

LIGHTWEIGHT AGGREGATE USING PALM OIL FUEL ASH AND FLY ASH

Abstract

Palm oil fuel ash and fly ash are industrial by products that are produced in coal-fired power station and palm oil industry. Due to the abundance of coal-fired power plant and palm oil industry in Malaysia, the increasing amount of palm oil fuel ash and fly ash generated each year is not well utilised. This study aims to give reuse value to palm oil fuel ash and fly ash by investigating the appropriate mix proportion to produce artificial lightweight aggregate that is suitable for construction applications.

The four acceptable mixes using on of fly ash to palm oil fuel ash with binder percentage are 4F6P15, 4F6P20, 4F6P25 and 5F5P20 mix [such as Mix-1 with 40% Fly ash and 60% POFA with 15% binder, Mix-2 with 40% Fly ash and 60% POFA with 20% binder, Mix-3 with 40% Fly ash and 60% POFA with 25% binder, and Mix-4 with 50% Fly ash and 50% POFA with 20% binder are determined. These mixes are able to produce fresh pellets of crushing strength greater than 0.5MPa. The fresh pellets were sintered with microwave oven in the presence of silicon carbide susceptor and zirconium insulation for 10 minutes, 15 minutes and 20 minutes. The properties of sintered aggregate namely crushing strength (show strength values), particle density (show values) and water adsorption (show values) was investigated.

Scanning electron microscopy analysis was conducted to study the microstructure of sintered aggregate (writing SEM findings). The sintered aggregate strength increases at 10 minutes sintering, decreases at 15 minutes and increases back sharply at 20 minutes sintering. The increase in particle density result in decrease in water absorption. Pls write significance of this finding

Keywords: lightweight aggregate, fly ash, palm oil fuel ash, microwave sintering

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I. INTRODUCTION

- 1. Background:** Cement, water, coarse aggregates, and fine aggregates are the main constituents of concrete. River sand and crushed stones are usually used as aggregates in normal-weight concrete mixing, with a bulk density of aggregate ranging from about 1200 – 1750kg/m³, these aggregates produce normal-weight concrete of density ranging from 2200 – 2400kg/m³ [1] and cube compressive strength of about 15 – 100MPa and above [2]. Lightweight aggregate concrete has similar constituents as normal-weight concrete, the main difference is that lightweight aggregate (LWA) with maximum loose bulk density of 880kg/m³ and 1120kg/m³ for coarse and fine LWA respectively are used in lightweight aggregate concrete mix [3, 4]. The resulted mix is lightweight concrete with density of range about 300 – 2000kg/m³, and cube compressive strength of about 1 – 60MPa and above [5].

Higher dead load resulting from the self-weight of normal-weight concrete members will require structural members to be designed with larger dimension to withstand the dead load [6]. With the correct mix proportion and aggregate selection, the performance of lightweight aggregates concrete similar to normal-weight concrete can be produced. Usage of lightweight aggregate concrete can reduce the dead load imposed by structural members, resulting in reduction in size of structural members, larger usable space, lighter structure, and better cost saving [7, 8, 9, 10].

Lightweight aggregate concrete is commonly produced by using both lightweight coarse and fine aggregate, or a combination of normal-weight fine aggregate with lightweight coarse aggregate [11]. Apart from naturally occurring lightweight aggregates such as pumice, scoria and tuff, structural-grade lightweight aggregates are also produced artificially in the industry by using expanded shale, clay, slates, blast-furnace slags, fly ash (FA) etc. Artificial lightweight aggregates are commonly produced by using either pyro processing methods, autoclaving process, or cold bonding [12].

In Malaysia, there are abundant of industrial solid wastes such as fly ash and palm oil fuel ash (POFA) from power plants and palm oil mill industry. It is estimated that 4.5 million tonnes of fly ash and 2.8 million tonnes of palm oil fuel ash were generated in the year of 2015 and 2014 respectively [13]. Reports have shown that in year 2013, only 42.6% of the fly ash produced was reused while 57.2% was disposed in landfills in Malaysia [14]. The disposal method of palm oil fuel ash is similar to fly ash. It is estimated that the amount of industrial solid waste will increase over the years due to higher production rate and demands. As both palm oil fuel ash and fly ash are usually disposed in a landfill, it is beneficial to incorporate fly ash and oil palm fuel ash in production of artificial lightweight aggregate give reuse value to fly ash and oil palm fuel ash. By utilising industrial solid wastes in production of artificial lightweight aggregate, depletion rate of natural resources will be reduced due to less demand from construction industry, in addition to that, pollution problems arise from high volume disposal of industrial solid wastes to landfill will also be reduced.

In order to fully reap the benefits of utilising industrial solid wastes in production of artificial lightweight aggregate (ALWA), consideration on the constraints imposed by various industrial processes shall be taken into account when developing or investigating

the properties of ALWA and research aim should focus on producing ALWA that is able to withstand the imposed load and impacts from various automated industrial process. Mass production cannot be achieved if the ALWA are unable to survive during the transportation via conveyor belt to other processing stage. Therefore, sufficient strength must be attained during early stage to allow for industrialised, mass production of ALWA to achieve a sustainable growth by increasing the utilisation rate of industrial solid wastes. In an effort to ease application for mass production using automated system, one of the objectives of this research is to identify the optimum mix proportion of palm oil fuel ash, fly ash and binder to produce fresh pellets that have crushing strength of 0.5 MPa and above after 24 hours of air-drying post pelletization.

In short, the aims of this paper are to produce artificial lightweight aggregate that has adequate compressive strength and is able to replace usage of natural aggregate in various construction activities. The impact of this research is giving potential reuse value to palm oil fuel ash and fly ash as lightweight aggregate to reduce depletion of natural aggregate and control the environmental pollution due to disposal of these solid wastes. In addition to that, the performance of lightweight aggregate produced by using hybrid microwave sintering techniques have been investigated in an effort to reduce the energy consumption of lightweight aggregate production whilst maintaining the high quality of sintered lightweight aggregate.

- 2. Hardening Method:** Cold bonding method involves formations of matrix bonding when cured that improve the mechanical properties of aggregate. Cold bonding method is achieved by pressure or non-pressure agglomeration method along with the aid of a binder that contain sources of calcium hydroxide or alumino-silicate gel to react with the pozzolanic material to form the matrix bonding required [15]. Ordinary Portland Cement and alkaline activator are usually used as binder for cold bonding process. The curing process of cold bonding method involves curing at room temperature or in steam chamber for 28 days, the ease of curing substantially reduces the cost and environmental impact resulting from mass production of lightweight aggregate using cold bonding method. However, the limitation of this method is the long curing time required which limits the production capacity of industry and its ability to fulfil sudden surge in demand in a short period of time.

Autoclaving method uses pressurized steam and elevated temperature to aid the strength gain of aggregate, this method allows lightweight aggregate to be produced with fewer binding materials and involves shorter curing period [16]. The process involves curing of fresh pellets in an autoclave machine at required pressure and temperature to harden and increase the mechanical properties of lightweight aggregate. Latest research shows that autoclaving technology allows lightweight aggregate to be produced from a variety of industrial solid wastes with a curing time as short as 4 hours [17]. It has been investigated that the mechanical properties of autoclaved fly ash aggregate are similar to the fly ash aggregate produced by cold bonding method [18]. The downside of autoclaving method is the high initial capital investment in equipment acquisition, high power consumption and large space requirement to house the manufacturing facilities [19].

Pyro processing method is commonly known as sintering method, sintering process is a process that involve heat treatment of raw materials with aims to improve the mechanical properties of materials, it involves heating of raw materials up to the eutectic point to induce bridging of material particles into a solid coherent mass [20]. Sintering method usually uses binder such as bentonite or sodium silicate to aid fusion of the materials before and during sintering. According to [21], the mechanism of sodium silicate acting as a binder for production of LWA involves several chemical reactions; the reaction of sodium silicate with air produces sodium carbonate, which is soon decomposed in heat to form sodium oxide, the presence of alkali metal oxide is the main factor in lowering the eutectic temperature of the LWA [22]. Lowering the eutectic temperature of LWA will result in energy saving as the vitrification of LWA surface and fusion of materials are able to occur at lower temperature, this has a direct effect in the energy required to produce the LWA of desired quality [22]. The lightweight aggregate produced using sintering method have lower water adsorption compared to other hardening method [18], however the high energy usage associated with sintering method due to its high temperature requirement which is usually around 900°C to 1300°C remains to be a major concern to the manufacturing industry.

- 3. Sintering Method:** Sintering process occurs below the melting point of raw materials and usually can be achieved by several methods. Among the most commonly adapted sintering method are conventional sintering, microwave sintering and hybrid sintering technique. Conventional sintering involves usage of muffle furnace or any appliances capable of achieving high enough temperature for sintering. This sintering method usually involves higher energy usage due to the nature of heat transfer, where heat is transferred from the outermost part of the material to the core through the establishment of temperature gradient. The heating rate of conventional sintering is usually much slower than other sintering method due to the mechanism of heating that involve conduction, convection and radiation, many researchers have reported that the rate of heating for observed for convention sintering method is in the range of 5°C – 8°C per minute [23]. At such, conventional sintering method proves to be less energy efficient compared to microwave sintering and hybrid sintering method.

Microwave sintering method involves usage of microwave oven to achieve heating of the raw materials. This sintering method is less energy intensive compared to conventional sintering. The mechanism of heating involves utilization of microwave energy with frequency in the range of 300 MHz to 300GHz, the materials absorb microwave energy volumetrically and produces heat internally. Due to the mechanism of microwave heating, the heating rate is usually much higher than that of using a furnace. The heating rate reported for microwave sintering ranges from 55°C – 65°C per minute [23, 24]. The faster rate of heating is effective to reduce the energy consumption and simplify processing time.

Hybrid sintering method involves usage of microwave oven and microwave susceptor to achieve heating. This sintering method has similar energy usage to microwave sintering method. The mechanism of heating involves a combination of that in conventional sintering and microwave sintering, utilization of microwave energy and heating through conduction, convection and radiation is able to achieve bi-directional heating of the materials. This is done by the usage of a microwave susceptor that function

as a material to absorb microwave energy and emit infrared energy due to induction heating. Hybrid sintering method has several advantages to the properties of sintered ALWA as discussed below. Literatures have shown that aggregate produced by using hybrid sintering process have shown superior properties compared to that produce by using conventional sintering. Microstructure refinement is observed for ALWA produced using hybrid sintering method compared to conventional sintering method, resulting in increased crushing resistance and decreased water adsorption of the sintered aggregate [23]. Reference [25] have mentioned that microwave sintering is able to lower the sintering temperature of fly ash due to the localised heating of carbon within the fly ash, effectively achieving energy saving and microstructure refinement.

In short, hybrid sintering method has been proven to improve the mechanical properties of sintered ALWA compared to conventional sintering. In addition to that, it also consumes significantly less energy due to shorter sintering time compared to conventional sintering. Therefore, this paper aims to produce ALWA from fly ash and palm oil fuel ash using hybrid sintering method, where silicon carbide powder is used as microwave susceptor to achieve bi-directional heating of aggregate.

- 4. Chemical composition of raw materials:** Formulations of LWA are usually designed empirically; it is not uncommon to see that formulation are done according to the chemical composition of the raw materials. Chemical composition of raw materials is important to identify the sinterability and performance of sintering technique in hardening of fresh pellets. Reference [26] has investigated the relationship between chemical properties and bloating behaviour of clays; the findings indicating the suitable chemical composition for bloating of clays to occur has been identify as shown in Table 1 below. Bloating is an important phenomenon that affect the quality of LWA from the aspects of its mechanical strength, particle density, bulk density and water absorption [27]. Bloating involves liberation of gases in the presence of sufficiently viscous liquid phase, leading to entrapment of gases that forms the porous microstructure of LWA; the degree of expansion has a direct effect on the physical and technological properties of LWA, it can be understood as the key point of ensuring the quality of LWA.

According to [26], the chemical components desirable for bloating to occurs are silicon dioxide, aluminium oxide and other chemical components classified as flux. Silicon dioxide and aluminium oxide is responsible for formation of mass with proper viscosity to trap the gases liberated at bloating temperature. Presence of flux such as ferric oxides, calcium oxides, magnesium oxide, potassium oxide and sodium oxide will aid in lowering the melting point of raw materials, allowing the materials to undergo bloating at a lower temperature.

Comparing the recommended chemical composition for production of LWA to the chemical composition of fly ash and palm oil fuel ash shown in Table 2 and 3, it can be clearly identified that the silicon dioxide, aluminium oxide and flux contents are within the range of recommended values. Although it has been observed that the aluminium oxide content for fly ash meets the recommended maximum value of 25%, the addition of POFA to FA is expected to decrease the aluminium oxide contents to the range recommended. In short, literatures reviewed in this subsection has supported that the raw

materials used in this research have adequate chemical composition in production of LWA. It is feasible to produce LWA by using a mixture of both FA and POFA.

Table 1: Recommended Chemical Composition Range for LWA Production

According to Riley's Chemical Composition	Recommended Required Percentage (%)
Silicon dioxide	48 – 70
Aluminium oxide	8 – 25
Iron (III) oxide + Iron (II) oxide	3 – 12
Calcium oxide + Magnesium oxide	1 – 12
Potassium oxide + Sodium oxide	0.5 – 7

Table 2: Chemical Composition of Fly Ash

Chemical Composition	Percentage present in fly ash
Silicon dioxide	55.00
Calcium oxide	4.20
Alumina	24.50
Ferric oxide	7.10
Sodium oxide	0.30
Potassium oxide	3.88
Manganese oxide	1.95
LOI	0.32

Table 3: Chemical Composition of Palm Oil Fuel Ash

Chemical Composition	Percentage present in POFA
Silicon dioxide	57.8
Calcium oxide	3.60
Alumina	2.30
Ferric oxide	9.60
Sodium oxide	0.56
Potassium oxide	3.50
Manganese oxide	1.40
LOI	20.70

Note. Reference: Brabha H. N (2016). Utilisation of agricultural and industrial waste in self-compacting concrete, unpublished PhD Thesis, Universiti Malaysia Sabah, Sabah, Malaysia

5. Past research: Various past research has shown the feasibility of using fly ash and palm oil fuel ash to produce lightweight aggregate [28, 29, 30, 31, 32, 33, 34]. It is found out that the smaller the size of pellets, the higher the crushing strength [35]. The optimum disc pelletization angle is dependent on the critical revolution per minute, diameter of disc and angle of inclination [36], the optimum pelletization time and water content for maximum strength gain is 17 minutes and 28% by weight respectively [37]. As for the selection of binder material, study shows that sodium silicate binder is capable of lowering the eutectic point of raw material, allow it to be sintered at lower sintering temperature [22]. Based on research in an effort to classify the applications of lightweight aggregate, the potential field of application of lightweight aggregate based on water absorption and crushing strength was produced with reference to EN-13055-1 and basic properties of commercial lightweight aggregates [38]. The reproduced table is presented in Table 4 below. The potential applications of the microwave sintered lightweight aggregate were explored based on the particle density, 24 hours' water absorption percentage and crushing strength. The potential usage of sintered aggregate was presented in the result and discussion part of this paper.

Table 4: Potential Application of Lightweight Aggregate

Particle Density(g/cm ³)	24 hours Water Absorption (%)	Crushing Strength (MPa)	Application
Less than 2.00	0 – 20	Greater than 5.00	High-strength concrete
	0 – 20	Greater than 2.30	Structural lightweight concrete
	0 – 34	Greater than 1.80	Non-structural lightweight concrete and mortar
	10 – 38	Greater than 1.80	Geotechnical applications
	10 – 38	Greater than 1.00	Gardening, landscaping, thermal and acoustic insulation

Note. From “Developing and Classifying Lightweight Aggregates from Sewage Sludge and Rice Husk Ash,” by M. Souza, M. Anjos, M. Sa, N. Souza, 2020, Case Studies in Construction Materials, 12, (<https://www.sciencedirect.com/science/article/pii/S2214509520300127>). CC-BY-NC.

II. MATERIALS AND METHODS

- 1. Raw materials used:** The materials used in this research are fly ash (FA), palm oil fuel ash (POFA) and sodium silicate as shown in Figure 1 and Figure 2 respectively. The main materials that used in production of LWA were FA and POFA while sodium silicate served as a binder and sintering additive for the lightweight aggregates. In addition to that, silicon carbide powder with 37µm particle size is used as a microwave susceptor to achieve hybrid sintering effect.



Figure 1: Fly ash.



Figure 2: Palm oil fuel ash.

- 2. Preparation of Raw Materials and Pellets:** The raw POFA were wet sieved with a 1.18mm BS sieve to remove excessively large particles and other impurities, the wet sieved POFA were oven dried at 110 ± 5 °C for 24 ± 1 hours with fan-circulated ELE International 425 litres drying oven. After drying, it was sieved using a 212µm BS sieve, POFA passing 212µm BS sieve was kept in air-tight container for storage purpose. During preparation for pelletization, POFA and FA were weighted separately in accordance with Table 5 and was not mixed together during the weighting process. POFA and sodium silicate binder were mixed homogeneously before pelletizing with a disc pelletizer of diameter 570 mm, operated at a fixed speed of 55 rpm and inclined at an angle of 74°. The design of pelletizing disc was based on literatures that studied the critical revolution per minute with respect to the disc diameter, speed and angle of inclination [36]. After POFA and sodium silicate mixture were successfully pelletized, fly

ash was added to the surface of the pellets during the pelletization process to introduce a less water permeable layer to the fresh pellets. The fresh pellets shown in Figure 3 were air dried at room temperature for 24 ± 1 hours before subjected to crushing test for fresh pellets. After identifying the mix proportion that was capable of producing fresh pellets of strength greater than 0.5 MPa, mass production of fresh pellets was done according to the identified mix proportion for microwave sintering.

- 3. Sintering procedure:** Sintering of fresh pellets involves several materials and apparatus; the materials used were (1) fresh pellets having crushing strength greater than 0.5 MPa, (2) Laboratory grade silicon carbide powder with $37\mu\text{m}$ particle size while the apparatus used were (1) Microwave oven with maximum output of 1000W, (2) Small size ceramic crucible with lid, (3) Medium size ceramic crucible and (4) Zirconium Insulation. Figure 4 shows the schematic diagram of microwave sintering setup used in this paper. The microwave sintering chamber consists of 3 components, (1) an inner ceramic crucible equipped with lid acting as a sample sintering chamber, (2) an outer ceramic crucible functions to hold the silicon carbide powder susceptor, and (3) an insulation box made out of zirconium insulation. After identification of mix proportions that were able to produce fresh pellets with crushing strength of at least 0.5 MPa, mass production of the fresh pellets with appropriate mix proportion was done. Sintering of the pellets were conducted at the sample chamber shown in Figure 4 The sintering time for fresh pellets were 10, 15 and 20 minutes.



Figure 3: Fresh pellets.

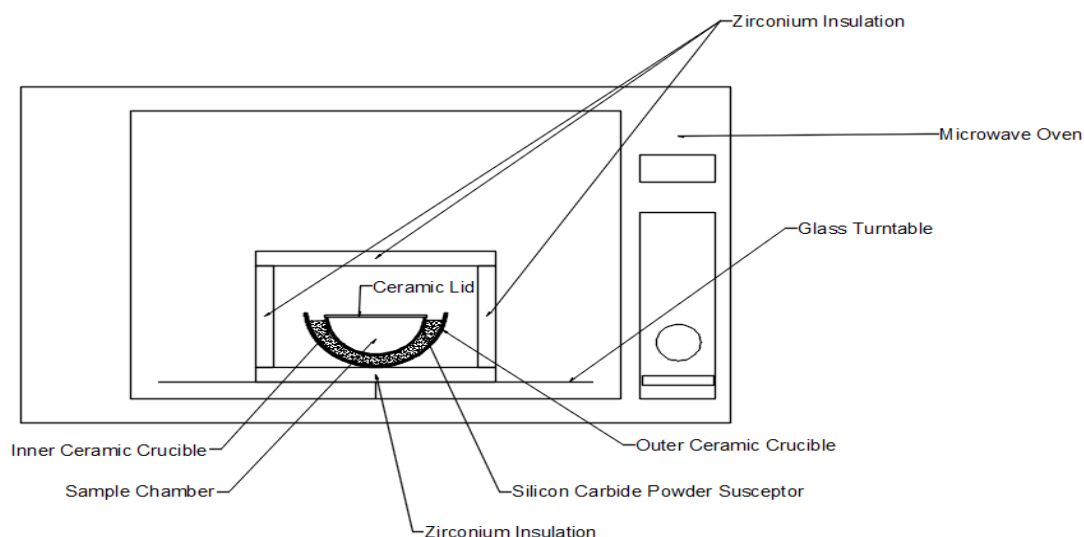


Figure 4: Schematic diagram of sintering setup for microwave sintering.

Table 5: Mix Proportion of POFA, FA and Sodium Silicate Binder

Mix ID	FA (%)	POFA (%)	Sodium Silicate (% weight)
5A46	40	60	5
5A55	50	50	
5A64	60	40	
10A46	40	60	10
10A55	50	50	
10A64	60	40	
15A46	40	60	15
15A55	50	50	
15A64	60	40	
20A46	40	60	20
20A55	50	50	
20A64	60	40	
25A46	40	60	25
25A55	50	50	
25A64	60	40	

- 4. Testing Method:** Crushing strength test was done by using ELE Digital Tritest 50 load frame equipped with proving ring of capacity 2 kN and 50 kN. The apparatus is shown in figure 5 below, 2 load ring with maximum capacity of 2 kN and 50 kN respectively were used in monitoring of the loading force throughout the test. The deformation rate used in crushing strength test was fixed to 0.01mm/s. The green pellets were tested using 2kN load ring while sintered pellets were tested using 50kN load ring. The pellets were placed between two parallel plates and crushed with deformation rate stated above. The crushing strength of green/sintered pellets was computed using the following formula:

$$\text{Crushing strength, } \sigma = \frac{2.8P}{\pi X^2} \quad (1)$$



Figure 5: UCS testing machine.



Figure 6: Sintered pellets.

Where P is the load when fracture occurs, and X is the distance between the two parallel plates. In order to obtain a reliable outcome for crushing strength test, a total of 10 pellets per batch were crushed to obtain the average crushing value that was recorded in a table form. Particle density test and 24-hours water adsorption test for sintered pellets shown in Figure 6 were done in accordance with the standard steps and procedures stated

in BS 812 – Part 2 [39]. The particle density test and 24-hours water adsorption test were conducted in accordance with section 5.5 “method for aggregates 10mm nominal size and smaller”,

III. RESULTS AND DISCUSSIONS

Particle Size of Fly Ash and Palm Oil Fuel Ash : Particle size analysis was conducted on dried palm oil fuel ash and fly ash using Cilas 1090 Particle Size Analyzer. The particle size distribution of fly ash and palm oil fuel ash are shown in Figure 7 and Figure 8 respectively. The particle size of palm oil fuel ash was in the range of 0.04 μm to 240.00 μm , with majority of the particles fall within the range of 28 μm to 130 μm . It was observed that the mean diameter of palm oil fuel ash particles was 60.06 μm . Cumulatively in terms of particle diameter, 90%, 50% and 10% of the palm oil fuel ash particles were smaller than 117.33 μm , 53.39 μm and 11.31 μm respectively.

In comparison, the particle size of fly ash was in the range of 0.07 μm to 71 μm , with majority of the particles fall within the range of 8.5 μm to 17 μm and 25 μm to 43 μm . The mean diameter of fly ash particles was 17.11 μm , with 90%, 50 % and 10% of the particle diameter were smaller than 37.97 μm , 12.35 μm and 2.59 μm .

Comparing the mean particles size and cumulative particles value, the particle size of palm oil fuel ash is relatively larger than fly ash; the mean particle of palm oil fuel ash is 71.5% larger than that of fly ash. The benefits of fly ash having smaller particle size compared to palm oil fuel ash is its ability to form a denser layer when used in pellets production, the denser layer also known as shell, is able to reduce the permeability of the pellets, thus effectively reducing the percentage of water adsorption after sintering of pellets. This is the main reason why fly ash was used as an outer layer coating during pelletizing process instead of being mixed into the palm oil fuel ash and sodium silicate mixture.

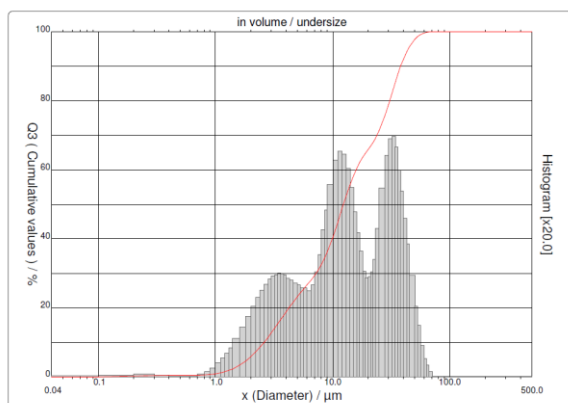


Figure 7: Particle size distribution of fly ash.

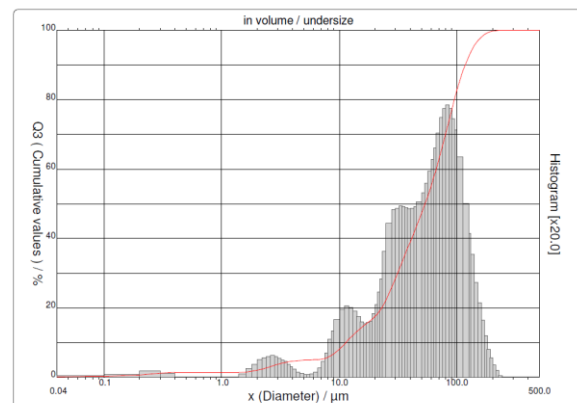


Figure 8: Particle size distribution of palm oil fuel ash.

Crushing Strength of Fresh Pellets : The effect of pellets size to the crushing strength of fresh pellets is shown in figure 9, the mix proportion were labelled in a way such that the numeric before alphabet A showed the binder content in percentage while the numeric after alphabet A showed the ratio of FA to POFA. The results showed that all fresh pellets

experience a reduction in crushing strength when the pellet size increases. The high strength exhibited by 40% FA + 60% POFA + 25% sodium silicate binder may be due to the capillaries of fresh pellets were fully filled with water during the pelletizing process, causing the fresh pellets to be stronger due to capillary action [40]. The observed reduction in crushing strength in relation to increasing particle size are in line with recent research observations [37]; this is due to the size effect as a decrease in strength of material is expected when the size of the material is large due to a larger volume that is subjected to strain during compression test [41].

The effect of binder content to the crushing strength of fresh pellets is shown in figure 10. It was noticed that the crushing strength for 40% FA + 60% POFA mix proportion with binder content less than 10% was very weak. However, the mix shows significant increases in strength when the binder content was increase to 15%, with a notable increase of 89% from 0.09 MPa to 0.84 MPa. The crushing strength of 40% FA + 60% POFA mix continued to increase beyond 15% binder content, with highest crushing strength of 1.74 MPa attained at 25% binder content. The crushing strength of 50% FA + 50% POFA increases and decreases with increasing binder content, with 0.56 MPa peak obtained at 20% binder content. The crushing strength of 60% FA + 40% POFA mix increases with increasing binder content except at 10% and 15% binder content where the crushing strength remains constant. The maximum crushing strength of 60% FA + 40% POFA mix was obtained at 25% binder content with a crushing strength of 0.49 MPa. This is due to the effect of binder which gives good binding properties to the palm oil fuel ash and fly ash particles [42]; the binding action effective wrap the particles to increase its crushing resistance, thus resulting in aggregate having higher crushing strength [43].

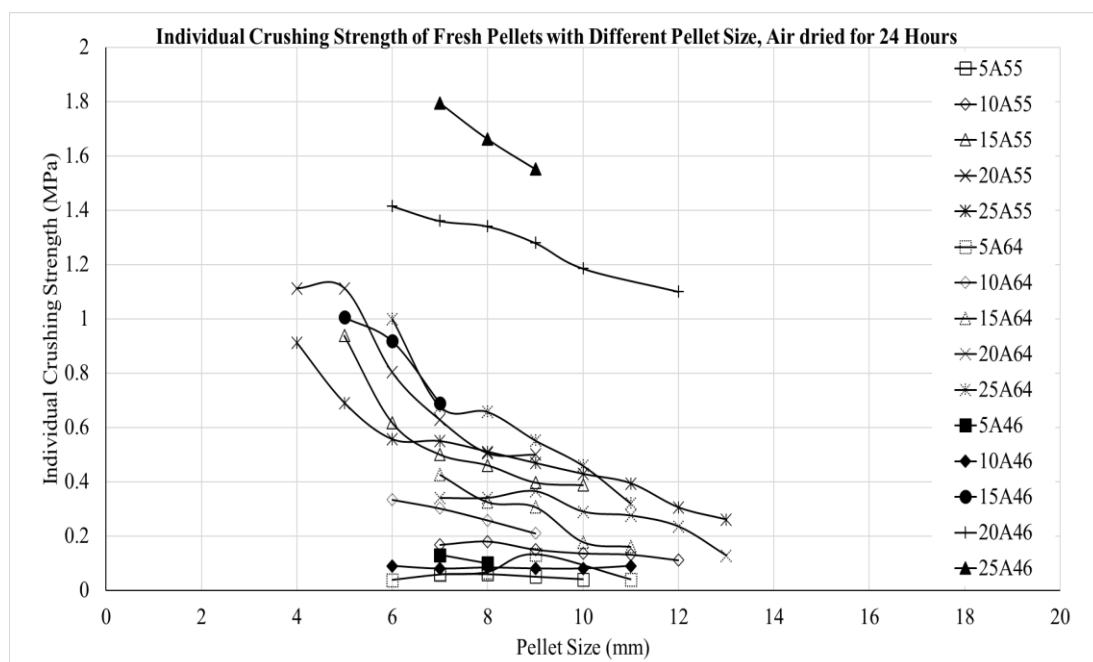


Figure 9: Relationship between pellet size and crushing strength of fresh pellets.

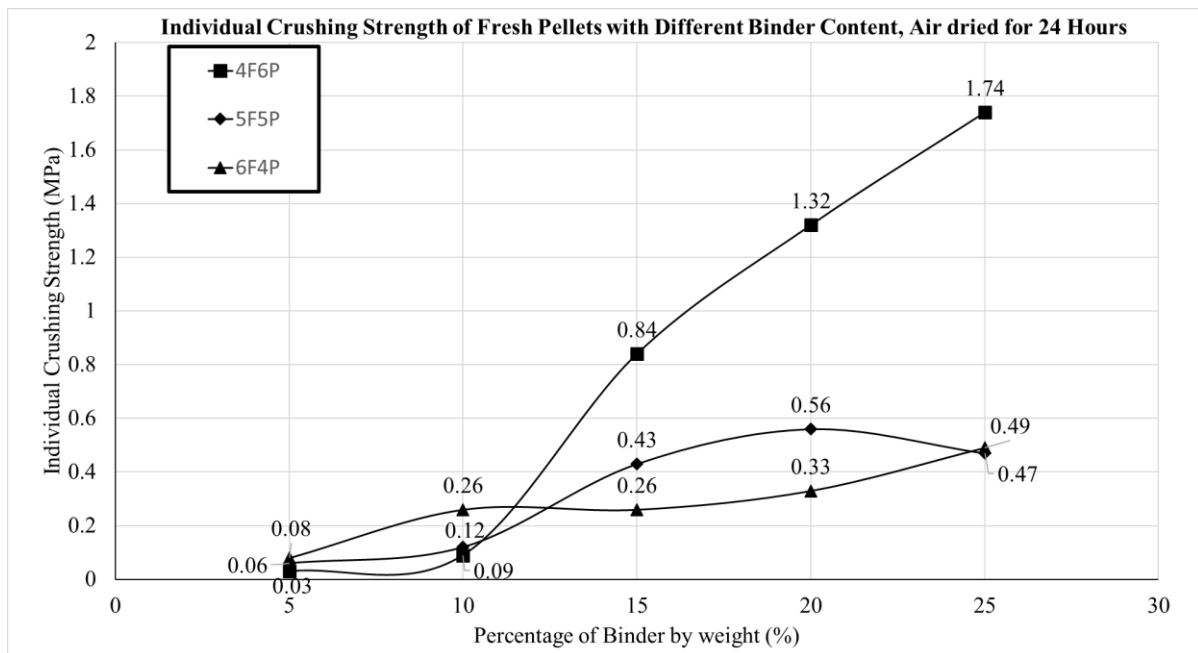


Figure 10: Relationship between binder content and crushing strength of fresh pellets.

From the fresh pellet crushing test, the mix proportion having crushing strength greater than 0.5 MPa has been identified. There were 4 mixes that was capable of producing fresh pellets with crushing strength greater than 0.5 MPa, the mixes are Mix-1 with 50% FA + 50% POFA + 20% binder, Mix-2 with 40% FA + 60% POFA + 15% binder, Mix-3 with 40% FA + 60% POFA + 20% binder and Mix-4 with 40% FA + 60% POFA + 25% binder, the pellets size having such strength were generally under 12 mm diameter.

Properties of sintered pellets: The effect of microwave sintering time to the crushing strength of sintered pellets is shown in figure 11, the alphabet F and P indicates fly ash and palm oil fuel ash respectively while the numeric following the alphabet indicates the binder percentage. Results showed that the crushing strength of sintered pellets generally decreases after 15 minutes sintering then increases after sintering for 20 minutes except for 4F6P20 mix. This phenomenon was most noticeable for 5F5P20 and 4F6P25 mix, the crushing strength of 5F5P20 mix sintered for 10 minutes was 2.79 MPa, it decreases to 2.52 MPa after sintering for 15 minutes then increased sharply to 3.26 MPa after 20 minutes sintering. Similarly, the crushing strength of 4F6P25 mix decreased from 1.93 MPa to 1.85 MPa, followed by sharp increment to 3.34 MPa after sintering for 20 minutes. The effect of increasing sintering time from 15 to 20 minutes resulted in significant increment in crushing strength for 5F5P20 and 4F6P25 mix, with a crushing strength improvement of 29.4% and 44.7% observed for 5F5P20 and 4F6P25 mix respectively. 4F6P20 mix shows decreasing crushing strength with increasing sintering time, the crushing strength decreased from 2.05 MPa obtained at 10 minutes sintering time to 1.43 MPa and 1.28 MPa at 15 and 20 minutes sintering time respectively. 4F6P15 mix shows a decrease in crushing strength at 15 minutes sintering followed by a marginal increase in crushing strength at 20 minutes sintering time, the crushing strength decreases from 0.66 MPa to 0.33 MPa followed by increment to 0.41 MPa for 10, 15 and 20 minutes sintering time respectively.

The highest crushing strength of aggregate were obtained at 20 minutes sintering time for 5F5P20 and 4F6P25 mix with 3.26 MPa and 3.34 MPa respectively, while the highest crushing strength for 4F6P15 and 4F6P20 mix was obtained at 10 minutes sintering time, with crushing value of 0.66 MPa and 1.93 MPa respectively. Sintering of 4F6P15 had yield unsatisfactory crushing strength that was lower than the crushing strength of pellets in fresh condition. The lower crushing strength observed on 4F6P15 and 4F6P20 mix may be due to insufficient binder content in the mixture to effectively bind the palm oil fuel ash particles. This can be proved by observing the difference between 5F5P20 and 4F6P20 mix, the binder content in terms of percentage was similar for both mixes which is 20% by weight of the total mix, however the ratio of palm oil fuel ash and binder was different due to the difference in designated mix proportion. 5F5P20 mix has a palm oil fuel ash to binder ratio of 2.5 compared to 4F6P20 mix that has 3.0. The fly ash content was not accounted due to the pelletizing process where fly ash was only added as an outer layer coating after the palm oil fuel ash and binder was mix homogenously and pelletized. Due to the low crushing strength of 4F6P15 mix, the investigation on particle density and water adsorption was not conducted on 4F6P15 mix.

Comparing the crushing strength value of sintered pellets to the recommended crushing strength value listed in table 4, the potential application of sintered pellets is discussed below. Table 4 had suggested that the sintered aggregate used in gardening or landscaping, geotechnical application or non-structural lightweight concrete and mortar, structural lightweight concrete and high-strength concrete should have crushing strength of at least 1.00 MPa, 1.80 MPa, 2.30 MPa and 5.00MPa respectively

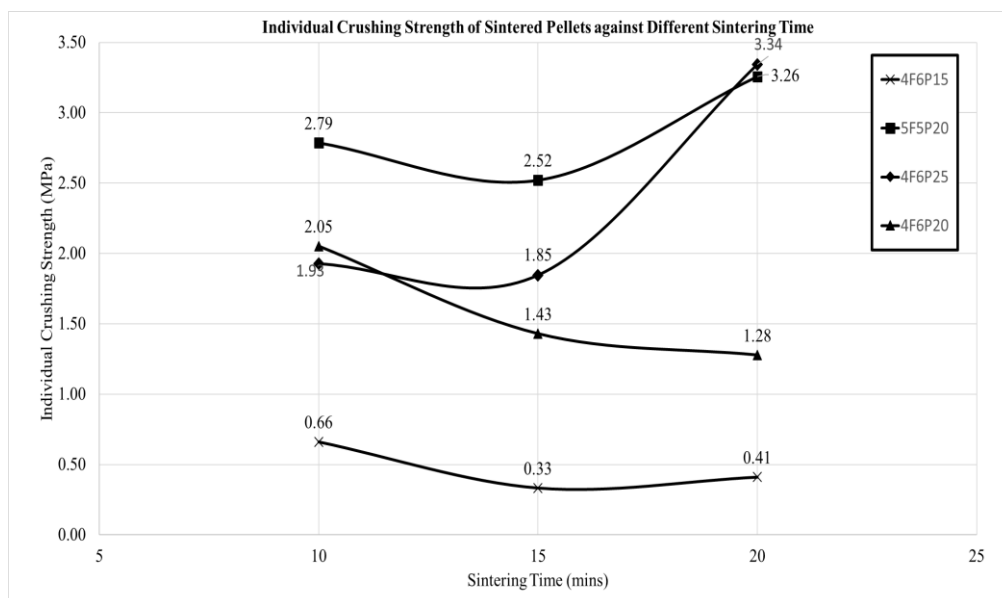


Figure 11: Relationship between microwave sintering time and crushing strength of sintered pellets.

In view of such requirements, 5F5P20 mix sintered for 10, 15 and 20 minutes exhibit crushing strength of 2.79MPa, 2.52 MPa and 3.26 MPa respectively and 4F6P25 mix sintered for 20 minutes exhibit crushing strength of 3.34 MPa, these mixes are potentially applicable in structural lightweight concrete. Apart from that, 4F6P25 mix sintered for 10 and 15

minutes and 4F6P20 mix sintered for 10 minutes are usable in geotechnical application and non-structural lightweight concrete and mortar as the crushing value meets the minimum requirement of at least 1.80 MPa. Lastly, 4F6P20 mix sintered for 15 and 20 minutes showed potential to be used in gardening, landscaping, thermal and acoustic insulation application.

The effect of microwave sintering time against particle density is shown in figure 12, the particle density of all mixes was less than 2.0 g/cm^3 which complied with the particle density requirement stated in table 4. The increased in microwave sintering time has resulted in an increase in particle density. The increment in density is more apparent after sintering for 20 minutes, the increment of particle density for aggregate ranges from 5% – 7%. Comparing the percentage of densification for aggregate sintered for 10 minutes and 20 minutes, 4F6P25 mix shows highest amount of densification up to 7% increment compared to 10 minutes sintering, 5F5P20 mix shows the least increment of 5% from 1.14 g/cm^3 to 1.20 g/cm^3 while 4F6P20 mix shows 6% increment in particle density with an increment from 1.40 g/cm^3 to 1.49 g/cm^3 . Among the tested mix, 4F6P25 mix shows the lowest particle density followed by 5F5P20 then 4F6P20 mix. This may be due to the bloating phenomenon that has occurred more prominently in 4F6P25 mix, this higher binder content in 4F6P25 mix has allowed sufficiently viscous glassy phase to be formed, some of the gas liberated was trapped by the viscous layer, leading to formation of porous core resulting in a decrease in particle density.

The effect of microwave sintering time against water adsorption is shown in figure 13. The water adsorption of aggregate increases when the sintering time increased from 10 minutes to 15 minutes sintering time, then the water adsorption of aggregate decreases when the sintering time increases from 15 minutes to 20 minutes. The sintered aggregate having the highest percentage of water adsorption is 5F5P20 mix, showing water adsorption of 16.56% at 20 minutes sintering time. In contrast, the water adsorption of 4F6P20 mix is the least, having only 10.46% for 20 minutes sintering time. The water adsorption for 4F6P25 mix fall in between the value exhibited by 5F5P20 and 4F6P20 mix, with a 13.69% of water adsorption recorded at 20 minutes sintering time. The relatively low amount of water adsorption of 4F6P20 mix is due to the densely packed fly ash outer shell reducing the water permeability of the aggregate, this also results in the mix having higher particle density.

Discussing on the potential application of sintered pellets with respect to water adsorption value, table 4 had suggested that sintered pellets with water adsorption of range 0% – 20% to be used in high-strength concrete and structural lightweight concrete; 0% – 34% to be used in non-structural lightweight concrete and mortar while 10% – 38% to be used in geotechnical applications, gardening, landscaping, thermal and acoustic insulation. Disregarding the requirements of minimum crushing strength, all mixes showed potential to be used in all of the listed applications above due to the value of water adsorption fall within the range of requirements. At such, the potential application of these mixes is identical to what had been discussed above when the requirement of minimum crushing strength is taken into account.

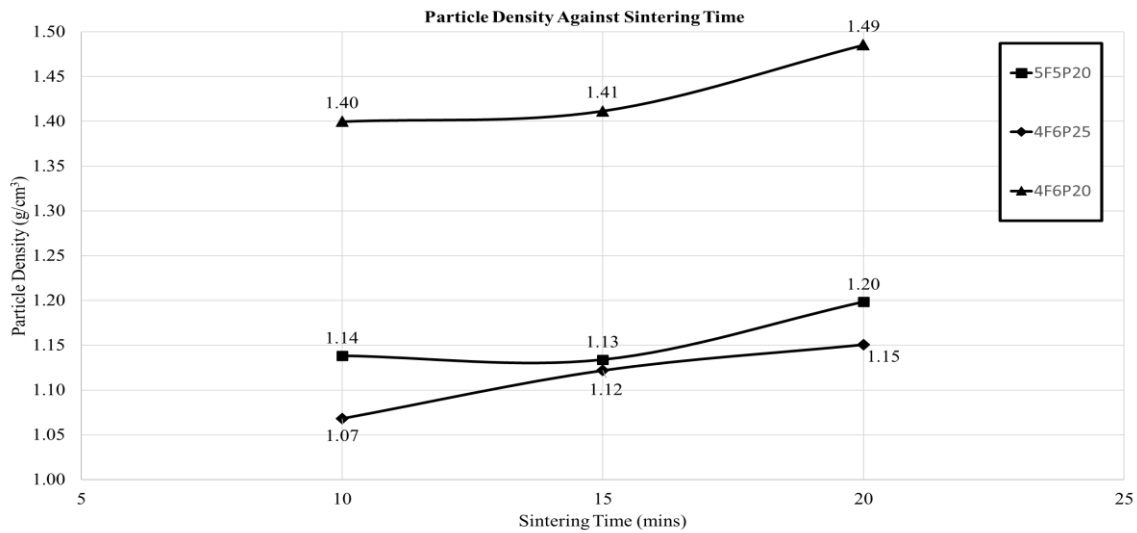


Figure 12: Relationship between microwave sintering time and particle density.

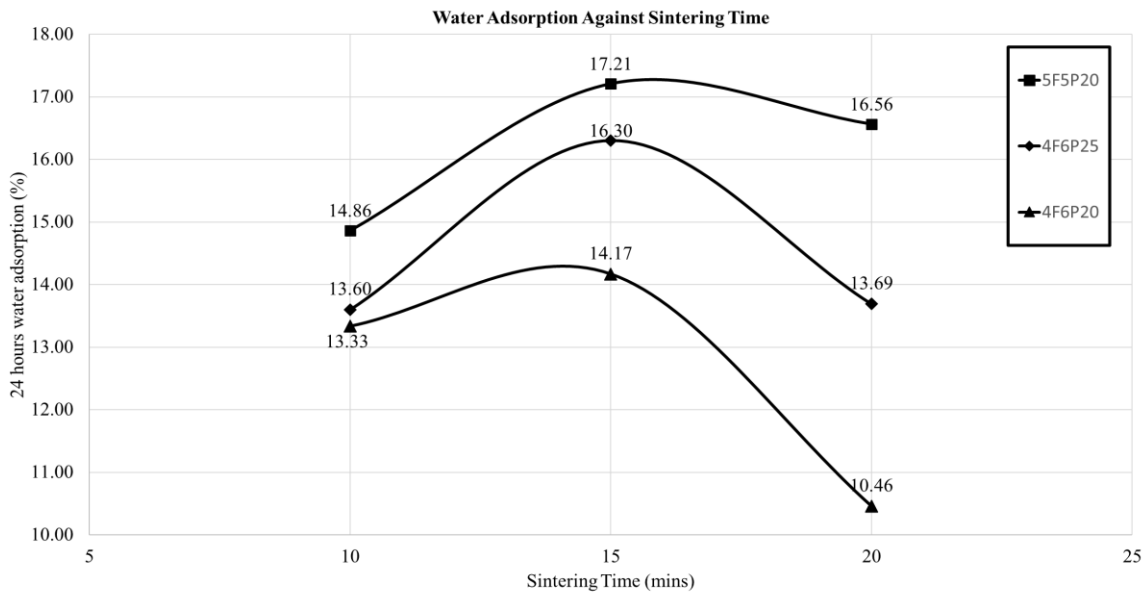


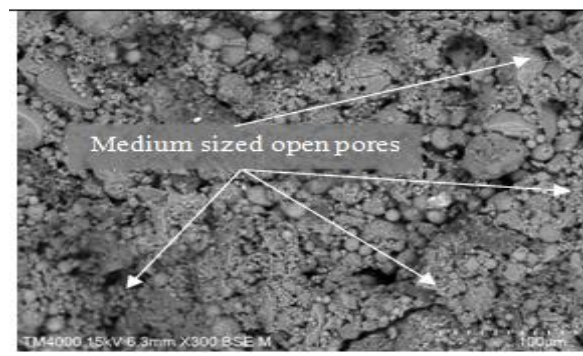
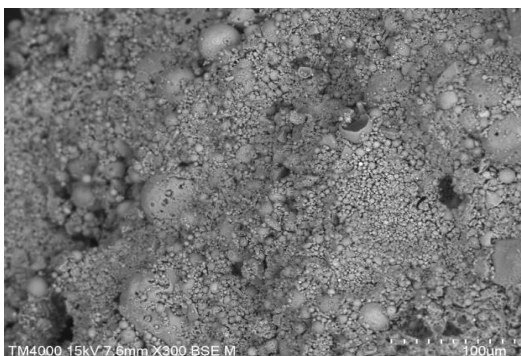
Figure 13: Relationship between microwave sintering time and water adsorption.

Scanning Electron Microscopy Analysis: The microstructure of sintered aggregate is shown in figure 14, the micrographs of each sintered pellets presented are inclusive of two elements, (1) the fly ash outer shell and (2) the core of the sintered pellets. The effect of sintering time can be observed from the micrographs showing the microstructure of the sintered pellets. The effect of sintering time towards the crushing strength of pellets can be observed from figure 14 (a)-(x). The micrograph (b), (d) and (f) shows the densification of the core for 5F5P20 mix, the increase in sintering time has resulted in the densification of the palm oil fuel ash microstructure. The particles of palm oil fuel ash show some degree of fusion with increasing sintering time, indicating that 20 minutes sintering time was sufficient to reach a temperature that partially melt the palm oil fuel ash particles, but not sufficient to allow the particle to fully reach the eutectic point. The insufficient glassy phase has limited the strength gain of sintered pellets. Similar observations were present on the 4F6P25 mix shown in micrograph

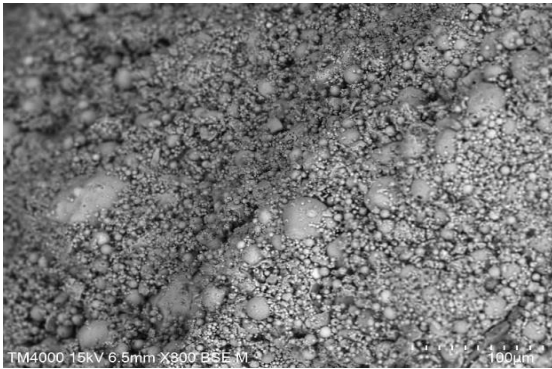
(h), (j) and (i), except that the fusion between palm oil fuel ash particles in the core of the sintered pellets was more prominent for 4F6P25 mix than in 5F5P20 mix. The higher degree of fusion presence in 4F6P25 mix may be due to the higher percentage of binder which effectively reduces the required temperature to reach eutectic point of the pellets. The combination of less open pores with more interconnectivity between the particles resulted in higher crushing strength of 4F6P25 mix and 5F5P20 mix.

The microstructure of 4F6P20 mix and 4F6P15 mix are shown in micrograph (m) to (x), both mixes showed decreasing crushing strength with increasing sintering time. This is due to the presence of numerous large open pores in the core of the sintered pellets, the micrograph (n), (p) and (r) shows that some connectivity between particles can be seen at 10 minutes microwave sintering time. However, with increasing sintering time, the interconnectivity between particles reduces and the number of open pores increases. In the case of 4F6P15 mix shown in micrograph (t), (v) and (x), the behaviours of microstructure changes show high degree of similarities with the observations made in 4F6P20 mix. The main difference between the microstructure of 4F6P20 mix and 4F6P15 mix is the amount of open pores, 4F6P15 mix shows numerous large open pores with minimal particle interconnection at 10 minutes sintering time while 4F6P20 mix showed significantly less amount of open pores with more interconnectivity between particles. This may be due to insufficient binder content that cause insufficient SiO₂ content to form sufficiently viscous glassy phase to trap the gases liberated, the gases liberated escaped without being trapped by the viscous glassy phase, leading to formation of void as a result of gas liberation. In conclusion, the presence of numerous large open pores and insufficient fusion between the particles were the main cause of the low crushing strength of 4F6P15 mix and 4F6P20 mix.

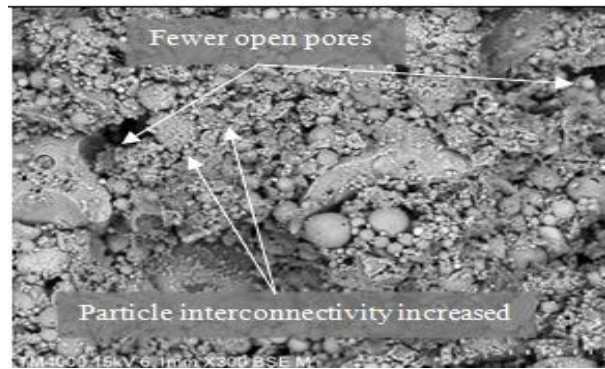
The water adsorption depends on the performance of the fly ash shell coating the aggregate, fly ash particles has a smaller size distribution which make it ideal for decreasing the water permeability of aggregate. The fly ash shell for 4F6P20 mix shown in micrograph (m), (o) and (q) indicates that the fly ash layer was tightly packed with little void presence between the fly ash particles, this has resulted in the higher particle density of 4F6P20 mix despite having lower crushing strength due to the presence of large open pores in the core, the tightly packed fly ash layer was also responsible for the lowest water absorption percentage exhibited by 4F6P20 mix. The microstructure of fly ash layer for 5F5P20 mix shown in micrograph (a), (c) and (e), and 4F6P25 mix shown in micrograph (g), (i) and (k) shows no significant changes with relation to the increasing sintering time. In short, the effect of sintering time on altering the microstructure of fly ash shell were not observed, probably due to insufficient temperature to induce microstructure refinement in fly ash shell.



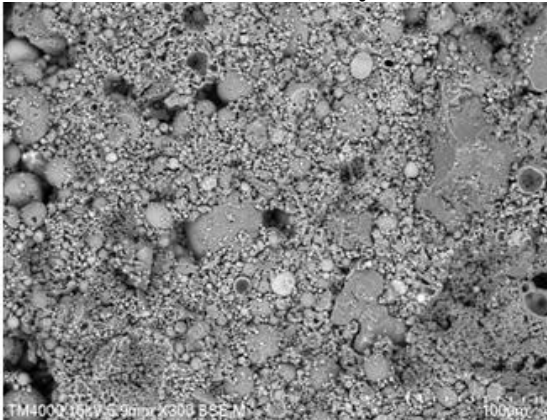
(a) 5F5P20 (10mins)-Fly Ash Shell



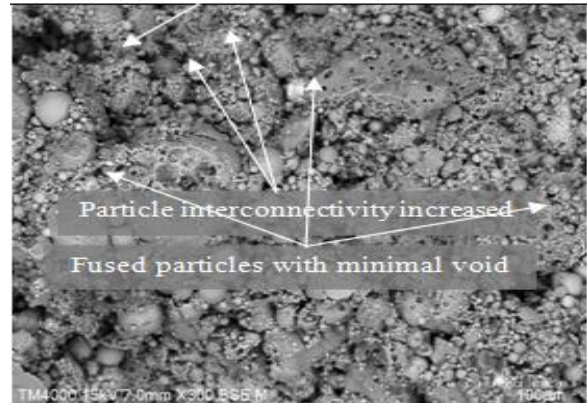
(b) 5F5P20 (10mins) -Core



(c) 5F5P20 (15mins)-Fly Ash Shell



(d) 5F5P20 (15mins)-Core



(e) 5F5P20 (20mins)-Fly Ash Shell



(f) 5F5P20 (20mins)-Core

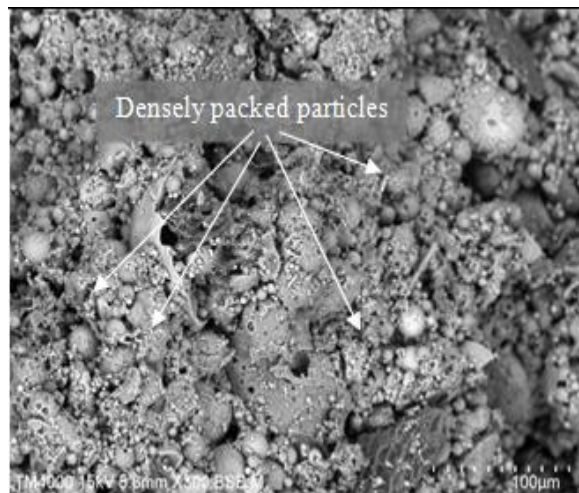
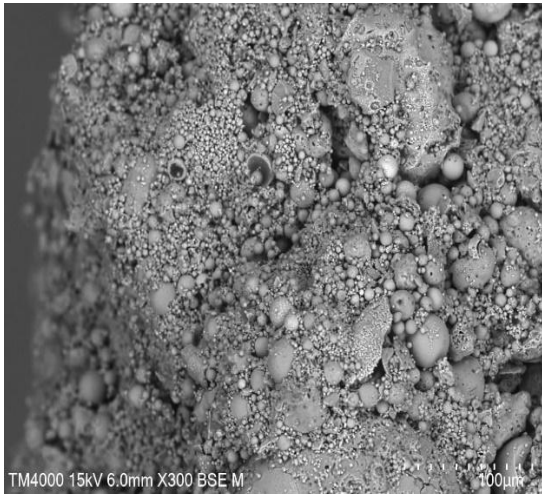
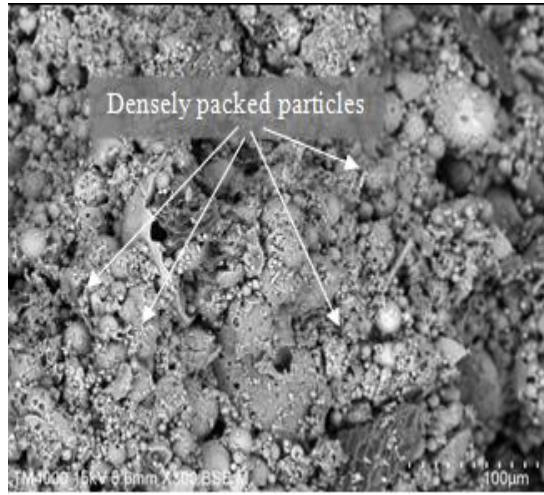


Figure 14: SEM images of sintered pellets.

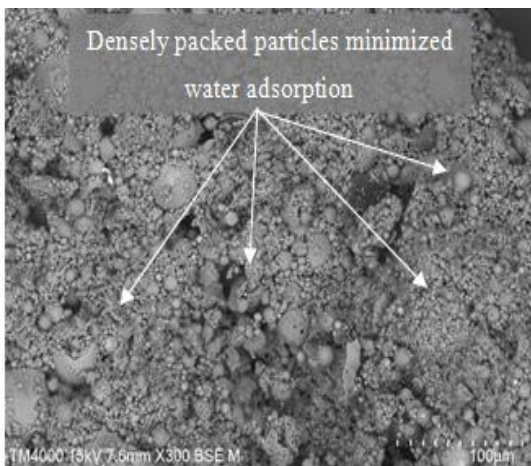
(m) 4F6P20 (10mins)-Fly Ash Shell



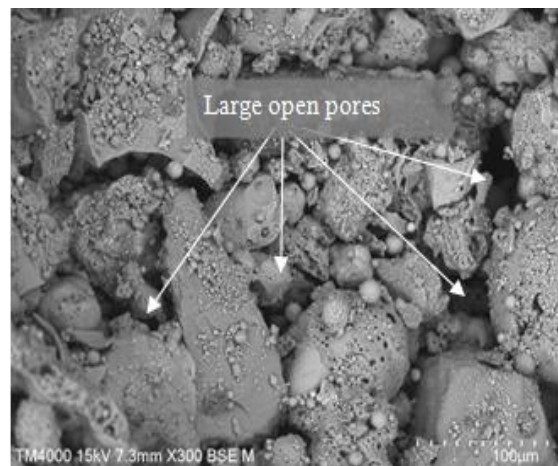
(n) 4F6P20 (10mins)-Core



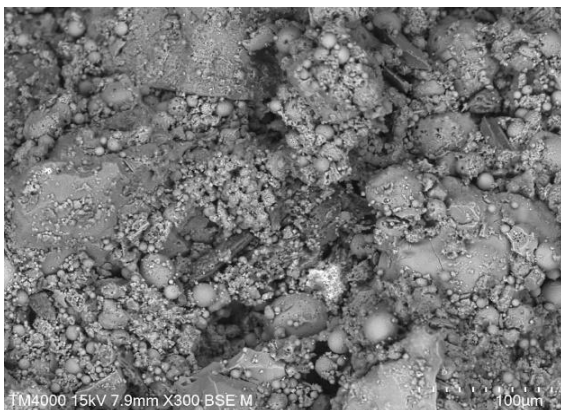
(o) 4F6P20 (15mins)-Fly Ash Shell



(p) 4F6P20 (15mins)-Core



(q) 4F6P20 (20mins)-Fly Ash Shell



(r) 4F6P20 (20mins)-Core

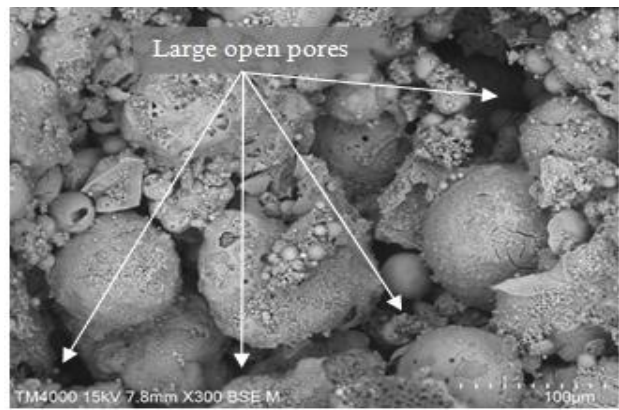
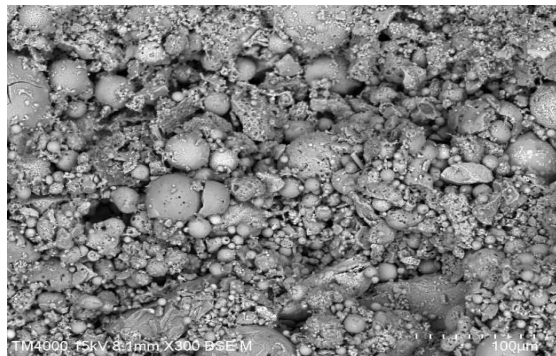


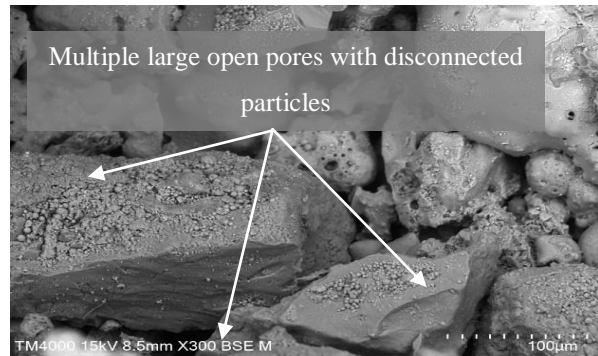
Figure 14: (Continued).

(s) 4F6P15 (10mins)-Fly Ash Shell

(t) 4F6P15 (10mins)-Core



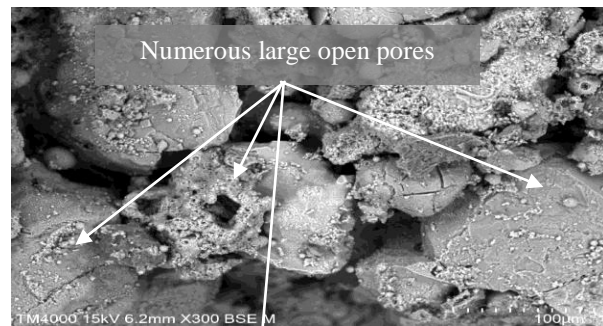
(u) 4F6P15 (15mins)-Fly Ash Shell



(v) 4F6P15 (15mins)-Core



(w) 4F6P15 (20mins)-Fly Ash Shell



(x) 4F6P15 (20mins)-Core

Figure 14: (continued)

Potential application of sintered lightweight aggregate: Comparing the properties of the produced lightweight aggregate from this paper with the classification of lightweight aggregate application suggested in literature [38], 5F5P20 mix sintered for 10 minutes, 15 minutes and 20 minutes and 4F6P25 mix sintered for 20 minutes shows possibility to be used in structural lightweight concrete applications. 4F6P25 mix sintered for 10 minutes and 15 minutes, and 4F6P20 mix sintered for 10 minutes fits the requirement to be used for geotechnical application and non-structural lightweight concrete and mortar. 4F6P20 mix sintered for 15 minutes and 20 minutes shows potential to be used in gardening, landscaping, thermal and acoustic insulation application. The summary of potential applications is tabulated in table 6.

Table 6: Potential Application of Sintered Lightweight Aggregate

Mix proportion	Description	Sintering Time (minutes)	Potential Application [Ref to Table 4]
5F5P20	50%FA+50%POFA+20% binder	10,15, 20	Structural lightweight concrete
4F6P25	40%FA+60%POFA+25% binder	20	
4F6P25	40%FA+60%POFA+25% binder	10, 15	Geotechnical application and non-structural lightweight concrete and mortar
4F6P20	50%FA+50%POFA+20% binder	10	
4F6P20	40%FA+60%POFA+20% binder	15, 20	Gardening, landscaping, thermal and acoustic insulation application.

IV. CONCLUSIONS

From the results of experimental investigation on the optimum mix proportion, properties of sintered aggregate with respect to sintering time and the relationship between crushing strength, particle density and water adsorption of aggregate. The following conclusions are drawn:

1. The acceptable mixes for the production of green pellets having crushing strength of more than 0.5MPa, expressed in the form of fly ash to palm oil fuel ash ratio and binder content are Mix-1 with 40% Fly ash and 60% POFA with 15% binder, Mix-2 with 40% Fly ash and 60% POFA with 20% binder, Mix-3 with 40% Fly ash and 60% POFA with 25% binder, and Mix-4 with 50% Fly ash and 50% POFA with 20% binder.
2. For Mix-3 and Mix-4, the crushing strength of sintered aggregate increases at 10 minutes sintering time, decreased at 15 minutes sintering time then increases sharply at 20 minutes sintering time due to insufficient temperature and gas liberation that cause void to increase momentarily at 15 minutes sintering time. However, the temperature was sufficient to partially melt the palm oil fuel ash particles at 20 minutes sintering time, leading to fewer void and higher interparticle connectivity resulting in higher crushing strength.
3. For Mix-1 and Mix-2, the crushing strength decreases with increasing sintering time due to insufficient temperature and binder content resulted in development of large void with minimum interparticle connectivity.
4. The crushing strength of aggregate increases with the increase in particle density, the water adsorption of aggregate decreases with the increase in particle density.

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