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# Three Points Modulus of Rapture (MOR) of Porcelain by Replacement of Quartz with Rice Husk Ash (RHA) And Palm Oil Fuel Ash (POFA) at Different Soaking Times

**Hassan Usman Jamo[1*], Mohamad Zaky Noh[2], Aminu Musa Liman[3], Salisu Abdu[4], Ibrahim Dauda Umar[5]**
[1,3, 4 & 5]Department of Physics,
Kano University of Science and Technology
Wudil, PMB 3244, Kano.
jamouhfce@gmail.com

[2]Faculty of Science,
Technology and Human Development,
Universiti Tun Hussein Onn Malaysia
86400 Parit Raja, Batu Pahat, Johor, Malaysia

## Abstract

*Porcelains in the quartz+kaolin+feldspar system with progressive replacement of quartz with rice husk ash (RHA) and palm oil fuel ash (POFA) were investigated to study the effect of soaking time on the three points modulus of rapture. The RHA and POFA were progressively incorporated into the porcelain body from 0wt% to 25 wt%. The powder was mixed and pressed into pellets at pressure of 91 MPa. All the pellets were sintered at a temperature of 1100 °C for 1h, 2 hs and 3 hs soaking times. The maximum modulus of rapture was achieved on 20 wt% of RHA and POFA containing porcelains at the soaking time of 2 h. But upon increasing the soaking time, after reaching the maximum level, the three points modulus of rapture decreases due to bloating of isolated pores and the disappearance of quartz, which are also associated with a decrease in bulk density. The distribution of closed pores, their geometric shapes and possible link with each other control the bending strength of the porcelain body. In addition, the presence of RHA and POFA in the porcelain body also increases the modulus of rapture.*

**Keywords**: MOR, SEM, XRF, XRD, Quartz

## INTRODUCTION

The utilization of agriculturalwaste materials has been found to be of increasing interest. The outer covering of rice grains obtained during the milling process otherwise knonwn as rice husk (RH), this, is one of the main agricultural residues. It mainly consists of hemicelluloses cellulose,lignin, silicon dioxides and other mineral composition (Noh *et al.,* 2016). Rice husk ash (RHA)is produced as a result of burning of RHs under the controlled temperature. The RHA is obtained as a waste product from the RH biomass power plants. The disposal of RHA in landfills could be cause serious environmental problems(Turmanova *et al.,* 2008;. Jamo *et al.*,2015; Hassan *et al.*,2014; Jamo *et al.*,2015; Jamo *et al.,* 2017a; Jamo *et al.,* 2017b; Noh *et al.,* 2017; Hassan *et al.,* 2015). The RHA contains substantial amount of amorphous silicon oxides,

**Author for Correspondence*

carbon dioxides, and small amount of other mineral composition. The amorphous silicon dioxides and carbon dioxides have potential scientific and industrial applications. Jamo*et al.,* (2017b)and Noh (2017) have reported that the cheap RHA can be used successfully as fillers for polypropyleneand tetrafluoroethylene–ethylene copolymer, thus replacing the costly synthetic additive aerosol in the preparation of a range of polymer composites. RHA-polymer composites can bring about development of innovative hybrid organic–inorganic materials with specific properties. However, RHA is not only ash causing environmental problems. POFA is one the ashes that is causing similar problems

POFA is grayish in colour, the more the unburnt carbon dioxides it contains the darker it becomes (Noh *et al.,* 2014; Mohammad *et al.,* 2018; Martín-Márquez *et al.,*2008;Pérez *et al.,* 2012; Stathis *et al.,* 2004; Prasad *et al.,* 2001). The chemical composition of POF Aindicates presence of a high amount of silicon dioxides, which makes it good potential candidate for cement replacement. The utilization of palm oil fuel ash in high-strength concrete showed that POFA can be used as a pozzolanic material to produce high strength concrete. In addition, the utilization of POFA can improve concrete strength and permeability (Jamo *et al.,* 2017). Moreover, partial replacement of OPC (Ordinary Portland Cement) with POFA helps sulfate resistance and chloride resistance of concrete (Mohammad *et al.,* 2018). Compressive strength of cement paste containing pozzolan materials is contributed by hydration reaction, packing effect and pozzolanic reaction. Hydration reaction is the chemical reaction between Portland cement and water as pozzolanic compound and calcium hydroxide. However, this is not enough as RHA and POFA still remain a problem to problem to the environment. There is need for more efficient ways of utilizing these ashes. The use of RHA and POFA to substitute quartz in the production of porcelain is expected to add value to the MOR of the porcelain.

**METHODOLOGY**

The rice husk ash (RH) was thoroughly washed with distilled water in order to remove adhering soil and dust. After that it was dried in an oven(Carbolite PF 200) at 100°C for 24 hours. Then the dried husk was subjected to chemical treatment; 2M HCL, 5% solid at 25 °C before calcinations to increase silica content. After the leaching process, the treated husk was washed with distil water and then dried again. The treated husk was then subjected to calcinations at 700°C for six (6) hours in a Carbolite HTF - 1800 Furnace was used for sintering. The POFA was dried in the oven at 100 $^{o}$C for 24 hours. After that it was grinded in a ball mill to reduce the needed particle size to improve reactivity. The milling time was approximately 90 minutes at 200 rpmusing Ball Mill with model number: (Labkorea-YJB-10). Afterwards, the materials were subjected to a set of sieves less than 50 μm in order to remove the particles coarser than 50 μm. The untreated POFA was heated at a temperature of 600 $^{o}$C for 1.5 hours in an electric furnace to remove excess carbon.

Porcelain powder was grinded separately in a ball mill. The powder was sieved using sieve shaker and dried in an oven. The RHA and POFA was gradually incorporated into the body of porcelain powder from 5 %wt, to 25 %wt (Table 1).The composition was mixed using a ball mill for one and half hours. Themixingwascarriedoutfor 90 minutesat250rpmusing Ball Mill with model number: (Labkorea-YJB-10). The mixed powder was pressed into pellets at mould pressure of 91 MPa. The mixtures werepressed into rectangular bars and pellets bar using hydraulic hand press Carver Press Model (C-3851). All the pellets were sintered at a temperature of of 1100 $^{o}$C for the soaking times of 1 hour, 2 hourss and 3 hours, at a heating rate of 5 $^{o}$C per minute.Thesamplemorphologywasobservedusing fieldemission scanning electron microscope SEM(model:VPSEM SUPRA35VP). The SEM is machine was operated at operated at 2.00 kV. The magnification of 1000X is used to capture photo of the sample.

Themixingwascarriedoutfor 90 minutesat250rpmusing Ball Mill with model number: (Labkorea-YJB-10)

Table 1: The composition with the substitution of quartz by RHA and POFA (wt %)

| Sample name | Kaolin | Feldspar | Quartz | RHA | POFA |
|---|---|---|---|---|---|
| AP1 | 50 | 25 | 25 | 0 | 0 |
| AP2 | 50 | 25 | 20 | 3 | 2 |
| AP3 | 50 | 25 | 15 | 6 | 4 |
| AP4 | 50 | 25 | 10 | 9 | 6 |
| AP5 | 50 | 25 | 5 | 12 | 8 |
| AP6 | 50 | 25 | 0 | 15 | 10 |

The composition was mixed using a ball mill for one and half hours. The mixed powder was pressed into pellets at mould pressure of 91 MPa. All the pellets were sintered at a temperature of 1100 $^{o}$C for the soaking times of 1 h, 2 hs and 3 hs, at a heating rate of 5 $^{o}$C per minute. The bending strength was determined. The chemical composition of the POFA was studied using X-Ray Fluorescence (XRF) machine (XRF Bruker S4 Pioneer) which was operated at 60 kV while the amorphous structure of POFA was identified through XRDBruker D8 Advance X-diffractometer. It was operated at 40kVand 40A..

## RESULTS AND DISCUSSION

X-ray fluorescence (XRF)analysis was used for the chemical analysis. Hencethe amount of chemical elementscan be observed (Table 2). Thistableshowsthe resultof XRFanalysisof kaolin, feldspar, quartz, RHA and POFA.Itisevidentthat $SiO_2$ is the major composition in all the raw materials: kaolin, feldspar, quartz, RHA and POFA with 69.3 wt%, 72.7 wt%, 99.4 wt%, 93.7 wt% and 66.9 wt% and then followed by alumina with 24.3 wt%, 16.4 wt%, 0.2 wt%, 2.1 wt% and 6.4 wt% respectively. Hence, RHA and POFA are good candidates to be used as materials for substitution of quartz in the fabrication of porcelain.

Table 2: X-Ray Fluorescence (XRF) Analysis

| Sample | Content (wt%) | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Oxides | $SiO_2$ | $Al_2O_3$ | $K_2O$ | $P_2O_5$ | CaO | MgO | $CO_2$ | $SO_3$ | $FeO_3$ | $Na_2O$ | $TiO_2$ | LOI |
| RHA | 93.70 | 2.11 | 1.18 | 0.96 | 0.81 | 0.53 | 010 | 0.45 | - | - | - | 0.16 |
| POFA | 66.91 | 6.44 | 5.20 | 3.72 | 5.56 | 3.13 | - | 0.33 | 5.72 | 0.19 | - | 2.30 |
| Kaolin | 69.30 | 24.30 | 2.44 | - | - | - | 0.10 | - | 0.27 | - | 0.27 | 0.36 |
| Feldspar | 72.70 | 16.40 | 0.50 | 2.42 | - | - | - | 6.87 | 0.40 | 0.29 | - | 0.10 |
| Quartz | 99.40 | 0.22 | - | - | - | - | 0.10 | - | - | - | - | 0.28 |

The graph of porosity of the sintered samples is presented in Figure 1. The graph indicates that the increase in soaking time and replacement of RHA and POFA leads to decrease in porosity.The minimum porosity value was achieved with values of 3.8%, 2.8% and 3.4% which was achieved at a soaking time of 1 hour, 2 hours and 3 hours respectively. However, substituting 20 wt% of RHA and POFA in the porcelain body contribute a lot to the decrease in porosity. The decrease in porosity is attributed to increase in the amount of liquid formed due to the presence of $K_2O$ and CaO in RHA and POFA (Prasad *et al.,* 2001). Martín-Márquez *et al.*(2008) mentioned that another factor that could contribute to the decrease in porosity is the progressive incommunication of the pores that takes place as a result of increase in the glassy phase proportion. The porosity increases after reaching minimum values as the substitution was increased to 25 wt%. This is attributed to excess glassy phase formation.

In terms of soaking time the porosity decreases as the soaking time increases from 1 hour to 2 hours. This is as aresult of increase in mullite and cristobalite (Table 3) when the soaking is increased to 3 hours the porosity increases. The pressure of the trapped gases within the pores rises at the soaking time of 3 hours, opposing the progressive densification of the material. The minimum value of apparent porosity were recorded, bloating occurs causing an increase in apparent porosity.

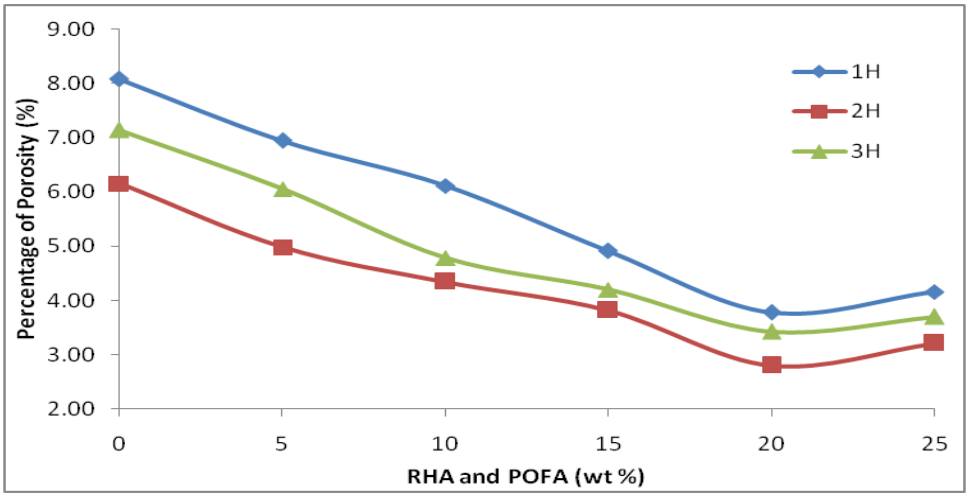


Figure 1: Graph of porosity versussoaking times the of of the samples with different amounts of RHA and POFA

Figure 2 displays differential changes in bulk density of the samples through the soaking times. The densities continued to increase, reached a maximum value of 2.33 g/cm$^3$ at the soaking time of 1 hour. While at the soaking time 2 hours amaximum value of 2.43 g/cm$^3$was recorded. Similarly, the value of 2.38 g/cm$^3$was attained at a the soaking time of 3 hours on 20 wt% of RHA and POFA. Replacement above 20 wt% of RHA and POFA causes the values of bulk density to decrease. The increase of soaking time is as a result of increase in liquid formation from RHA, POFA and feldspar which fills up the voids there by increasing the densification. Substitution above 20 wt% causes the porosity values to drop, due to excess glassy phase formation.

Considering soaking time the density increases as the soaking time increases from 1 hour to 2 hours. Pérez *et al.,* (2012) mentioned that it is as a result of increased mobility of glass when the $Na_2O$ is heated. The same scenario of glass mobility was also observed in case of $K_2O$ containing glazes during sintering(Youssef *et al.,* 2011; Das and Dana 2003; Ece *et al.,* 2002).In contrast to porosity result, the porosity decreases with increase in soaking time (Figure

5)because of the elimination of pores through liquid phase sintering. The gragh further indicates that vitrification took place at the soaking time of 2 hours. At a soaking time of 3 hours, the porosity tends to increases because of bloating.

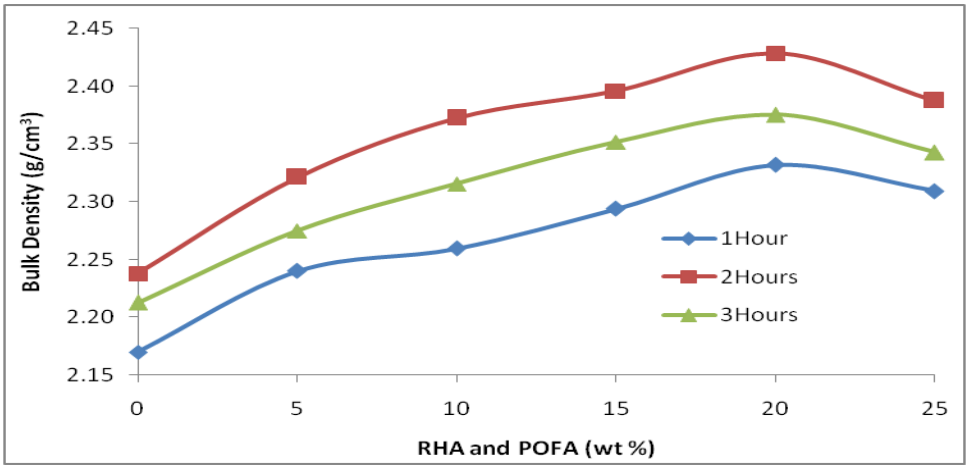


Figure 2: Graph of bulk density of the samples versus soaking time with different amounts of RHA and POFA

Figure 3 presents modulus of rapture as a function of the soaking time for the samples. In line with the porosity and bulk density results presebted in Figures 1 and 2 respectively. The maximum values of bending strength was achieved with approximate values of 30 MPa, 35 MPa and 32 MPa on 20 wt% of RHA and POFA for 1 hour, 2 hours and 3 hours respectively. Authors such as Stathis *et al.,* (2004) and Prasad *et al.*(2001) stated this behaviour could also be as a result of lower porosity. The higher modulus of rapture of samples could be attributed to the amount and type of phases in the bodies. Substitution above 25 wt% of quartz by RHA and POFA causes the values of bending strength to decrease, this is as a result of excess glassy formation.

In terms of soaking time the modulus of rapture increases as the soaking time increases from 1 hour to 2 hours. Increase in mullte and cristobalite contributed to the modulus of rapture as the soaking time increases.The increase in the values of modulus of rapture between 1 h and 2 hs is as a result of increase of mullite and cristoballite the results of which is presented in Figure 4 and Table 3. As the soaking time increases to 3 hours the modulus of rapture decreases due to bloating.At higher soaking time mullite and residual quartz crystals dispersed in a vitreous matrix are expected to be formed. Youssef *et al.,*(2011) recorded 6 hours maximum soaking time. In this study 2 hours maximum soaking time is recoded.The modulus of rapturehas strong relationship with microstructure (Figure 5), especially on defects such as cracks and pores. The progressive replacement of quartz by RHA and POFA causes an increase in the modulus of rapture.

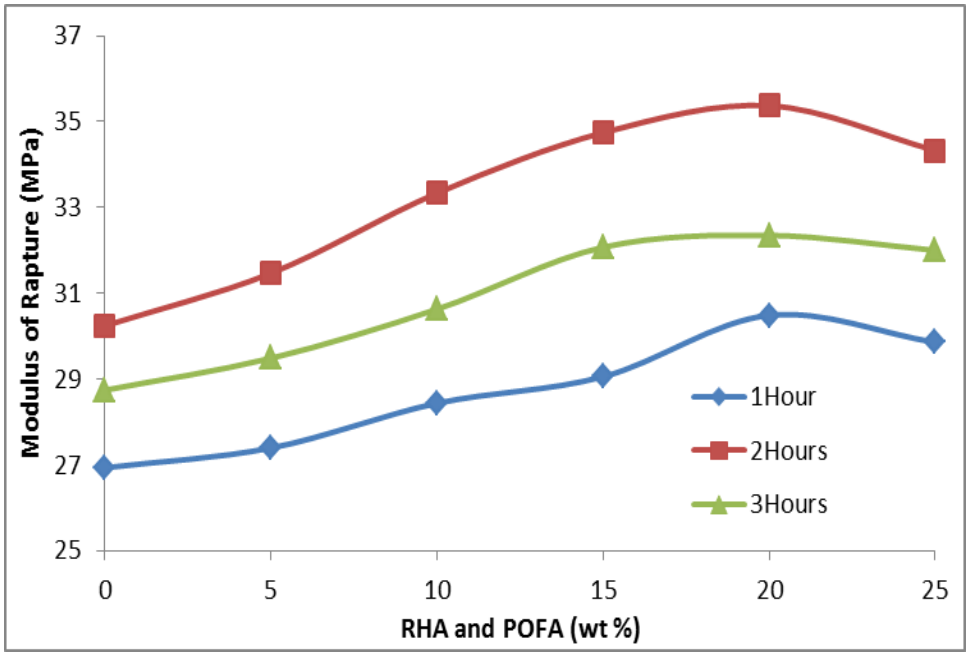


Figure 3: Graph of Modulus Rapture of the samples with different percentage of RHA and POFA

Figure 4 and Table 3 show the XRD analysis of the samples containing RHA and POFA. The major phases identified, are quartz (ICDD 046-1045), mullite (ICDD 074-4143) and cristobalite (ICDD 082-0512). The quartz decreases with increase in temperature. While the mullite and cristobalite increases between the soaking time of 1 hour and 2 hours. Between the soaking time 2 hours and 3 hours both mullite and cristobalite decreases.

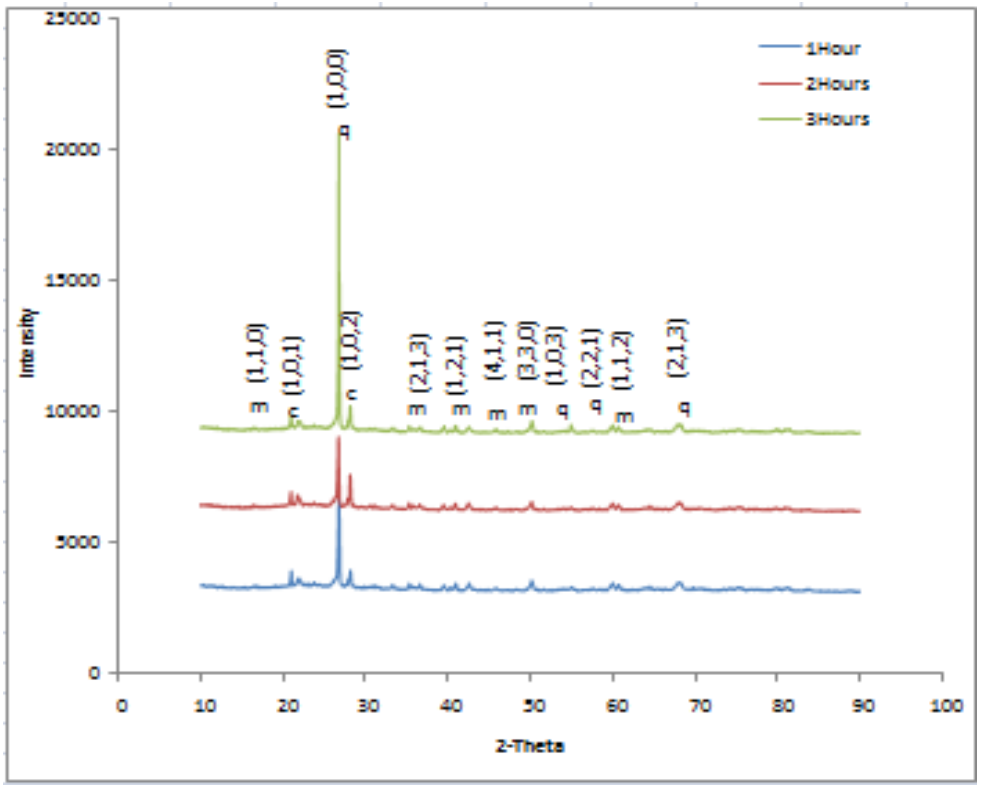


Figure 4: The XRD graphs of the samples containing 20 wt% of RHA and POFA

Table 3: XRD quantitative analysis of the samples containing RHA and POFA sintered at different soaking times

| Soaking Time (Hour) | Quartz (%) | Mullite (%) | Cristobalite (%) | Glassy Phase (%) |
|---|---|---|---|---|
| 1 | 60.7 | 12.8 | 9.7 | 16.8 |
| 2 | 57.0 | 25.9 | 12.7 | 4.4 |
| 3 | 43.2 | 24.0 | 6.5 | 26.3 |

Figure 5 shows how the microstructure of the samples containing RHA and POFA changes with increasing soaking time. The microstructure for the samples at a soaking time of 1 hour (Figure 5a) contains highly inter-connected pores and elongated grains. As the soaking time increases to 2 hours (Figure 5b) the grains becomes very small as a result of vitrification, majority of the pores are eliminated. At soaking time of 3 hours (Figure 5c) bloating takes place and pores expansion occurs.

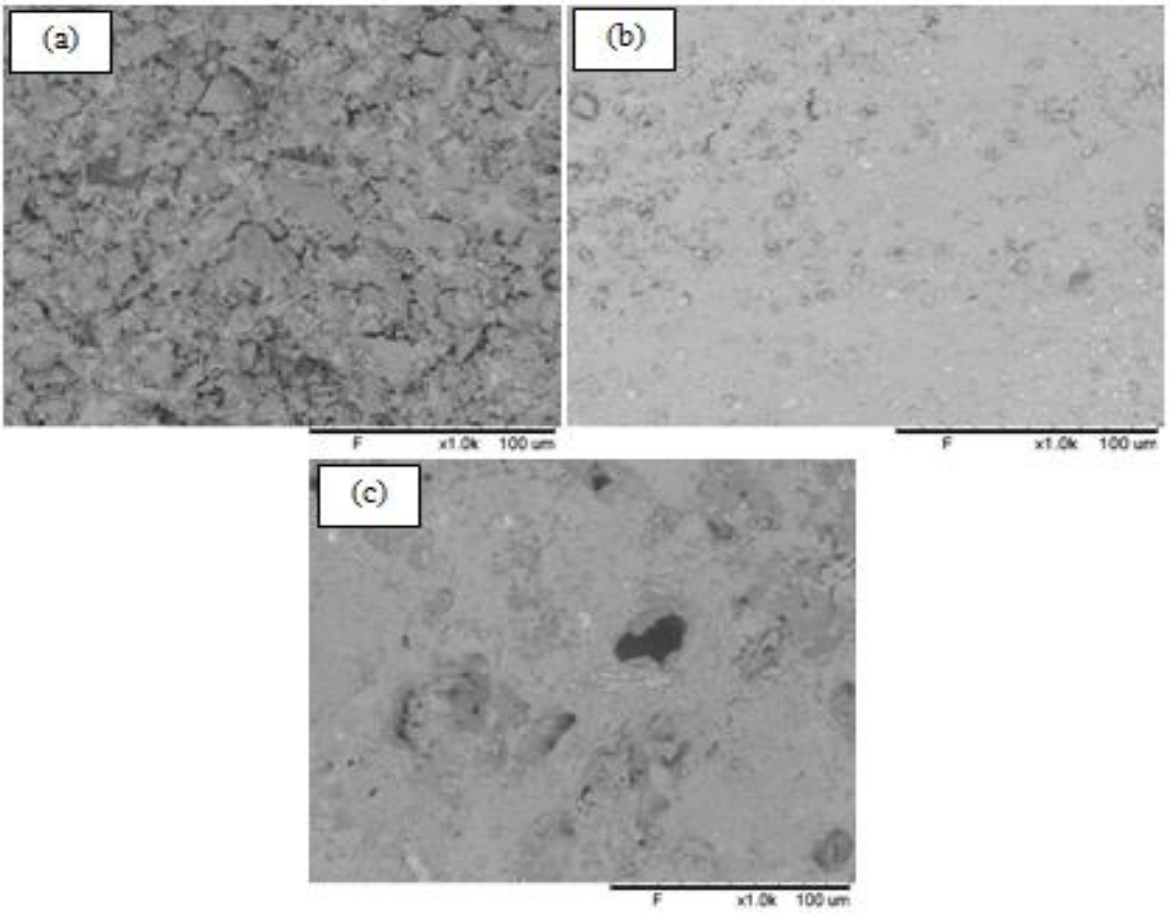


Figure 5: SEM of the samples containing RHA and POFA sintered at a soaking times (a) 1 hour (b) 2 hours (c) 3 hours. All micrograph were taken with 1000X magnification

## CONCLUSION

Modulus of rapture is affected positively by RHA and POFA, and also by soaking time. The Modulus of rapture of the samples was found to increase with increase in soaking time and also with the substitution of quartz by RHA and POFA. The highest Modulus of rapture for porcelain samples containing RHA and POFA occurred at a soaking time of 2 hours on 20 wt% substitution of quartz with RHA and POFA. The typical sequece enhanced solidification with increasing soaking time contributed to the increase inModulus of rapture. The soaking time of 2 hours exhibits highest bulk density and minimum porosty. The increase in the soaking time and the substantial decrease in porosity of the samples containing RHA and POFA, are as a result of glassy formation and densification of the individual grains during the vitrification process.

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