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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 addition of iron (111) oxide (Fe_2O_3) on physico-mechanical properties of porcelain. Quartz was substituted with treated 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 mould 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 5 wt% had enhanced both physical and mechanical properties of porcelain.

Introduction

Palm oil factories use palm fiber, 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 with no any economic value, only few is utilized in concrete as cement partial replacement, while in Malaysia 60,000 tonnes is annually produced [1]. Researchers like [2] discover some of the potentials of FPOFA (high fineness POFA) as a pozzolanic material that can be used to replace type 1 Portland cement at 30 wt% of binder. By 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.

In Malaysia and other African countries, palm oil industries are one of the most important agricultural industries. 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 fiber, empty fruit bunches and palm oil shell [5]

POFA is a supplementary cementitious materials that have been studies 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 as landfill and requires stockpile. Thus, recycling waste materials leads to reduction in cost of production in the ceramic industries and solve environmental imbalance, hence incorporating POFA in cement or porcelain production has been proven to improve the physico-mechanical 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 an uncompromising legislation on 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 few amount was 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, table wares 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 ≤ 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), this support layer is 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. To reduce the cost of traditional porcelain and also due to the large production of solid waste generated and disposed by palm oil industries, ceramic and construction industries have now focused their research on 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].

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 is classify as highly vitreous, translucent and dense white ceramic [17]. Porcelain have 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 make it resist liquid permeability and good resistant to frost, porcelain have proven to have good resistant to chemical attack and it is also easy to lean [17]. This research investigate the effect of addition of iron (111) oxide on physico-mechanical properties of porcelain.

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 15 wt%, 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 level part 1 and part 2. Part 1 was dry pressed at a mould pressure of 91

MPa and sintered at temperature of 1150°C for 2 hours soaking time 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 effect of its addition was 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 physico-mechanical properties of porcelain, X-ray fluorescence analysis, X-ray diffraction analysis, scanning electron microscopy, Vickers micro hardness, compressive strength test and bulk density were conducted.

Results and Discussion

X-ray fluorescence analysis is 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 below.

Table 1. 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 consist mainly of silica (SiO_2) and other oxides in smaller percentage. From the above table, 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 indicated that POFA is good candidate to be used as quartz replacement and as pozzolanic materials that react with calcium hydroxide at room temperature and form cementitious material for construction industries [18]. Similar result was obtained by [19][20].

The morphology and microstructural analysis was conducted using scanning electron microscopy (SEM), JOEL-JSM 6380 model operated at 15 KV. Figure 1 shows the SEM of POFA powder and porcelain powder.

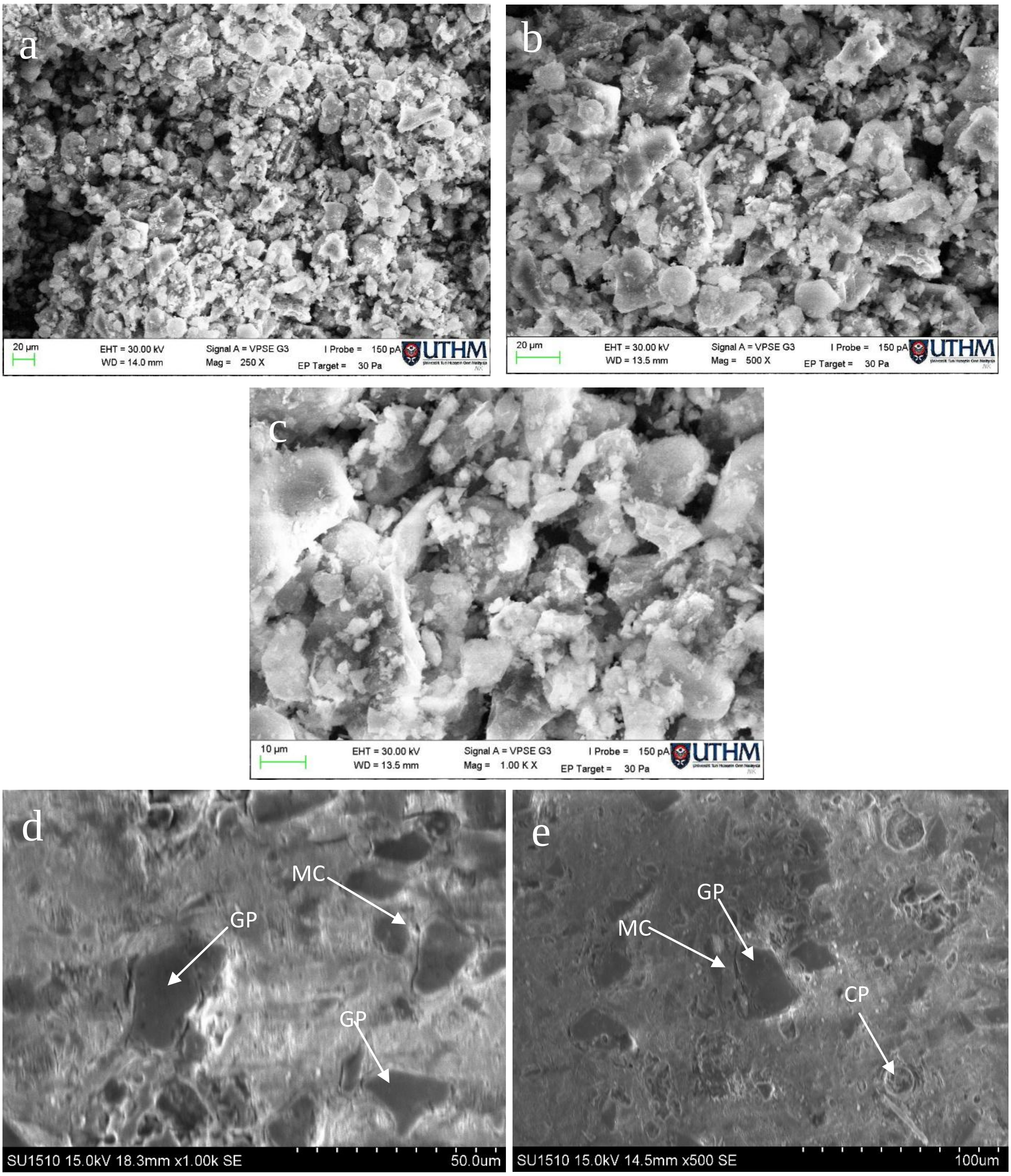

Figure 1. SEM of POFA powder (a,b and c) and porcelain without addition of ferum Fe_2O_3 (d) and after addition (e). [MC = micro crack, CP = closed porosity, GP = Glassy phase]

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, the powder particles after acid treatment agglomerates and compacts together.

It is good to note that, the properties and microstructural behavior of porcelain is 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 physico-mechanical properties of porcelain [10]. The micro cracks 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 presence of low porosity, it is also observed that after addition of Fe_2O_3 there is evidence of closed porosity and complete densification and crystallization, Figure 1 (e).

To obtain the intensities of the X-ray as a function of angle between the incident and the diffracted beams, X-ray diffraction analysis (XRD) machine was used, Cu-Kα radiation with scanning rate of 0.05° per second 40KV/40mA at an angle of $10° \geq 2\theta \leq 90°$ was adopted and the result is presented in Figure 2 below.

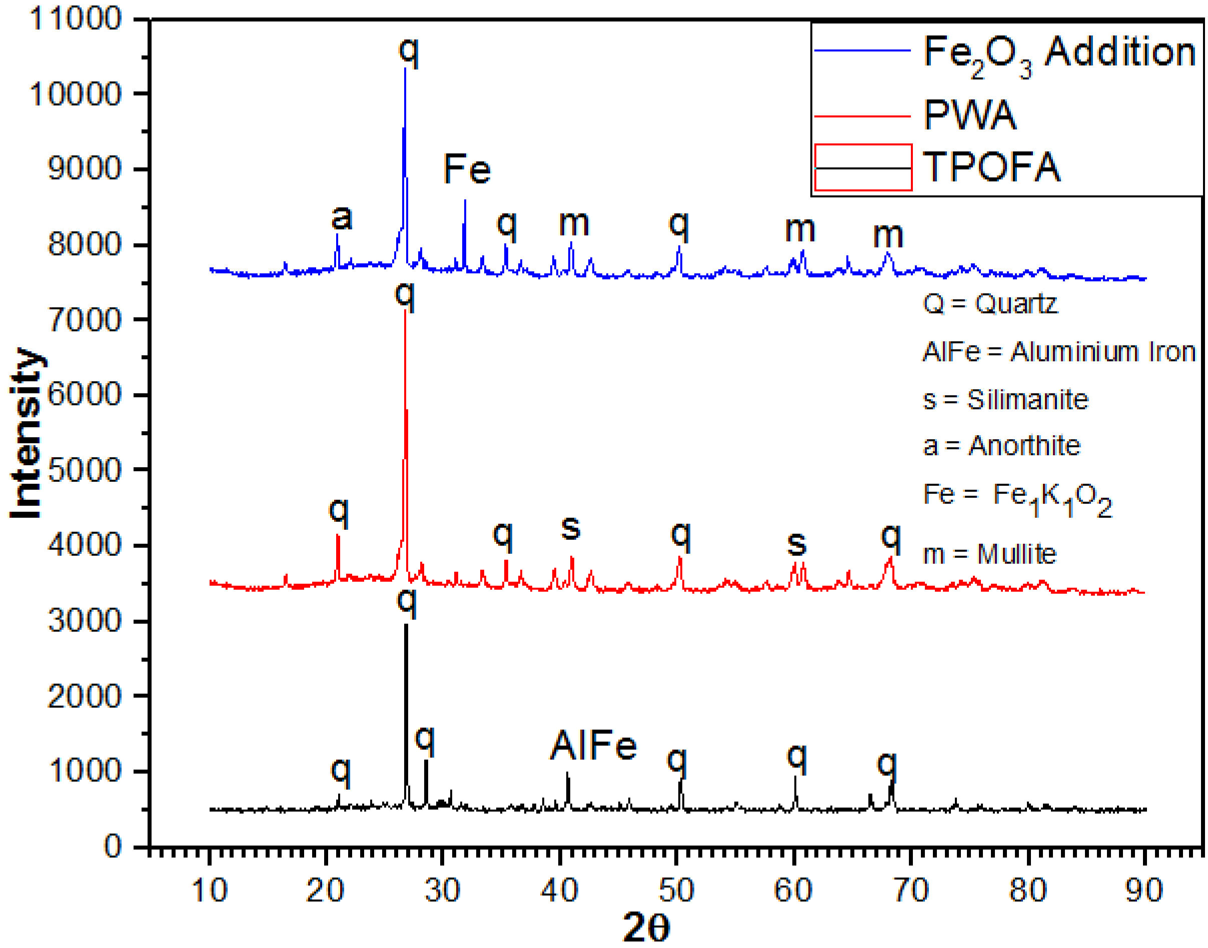

Figure 2. XRD pattern of treated POFA (TPOFA), porcelain without addition of Fe_2O_3 (PWA) and porcelain with addition of Fe_2O_3.

Figure 2 shows the XRD pattern for all the samples with quartz as major composition and few other compounds as minor phase. The quantitative analysis of the peaks reveals that, for porcelain without 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, its presence influences some changes in the upcoming analysis. 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 addition of Fe_2O_3, (PWA), the peaks are broadened that make it difficult to indicate the actual peaks present which ultimately affect the strength of the final products. Hence, by introducing Fe_2O_3, some peaks crystallizes and there is present of mullite and anorthite which their present lead to better densification and improved mechanical properties of porcelain ceramic.

It is obvious that, after addition of Fe_2O_3 to the composition of porcelain, the second largest phase is Fe which greatly influence 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, but it is clear that for the porcelain sample with substitution of POFA at 15wt% the peak crystallizes and there is no much impurities. Likewise, for the porcelain with addition of Fe_2O_3, a decrease of peak intensity was observed and a presence of Fe as 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 addition of Fe_2O_3, a sample is 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.

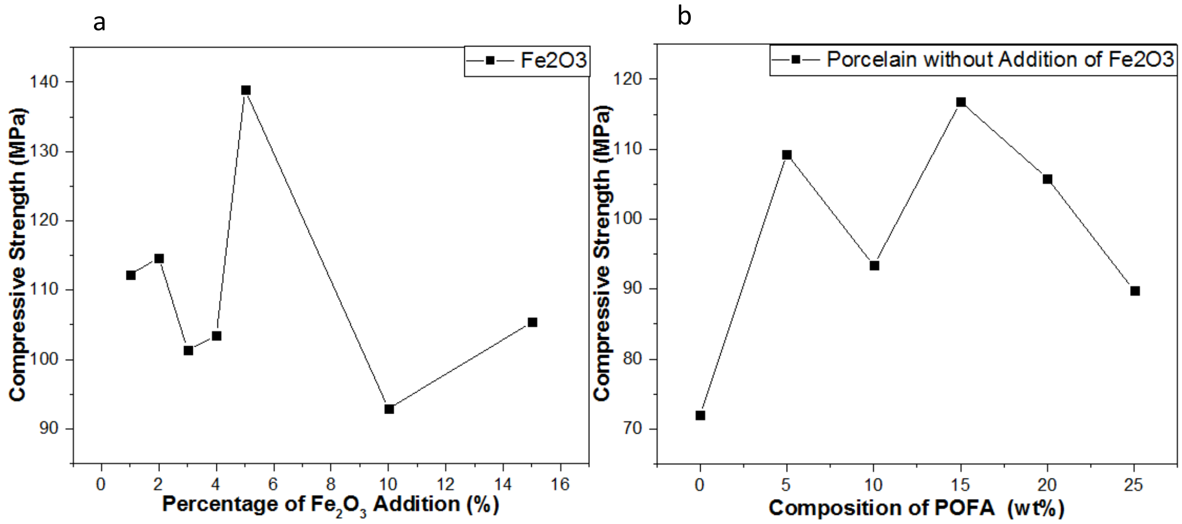

Figure 3. Compressive strength of porcelain with and without addition of Fe_2O_3

From Figure 3, it is sufficiently clear that, porcelain formed with substitution of quartz with treated POFA at 15wt% exhibit compressive strength of 116.77 MPa whereas after adding 1wt% of Fe_2O_3 the value was 112.25 MPa, as the percentage was increased so the strength, unless 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, similar result was obtained by previous researchers for the addition of POFA to concrete at certain weight percent [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 liquid phase that leads to complete densification process and dissolution of quartz as suggested by [30], it is further declared that, for porcelain that consists of clays and feldspar, higher mullite formation is possible as the temperature is increased which consequently improves the mechanical strength of the body, nevertheless as the temperature reaches its climax, the mullite crystals became coarse thereby decreasing the mechanical strength [31]. Compressive strength of porcelain was found to increase due to addition of Fe_2O_3 owing to its dissolution with other crystals in the melt to form a solution, this lead to the 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 addition of Fe_2O_3 at 5wt% of POFA, it is pronounced conclusively that Fe_2O_3 plays a vital role in the mechanical strength of porcelain at certain weight percent, 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 techniques adopted 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 air to 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.

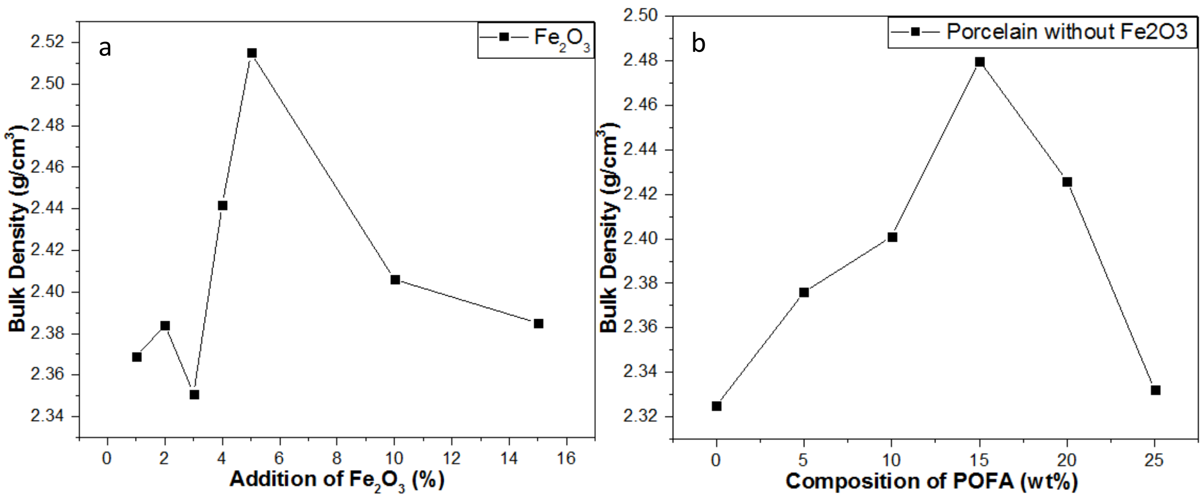

Figure 4. Bulk density of porcelain with and without addition of Fe_2O_3

It is very clear that sample with addition of Fe_2O_3 exhibits 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 above depicts that Fe_2O_3 addition from 1 to 5wt% increases the bulk density of porcelain whereby after reaching maximum, the value drastically drop for 10 and 15wt% addition. Findings of this research is in agreement with several other studies conducted on addition of either fly ash or POFA to the composition of porcelain [24-25], [32-33], this is so because Fe_2O_3 is present in both the two ashes. In other way, sample without addition of Fe_2O_3 also exhibits increasing trend of bulk density whereby at certain level the value drops to as low as 2.06 g/cm^3 with the maximum of 2.426 g/cm^3. Due to addition of Fe_2O_3 secondary mullite crystallized and this lead to increased formation of mullite and subsequent improved in the bulk density [32]. Conclusively, it is clear that addition of Fe_2O_3 up to 5wt% of POFA greatly influence the compressive strength and bulk density of porcelain.

To measure the hardness, toughness and tensile properties of material, conventional Vickers micro hardness has been widely used, this process involves the use of a rigid indenter by toughing the surface of the material, 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 hardness of a material [35]. Shimadzu HMV-2 series was used to determine the Vickers micro hardness of the sample and the result is presented in Figure 5 below.

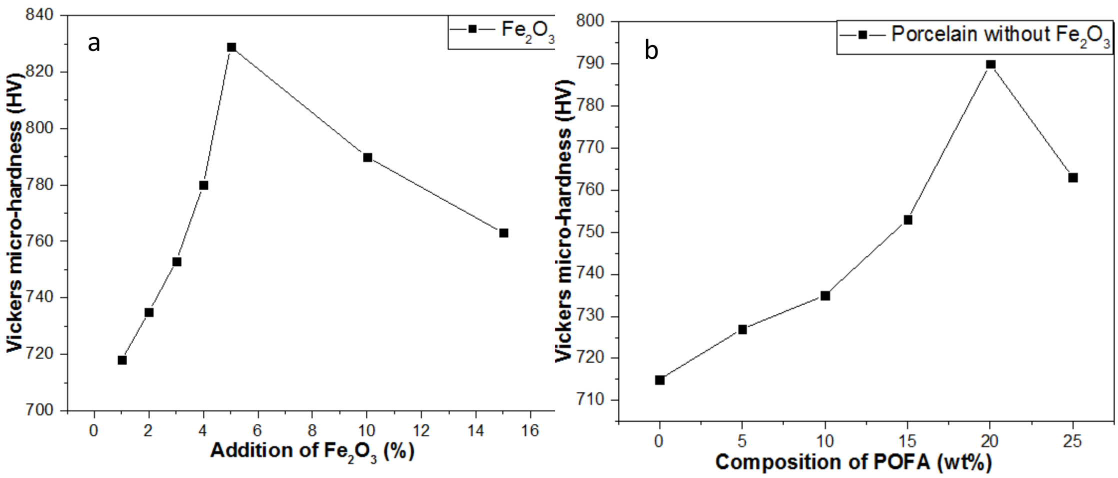

Figure 5. Vickers micro hardness of Porcelain made with and without addition of Fe_2O_3.

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 evidently clear that addition of Fe_2O_3

progressively increases the Vickers micro hardness of the material up to a certain level after reaching maximum the results shows a reverse trend. For this, it is enough to conclude that, porcelain made without addition of Fe_2O_3 exhibits less Vickers micro hardness than the one with addition of Fe_2O_3 at 5wt%. hardness of porcelain is greatly affected by the development of porosity, thus, addition of Fe_2O_3 lead to formation of glassy phase that subsequently reduced the porosity and improve the hardness of the material [36].

Conclusion

Effect of addition of Fe_2O_3 on physico-mechanical properties of porcelain have been studied, it can be concluded that addition of Fe_2O_3 at 5wt% of POFA treated with 2 molar of HCl acid increases both 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 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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