# Investigation on the Effects of Various Pore-Forming Agents on Bending Strength and Porosity of $Al_2O_3$ Ceramics

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**Abstract**

The current paper shows an economic and simple way which useful approach to produce porous ceramics (alumina) using three types of pore-forming agents (PFAs) utilizing a fugitive materials technique. Yeast material (natural active), ash of rice husk (commercial), and waste of graphite from used primary batteries have been used as (PFAs). Practical results revealed that with increasing the PFA ratio for yeast material (natural active), ash of rice husk, and waste of graphite the porosity increased from 30.2 to 63.8 %, 42.9 to 49.0%, and 37.3 to 61.1% respectively. Utilizing the three-point bending test, the evaluation of the mechanical behavior of porous ceramics (alumina) specimens was a function of the level of porosity. The characterization of mechanical behavior exhibit that the bending strength using the ash of rice husk increased at 50 wt% (92.38 ± 2.68 MPa) and 30 wt %( 93.03±4.07) due to the presence of the ceramic phases. While the bending strength of porous ceramics (alumina) using yeast material (natural active) and waste of graphite as PFAs decreased from 72.56±3.07 to 20.72±1.58 MPa and from 71.28±1.78 to 30.42±2.15 MPa respectively. Recommended application fields include the metal of molten, hot gas filters, and implantation processes.

***Keywords***: Porous ceramics alumina; waste of graphite, ash of rice husk; active yeast; porosity; bending strength.

## 1. Introduction

One of the important factors that have effects on the mechanical properties of porous ceramics such as bending strength, hardness, compressive strength, and modulus of elasticity, and others is the porosity forming. Therefore, the presence of porosity in the ceramic material leads to a decrease in mechanical properties due to the large deficits that formed inside the body of ceramic material [1]. Pore-forming agents (PFAs) play a significant role in mechanical properties such as the bending strength of porous ceramic materials due to porosity. Yang et al [2] reported the effects of PFAs and the morphology of pore on the mechanical properties of porous ceramics ($Si_3N_4$) utilizing organic whiskers to fabricate pores of rod-shaped and starch to form equiaxial pores using the technique of die press. It is noted that the bending strength decreases (from ≈ 450 MPa to ≈ 50M Pa) with increasing porosities (from 2.5% to 45%). Novaisn et al. [3] studied the effect of poly-propylene and poly methylmethacrylate (PMMA) as a pore former with particle sizes of 250 - 425μm on the mechanical properties of porous tile ceramics. The study showed that a significant decrease in the bending strength was obtained with an increasing level of porosity and pore-forming agent content. By using (PMMA) as a pore-forming agent at a particle size of 250 μm and ratios of 2.5-15wt %, the bending strength was 86.5-54.7MPa respectively. Ding et al. [4] reported the fabrication of mullite bonded porous SiC ceramic using graphite as a pore former. The study showed that with increasing porosity the load-bearing area decreases when the porous SiC is fractured, which leading to a decrease in bending strength from 22.5 MPa (38% porosity) to 3.5 MPa (56% Also, with high porosity, the porous ceramics fail suddenly at a stress that is far below the bulk material's strength. While a decrease in the mechanical properties such as the bending strength of 207.6–22.3 MPa, the compressive strength of 180–9.18 MPa, modulus of elasticity of 250–18 GPa and hardness of 149–18 HRD with increasing porosity have reported by Mohanta *et al* [5]. Porous ceramics (alumina) have used in the various engineering fields, such as thermal insulation [6], molten metal filtration, catalyst supports, hot-gas purifiers, and biomedical implants, due to their corrosive environments resistance [7, 8], chemical stability, high thermal resistance [9], high surface area, and high porosity [10]. One of the important and common ways to produce porous ceramic materials is a pore-forming (PF) process [11, 12]. Recent developments in the field of materials science have led to a renewed interest in the use of waste of materials. Generally, two types of waste of materials, industrial waste materials such as glass [13], waste of paper pulp, and waste of fly ash [14] , phosphoric acid [15] and agricultural waste materials for example waste of kenaf [16] and rice husk waste [17]. Porous ceramics have been successfully fabricated using these types of waste such as the waste of paper pulp [13], fly ash waste [14] rice husk waste [17], corn cob [18] lamellar graphite [19] [20], and yeast material [11, 21]. Due to the decreasing consumption of natural resources and the increasing cost of raw materials, many efforts have been made to use wastes and byproducts as a raw material for various manufacturing processes. It's an attempt to reduce environmental pollution and sustainable development. Up to now, due to increasing the cost of the raw materials, the researchers look for materials are available, low cost, and have a unique composition to produce the porous ceramics such as agricultural and industrial waste which have ceramic oxides ($SiO_2$, $Al_2O_3$, $ZrO_2$, and others). A few of researchers studied the effect of waste of graphite, yeast, and ash of rice husk on the bending strength of porous ceramics (alumina).This study investigated the effects of three types of PFAs ash of rice husk (commercial), yeast, and waste of graphite) on the level of porosity and bending strength of alumina ($Al_2O_3$) ceramics.

## 2. METHODOLOGY

### 2.1.1 Materials Preparation

a- The natural active yeast is considered as microorganism's materials were purchased from the company of Health Paradise Sdn. Bhd. (454299-w), Subang Jaya, Selangor, Malaysia.

b- The rice husk ash (RHA) was purchased from the factory of Maerotech Sdn Bhd. Nilai, Negeri, Sembilan, Malaysia and is considered as agricultural waste material. The ash of rice husk and yeast (natural active) were milled in a miller (electrical) model (RT-02A, 300 RPM) around 60 secs then sieved in as sieve (electrical model Retsch, As 200) to 250 μm as a particle size separately.

c-The waste of graphite was obtained from the used batteries (primary) it has been chosen from the same brand produced by Panasonic company and taken from recycling containers in Academia, Block-A condominium, South City Plaza, Serdang, Selangor, Malaysia. The bars of graphite were prepared with a solvent of acetone to get out the residue materials and oil after that dried for 24 hrs in the air and then dried in an electric oven at 100 $^{o}$C for 1h. The milling process was done using an electric miller (model (RT-02A, 300 RPM). In the final step, the milled graphite waste was sieved to a size of the particle of around 250 μm.

### 2.1.2 Mixing Process

A commercial alumina (corundum-$Al_2O_3$) powder with high purity (99.9%), 0.5 μm particle size, and density 3.94 g/cm$^3$ was used as a starting ceramic material which purchased from Chinese company. The binder used was a commercial sugar (sucrose). Depending on the solubility (in maximum) of sucrose in distilled water the sucrose (10-12%) was added to the alumina powder.

In this study, a concentration solution (60%) of sucrose (sugar) was adopted in use as the binder material [22]· The ceramic powder was mixed with the binder using a mortar of agate around (3-5 minutes). The yeast, ash of rice husk (commercial), and the waste of graphite powders were added as a weight ratio (10, 20, 30, 40, and 50 wt%) to the ceramic mixture (table 1). By using mortar (5-10 minutes), all materials were mixed after that milled using balls of alumina for 3hrs in a container of plastic to make them in the homogenous state in the weight ratio of balls (alumina) to the weight of the powder of 3:1.

Table 1.1: Weight contents percent of the porous alumina ceramics composites additives.

| PFAs*content (wt.%) (g) | Alumina ($Al_2O_3$) content (wt.%) (g) | Alumina + PFAs composite materials (wt.%) (g) |
|---|---|---|
| 0% | 100% | 0 (pore agent) +100 ($Al_2O_3$) |
| 10 | 90 | 10 (pore agent) + 90 ($Al_2O_3$) |
| 20 | 80 | 20 (pore agent) + 80 ($Al_2O_3$) |
| 30 | 70 | 30 (pore agent) + 70 ($Al_2O_3$) |
| 40 | 60 | 40 (pore agent) + 60 ($Al_2O_3$) |
| 50 | 50 | 50 (pore agent) + 50 ($Al_2O_3$) |

PFAs*(such as waste of graphite, yeast (natural active), and ash of rice husk.

### 2.1.3 Specimens Preparation for Porosity and Mechanical Test

To measure the porosity, a circular steel die (thickness= 5mm and diameter= 20 mm) utilized to produce porous ceramics (alumina). In a rectangular die of steel (width = 4mm, length = 40 mm, and thickness = 4 mm), dry batches were pressed uniaxially using the hydraulic press (Instron model) with 90 MPa applied pressure, the pressure applied has chosen according to the literature. After that, in an oven at 100 °C, green compacts were dried for 24 hrs. The burnout of organic materials of dried specimens was done in the ambient atmosphere using a programable furnace which electrically heated. The heating rate was 1.5 °C/min for each temperature increase. Depending on the thermogravimetric data of all PFAs [23], the waste of graphite and ash of rice husk specimens were burned at 200 °C, 300 °C, 500 °C, and 900 °C. While the yeast specimens were burned at 200 °C, 325 °C, 500 °C, and 900 °C to remove the sucrose, inorganic, and organic materials. Finally, all ceramics specimens were sintered in another furnace in soaking time of 2 hrs at 1600 °C with heating and cooling rates of 5 °C /min. (see Fig.1).

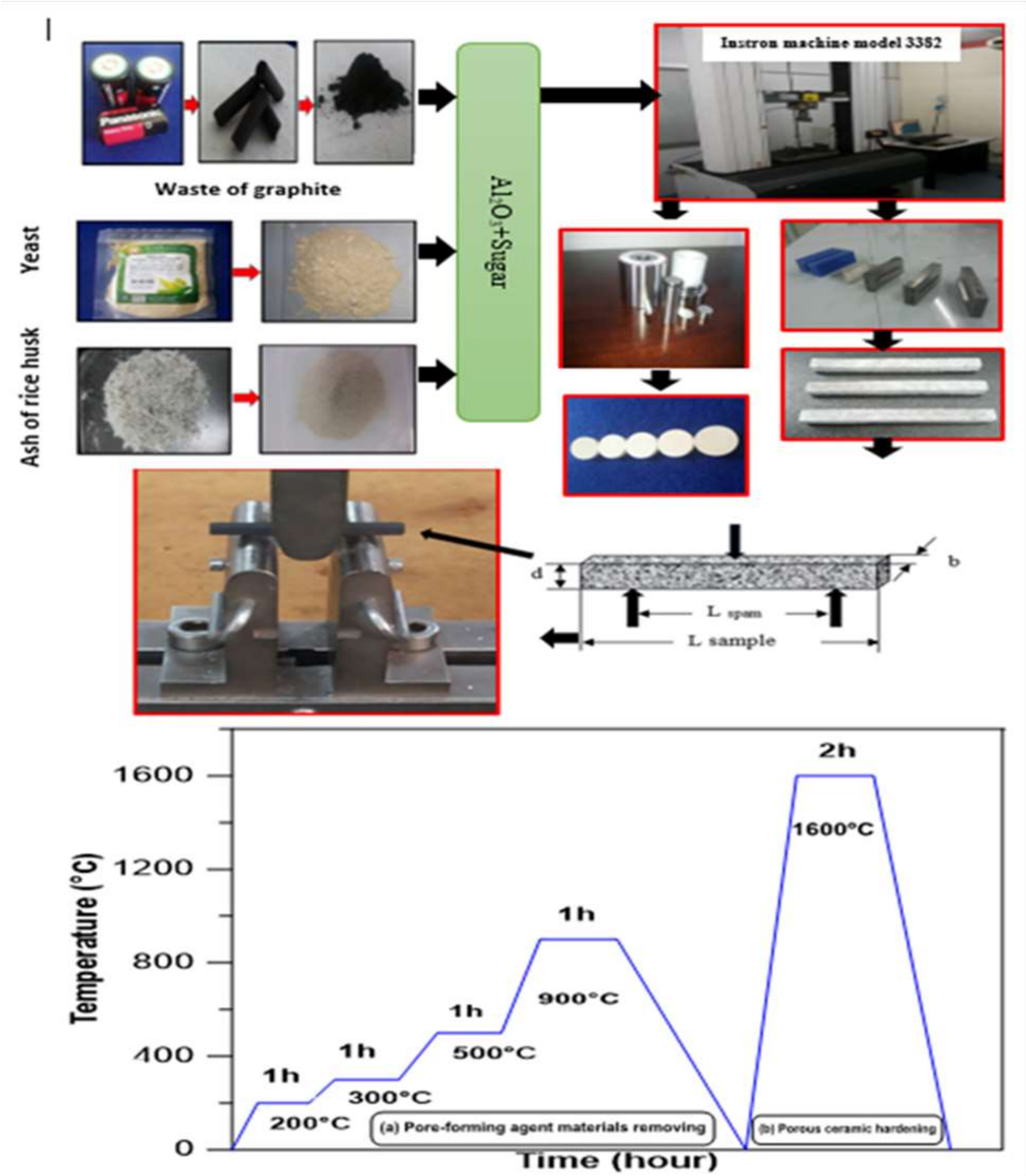



Figure 1. The experimental part of ceramic preparation

### 2.1.4 Characterization of Ceramics Specimens

To measure the porosity, a circular steel die (thickness= 5mm and diameter= 20 mm) was utilized to produce porous ceramics (alumina) as shown in figure 2.

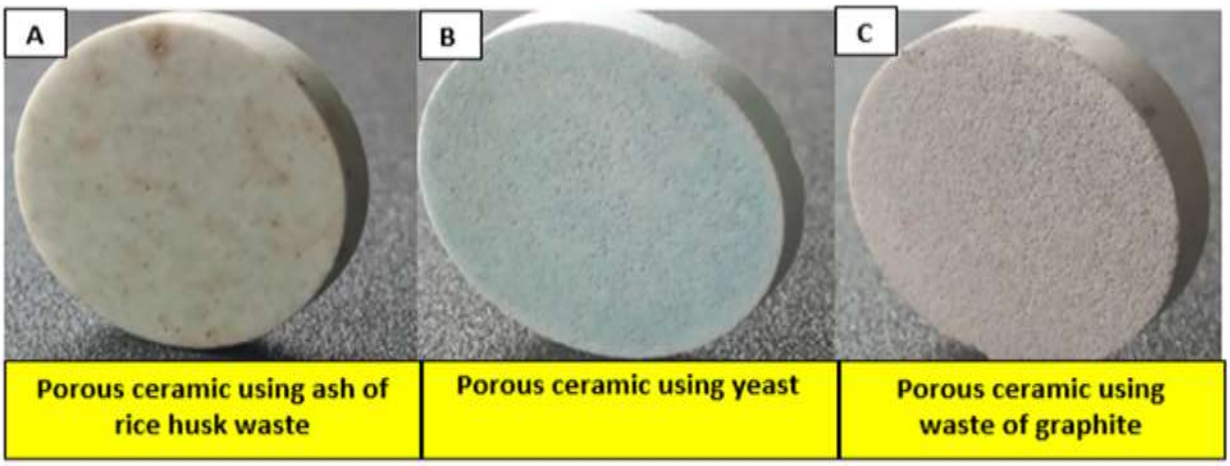



Figure 2. Porous ceramics specimens using three types of PFAs (A-ash of rice husk, B-yeast, and C-waste of graphite).

The total porosity and density of the burned ceramics specimens were measured using the technique of water immersion according to the principle of Archimedes (ASTM C20-00) utilizing the below equations [24].

$$P_{(overall)} = (1- \frac{\rho}{\text{theoretical density } (\rho)}) X100 \quad (1)$$

$$\rho = \frac{\text{MdryX } \rho\text{water}}{\text{Mwet}-\text{Msuspended}+\text{Mwire}} \quad (٢)$$

where $M_{dry}$ is the specimen dry mass [25].

$M_{suspended}$ is specimen mass which suspended in water (distilled).

$M_{wet}$ is specimen mass after water immersing.

$M_{wire}$ is the suspending system mass.

$P_{(overall)}$ is the total porosity volume fraction (vol.%) of the specimen [26-28]. The density of $Al_2O_3$ is ≈ 3.94 g/cm$^3$ that used as a reference determined utilizing Accupyc model II 1340.

The microscope of the field- emission scanning electron (FESEM) was used to examine the microstructure. The specimens used in the FESEM test were in dimensions (diameter = 20 mm and thickness 5 mm).

**2.1.5 Mechanical Characterization**

The bending test of three-point was conducted utilizing specimens of dimensions 40 mm ( length) x 4 mm (width) x 4 mm (thickness) and 20 mm as a length of spam using an Instron machine with a load cell of 5 KN depending on the standard of (ASTM C1161-02c )[29, 30]. The value of the bending strength has been measured according to the equation.

$$\sigma\ (MPa) = \frac{3*P*L}{2*b*d^2}$$

where σ is in MPa, P = the applied force (N), d = the thickness of the samples (mm), b = the width of the sample (mm), and L= outer (support) spam (mm) see Figure 3.

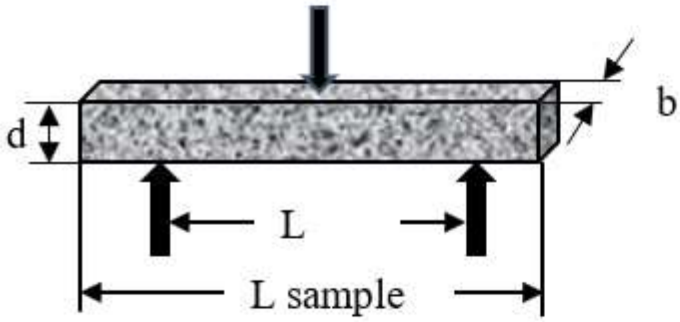



Figure 3. Bending strength specimen

## 3- Results and Discussion

### 3.1. Porosity, Microstructure, and Phase for Porous Alumina Ceramics

Figure 4 presents the bulk densities and total porosities variation with the content of PFA for the porous ceramics (alumina) specimens using yeast (natural active), ash of rice husk, and waste of graphite as PFA. The maximum porosities are 61.1%, 63.8%, and 49.0 % and the lower porosities are 37.3%, 30.2%, and 42.9% for the waste of graphite, yeast (natural active), and ash of rice husk respectively. Generally, increasing porosity results in decreased mechanical properties, however, the majority of applications involving porous ceramics require excellent mechanical properties [31], such as the filtration of hot gas, implantation processes, and molten metal. The experimental data of porosity and PFAs was used to determine the relationship between the total porosity and PFAs (linear regression equation). The data have been tested for linear analysis to obtain the following equations.

| Pore-forming agent (PFA) | Equation | R |
|---|---|---|
| Yeast material (natural active) | $P_{(yeast)} = 0.89 \times C_{(yeast)} + 19.81$ | 0.99 |
| Waste of graphite | $P_{(graphite\ waste)} = 0.75 \times C_{(graphite\ waste)} + 24.97$ | 0.95 |
| Ash of rice husk (commercial) | $P_{(RHA)} = 0.49 \times C_{(RHA)} + 30.33$ | 0.76 |

Where P is the overall porosity, C is PFA content, and R is a linearly dependent coefficient. Therefore, the total porosity and PFA content have a suitable relationship. A significant difference between R using yeast (natural active) and waste of graphite and R using the ash of rice husk in the linear regression equation. This may be due to incomplete removing of ash of rice husk and the porosity variation. As for the porous ceramics (alumina) densities using the ash of rice husk, waste of graphite, and yeast were in the ranges of (2.25 to 2.01 g/cm$^3$), (2.4 to1.40 g/cm$^{3)}$, and (2.75 to 1.42 g/cm$^3$) in respectively.

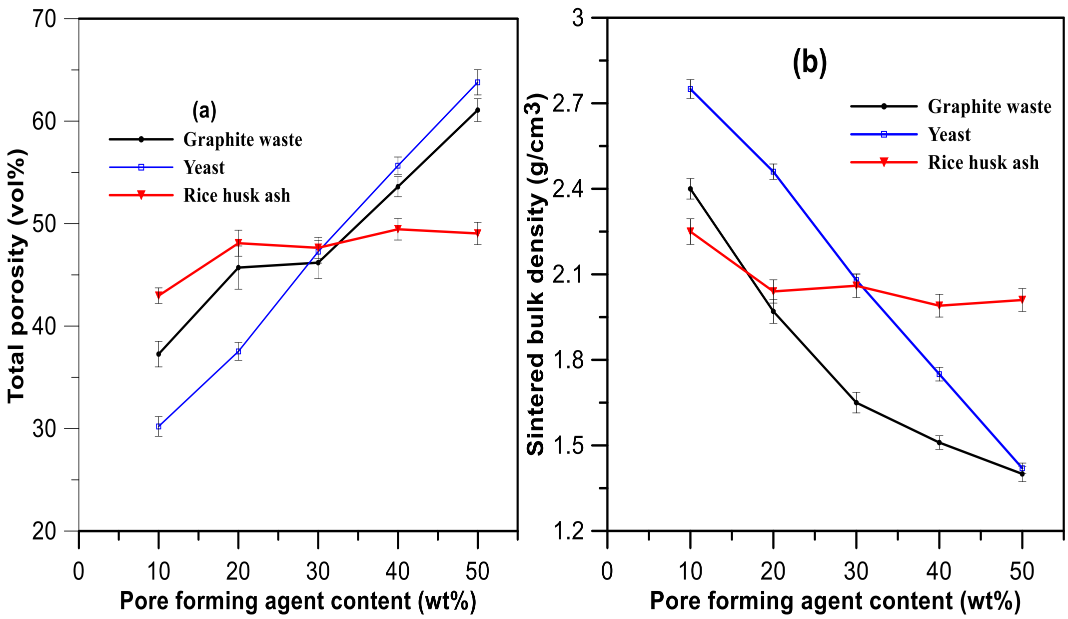



Figure 4. a) Increasing of porosity (total) with increasing PFA content. (b) decreasing sintered bulk densities with increasing PFA content. All specimens were sintered for 2hrs at 1600 C$^o$.

Figure 5 presents the FESEM image for PFA materials yeast (natural active), ash of rice husk, and waste of graphite), sugar (binder), and $Al_2O_3$ powder. The images of FESEM show different shapes of PFA particles. The longitudinal, irregular, and spheroidal shapes of PFA particles (see Figure 5 (a, b,c )) which lead to making pore various shapes in the porous ceramics (alumina) specimens after PFA materials burning out. Also, some researchers mentioned that there is a relationship between pore shape and mechanical properties. Zeng et al., (2007) reported that the porous ceramics present deterioration in mechanical properties with irregular pore shape due to the concentration of stress on the tip of the irregular shape compared with the porous ceramic with spherical pore shape.

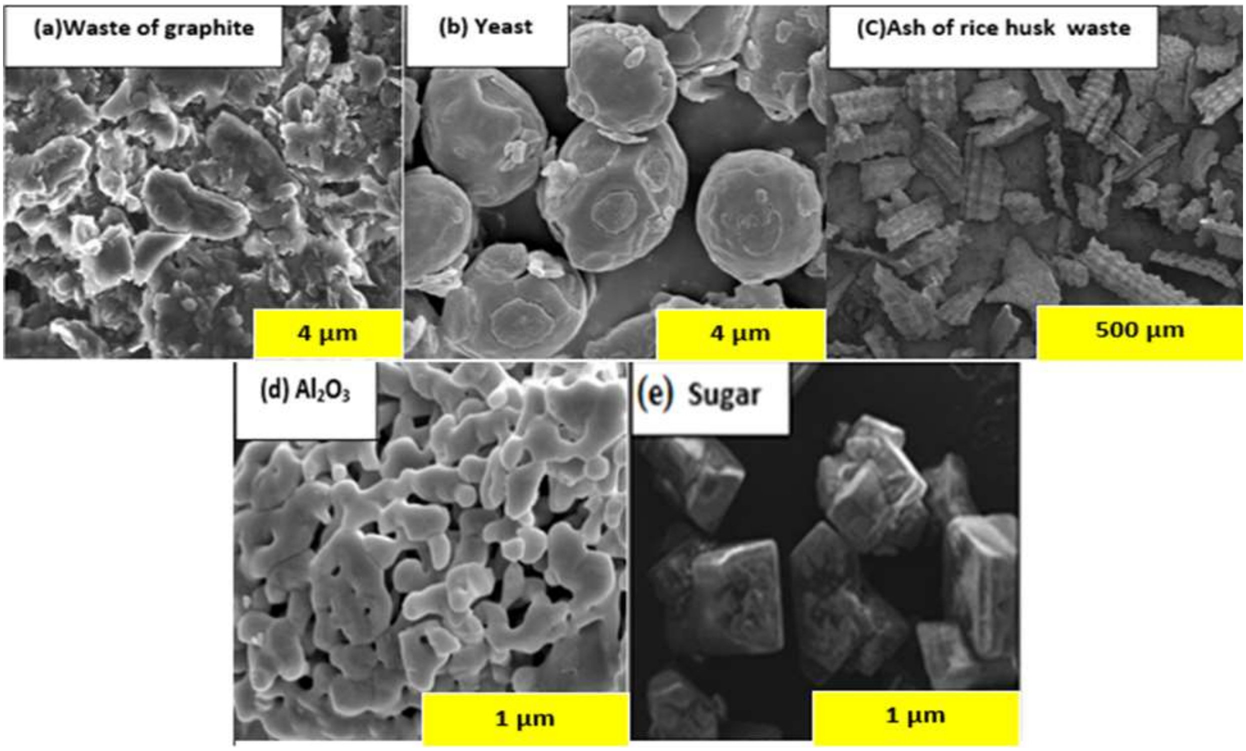



Figure 4. Images of FESEM for PFA materials (a)waste of graphite, (b)yeast (natural active), (c) and ash of rice husk, (d)$Al_2O_3$, and(e) binder (sugar)).

Figure 5 shows the FESEM images of porous ceramics specimens using three types of PFAs (waste of graphite, yeast, and ash of rice husk). The average pore sizes that have been investigated by FESEM between 97.35 µm and 118.97 µm using the waste of graphite and the average pore sizes recorded between 18.28 µm and 50.02 µm using the ash of rice husk. While the average pore sizes between 116.77 µm and 148.27 µm using yeast. The pore shape, distribution of pore size, and porosity are important factors utilized to determine the suitable application of ceramic materials. Moreover, often microporous can be appropriate and necessary to bone implant, closed pores are suitable for thermal insulation, open pores are suitable for transfer fluids such as metal filters, macroporous is suitable for filtering wastewater. The formation of different porosity forms in ceramic materials aims to develop the ceramic properties and lead to a wide ceramic application range. There are many applications of porous ceramic composite materials such as thermal insulation, sensors, catalyst support, impact-absorbing structures, fuel cell electrodes, membranes, and gas burners, etc. [32-35].

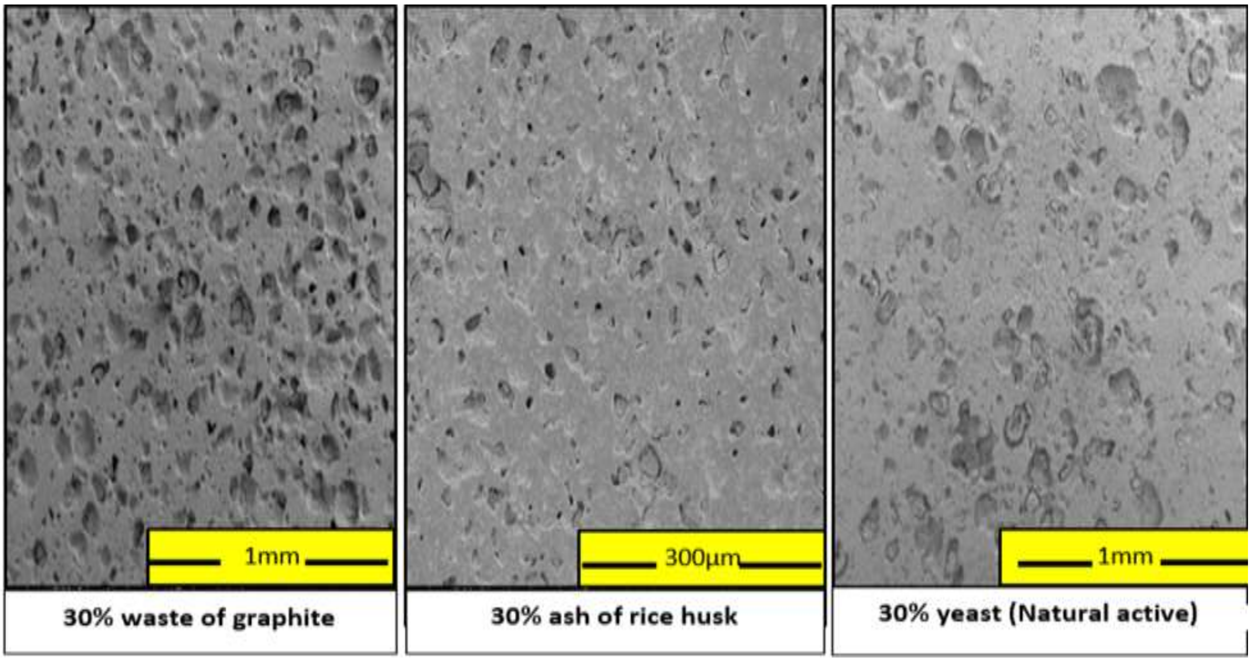



Figure 5. FESEM images of porous ceramics (alumina) using three types of PFAs (waste of graphite, ash of rice husk, and yeast).

Fig.6 presents that the XRD patterns of porous ceramics (alumina) specimens sintered at 1600C° for 2hrs with various content of ash of rice husk have various peaks, which refer to ceramic phases including mullite ($3Al_2O_3.2SiO_2$), corundum ($Al_2O_3$), and sillimanite ($Al_2SiO_5$). It was noted that with increasing contents of ash of rice husk and sintering at high temperature, the phases detected in the specimens of porous ceramics (alumina) with the ash of rice husk included corundum ($Al_2O_3$) at 10 wt% rice husk ash, cristobalite ($SiO_2$) and corundum ($Al_2O_3$) at 30 wt% rice husk ash, mullite ($3Al_2O_3.2SiO_2$) at 40 wt% rice husk ash while sillimanite ($Al_2SiO_5$) and mullite ($3Al_2O_3.2SiO_2$) existed at 50 wt% rice husk ash.

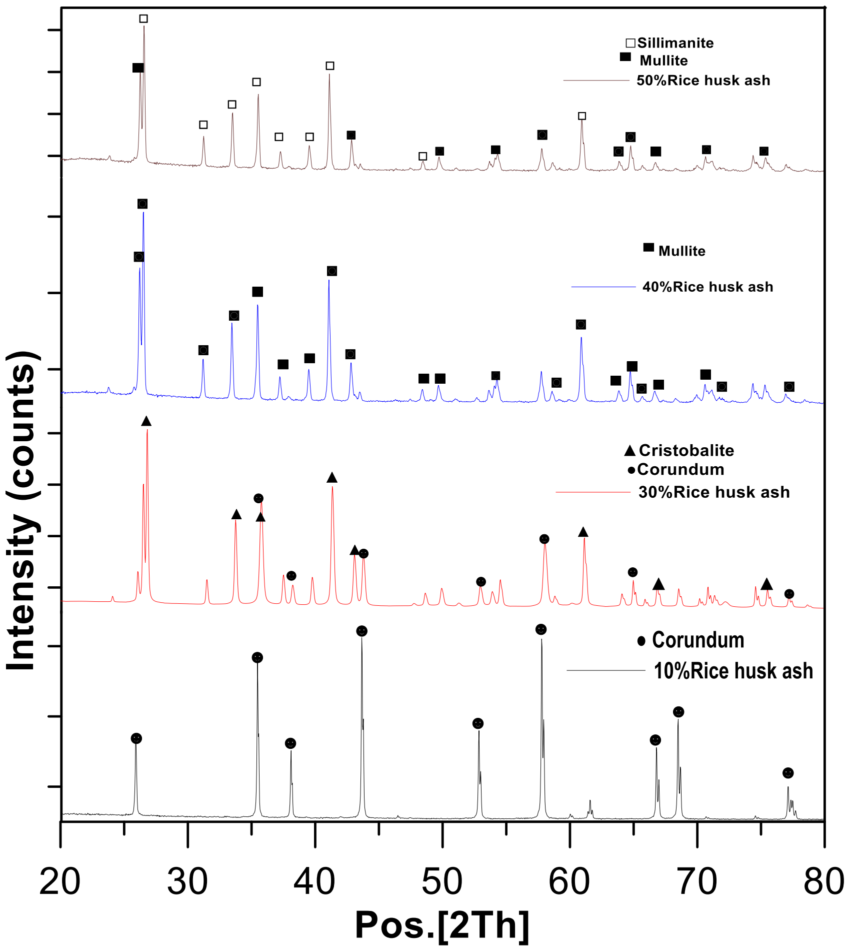
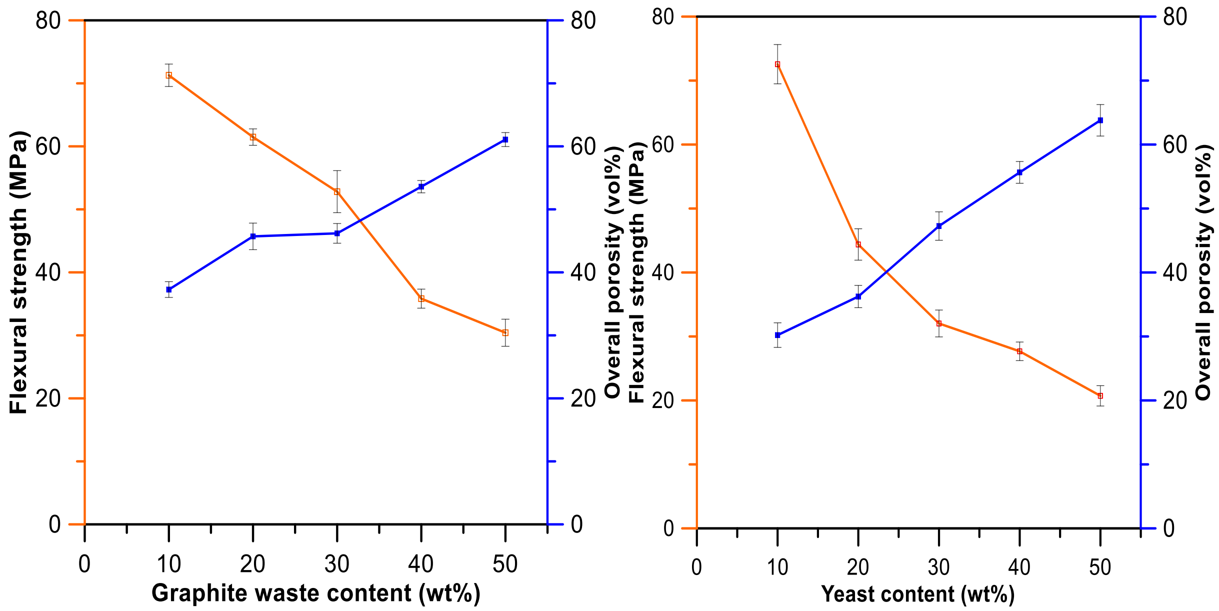



Figure 6. XRD for porous ceramics (alumina) specimens using ash of rice husk

### 3.2. Mechanical Properties

Fig.4 presents bending strength decreasing with the PFA content increasing. The bending strength decreases due to the increased level of porosity according to the formula of Rice ($\sigma = \sigma_o$ $exp(-bp)$) where $\sigma^o$ and $\sigma$ are the strengths of the nonporous and porous materials, $b$ is the constant which related to the characteristics of the pore, and $p$ is the porosity of the porous ceramics [36, 37]. Generally, the bending strength is affected by the necks of bonding between particles and porosity. Therefore, porous materials (ceramics) have a high bending strength due to its lower porosity and thicker necks [38]. It's a notable decrease in the bending strength of porous ceramics (alumina) with the increase in yeast content due to an increase in the level of porosity as shown in Fig. 4b. The flexural stress decreases from 72.57 MPa to 20.72 MPa in the level of porosity 30.21%

to 63.76% respectively. As for the bending strength of porous ceramics (alumina) using the waste of graphite, the bending strength decreases with an increase the waste of graphite content (see Fig. 4a). The values of bending strength between 71.28 MPa and 30.42 MPa with the level of porosity 61.08 % to 37.27% respectively. The bending strength decreasing was due to the increase in the level of porosity in the specimens of porous ceramics as a function of waste of graphite content increasing [39]. This is due to the reduction of the cross-sectional area when the pores are formed at the load applied and also pores worked as concentrators of stress [40] [41]. While the flexural stress of ceramics of porous using ash of rice husk as PFA. Fig. 4c presents the effect of ash of rice husk content on the bending strength of porous ceramics. The bending strength of porous ceramics near the peak (93.03 MPa) at 30 wt % ash of rice husk with the level of porosity of 47.64%. The flexural strength enhancement was due to ceramic phases formation such as cristobalite, corundum, and mullite [42, 43]. Thus, it can be concluded that all these phases of ceramics formed have an effect on the mechanical properties of porous ceramics[44] .



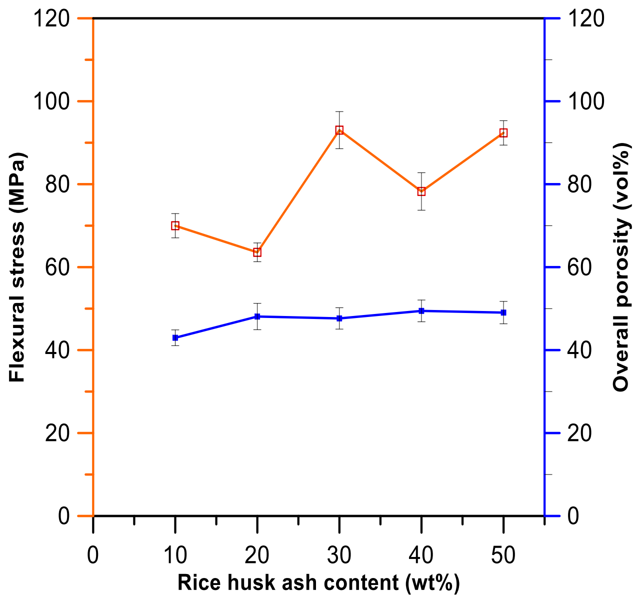



Fig. 4 Relationship between overall porosity and three types of PFAs (Yeast material (natural active), waste of graphite, and ash of rice husk) and flexural stress of porous ceramics (alumina) sintered for 2hrs at 1600 C°.

## 4. Conclusion

Porous alumina ($Al_2O_3$) ceramics with good porosity and high bending strength were prepared successfully using different PFAs. The results of this investigation show that the ash of rice husk material is used as PFA and as a strengthen factor at the same time due to the formation phases of ceramics (corundum, mullite, and cristobalite). The bending strength of porous ceramics near the peak (93.03 MPa) at 30 wt % ash of rice husk with the level of porosity of 47.64 %. The bending strength of porous ceramics (alumina) is highly related to the PFAs ratio of the porosity level which decreases when the level of porosity increase using a waste of graphite and yeast (natural active) as PFA. The flexural strength decreases from 72.57 MPa to 20.72 MPa in the level of porosity 30.21% to 63.76% respectively using yeast as PFA and the values of bending strength between 71.28 MPa and 30.42 MPa with the level of porosity 61.08 % to 37.27% respectively using the waste of graphite.  The future research of this study should have focused on the relationship between the PFAs content and fracture toughness porous ceramic (alumina).

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