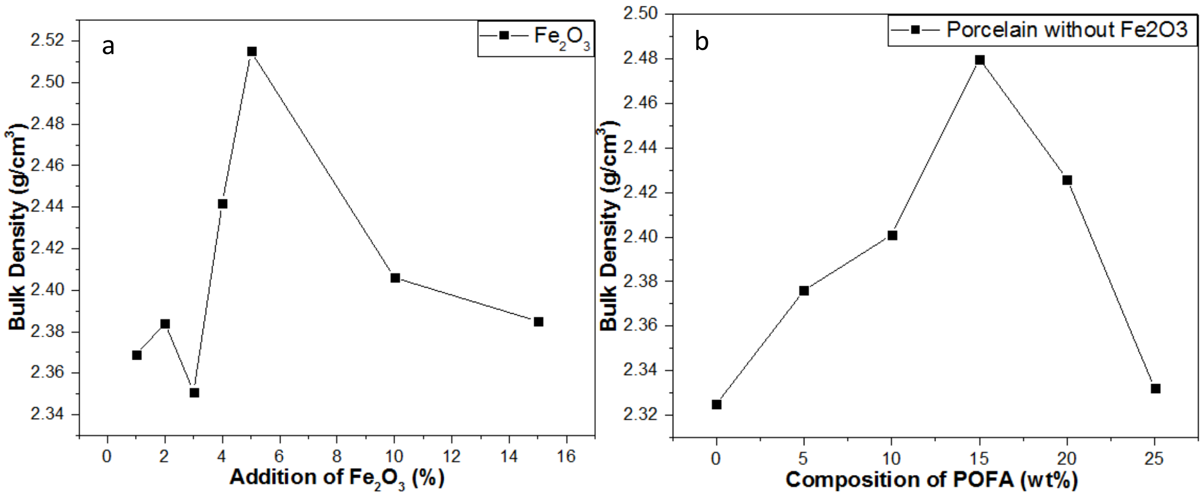

Figure 4. The bulk density of porcelain with and without the addition of Fe_2O_3.

It is evident that sample with the addition of Fe_2O_3 exhibits an increasing trend of bulk density from 1 to 5wt%. The bulk density increases as the amount of Fe_2O_3 addition increased with the maximum value of 2.515 g/m^3 for 5wt% addition. Figure 4 depicts that Fe_2O_3 addition from 1 to 5wt% increased the bulk density of porcelain whereby after reaching maximum, the value drastically drops for 10 and 15wt% addition. Findings of this research are in agreement with several other studies conducted on the addition of either fly ash or POFA to the composition of porcelain [24-25], [32-33] because Fe_2O_3 present in both ashes. Besides that, a sample without addition of Fe_2O_3 also exhibits an increasing trend of bulk density where the value drops to as low as 2.06 g/cm^3 with the maximum of 2.426 g/cm^3. The addition of Fe_2O_3 secondary mullite crystallized lead to the increased formation of mullite and subsequently improved the bulk density [32]. Conclusively, it is clear that the addition of Fe_2O_3 up to 5wt% of POFA greatly influence the compressive strength and bulk density of porcelain.

The conventional Vickers microhardness has been widely used to measure the hardness, toughness and tensile properties of the material. The process involves the use of a rigid indenter by toughening the surface of the material, and the dimension of the residual imprint left on the surface is measured. The ratio of applied load to the contact area between the indenter and material is known as the hardness of a material [35]. Shimadzu HMV-2 series was used to determine the Vickers microhardness of the sample and the result is presented in Figure 5.

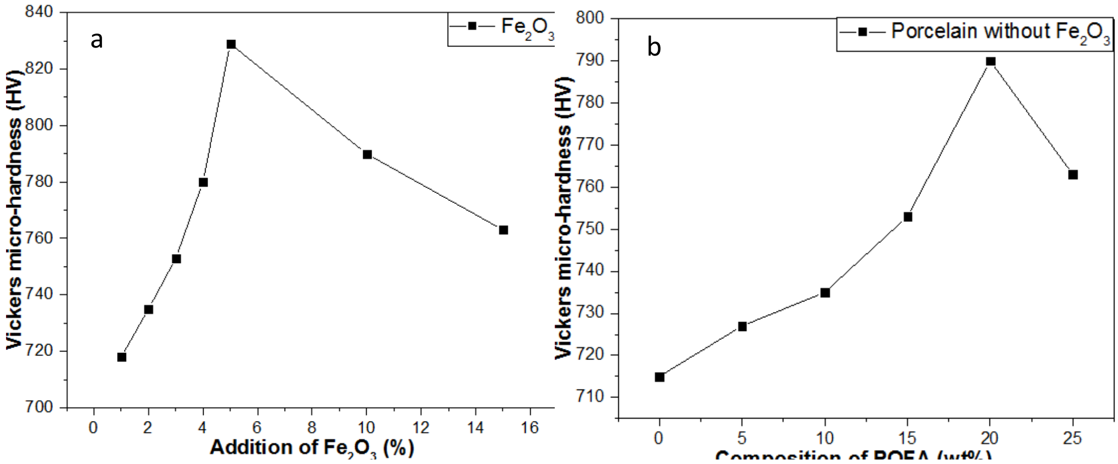

Figure 5. Vickers microhardness of Porcelain made with and without the addition of Fe_2O_3.

From Figure 5, the maximum hardness was determined when Fe_2O_3 was added at 5wt% of POFA with the maximum value of 829 HV. It is evident that the addition of Fe_2O_3 progressively increases the Vickers microhardness of the material up to a certain level, after reaching the maximum peak, the results show a reverse trend. Therefore, it can be concluded that porcelain made without the addition of Fe_2O_3 exhibits less Vickers microhardness compared to the one with the addition of Fe_2O_3 at 5wt%. The hardness of porcelain is greatly affected by the development of porosity, thus, the addition of Fe_2O_3 lead to the formation of glassy phase that subsequently reduced the porosity and improves the hardness of the material [36].

4. CONCLUSION

Effect of addition of Fe_2O_3 on physicomechanical properties of porcelain has been studied. It can be concluded that the addition of Fe_2O_3 at 5wt% of POFA treated with 2 molars of HCl acid increases both the mechanical and physical properties of porcelain. It is also noted that 1150°C is the best sintering temperature for this project. It is important to note that, this study reveals that after addition of Fe_2O_3 above 5wt%, both mechanical and physical properties such as compressive strength, bulk density and Vickers micro-hardness drop to the lowest value.

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