Production and Characterisation of Microfine Sized Palm Oil Fuel Ash (POFA) Originated from Bau, Lundu Palm Oil Mill

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Abstract. This paper investigates an effective and economical way for laboratory scale production of micro fine sized palm oil fuel ash (POFA) using an electric powder grinder. The raw POFA obtained from the palm oil mill is initially grinded by using Los Angeles abrasion machine, and then sieved using 150 μm sieve before it is burned in a furnace at 500°C. The burned POFA is then grinded using electric powder grinder to obtain the targeted micro fine sized. The physical, morphological and chemical properties of the micro fine sized POFA produced are analysed in the form of cement paste using Particle Size Analyzer (PSA), nitrogen sorption by using BET method, Fourier Transform Infrared Spectroscopy (FT-IR) and Scanning Electron Microscopy (SEM) with Energy Dispersive Spectroscopy (EDS). The results show that 96% micro fine sized POFA is produced when using the optimum grinding process. The microstructural analyses of cement paste with 20% micro fine sized POFA replacement give the optimum results that contribute to higher compressive strength. The overall results of this research show that the optimum grinding process by using electric powder grinder is relevant and can be used as pioneering work in the concrete production industry.

1 Introduction

In countries like Malaysia, Thailand and Indonesia, palm oil is one of the main agro-industries which produces a large amount of waste in the form of kernel shell, empty fruit bunches and fibers [2]. These by-products are used as fuel to heat up boiler for generation of electricity in the palm oil mill. As the second largest palm oil producer in the world, Malaysia has generated million tonnes of palm oil wastes each year [1,3,12]. The extracted product from the boiling process is known as palm oil fuel ash (POFA) which comprises a large amount of silica, thus, possesses pozzolanic properties suitable as cement replacement material.

Previous studies have showed that POFA can be used as a partial cement replacement material to produce high strength concrete with higher

impermeability and durability [8]. The inclusion of up to 40% micro fine POFA tends to increase the workability and viscosity of high strength concrete and could improve the corrosion resistance properties of steel inside concrete [4,5,6,7,9,13,14]. Furthermore, utilization of POFA as partial cement replacement materials contributes to the development of concrete with resistance to adverse environment condition [10,11].

Until now, the best method of producing finer size of POFA particle size (1-10 μm) is still unavailable. Although previous studies are able to produce micro fine sized POFA, the cost of production is still too expensive and the methods used are considered not effective [3,13]. Due to the significance contribution of micro fine sized POFA as a new potential cement replacement material, an effective and economical method to produce micro

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fine sized POFA is investigated. This research focuses on the simple and optimum grinding process and further investigates the physical and morphological properties of micro fine sized POFA which can affect the strength, workability and hardened concrete properties.

2 Materials and experimental procedures

2.1 Materials

The cement used in this experiment is Ordinary Portland Cement Type 1 whereas the Palm oil fuel ash (POFA) is obtained from Bau, Lundu Palm Oil Mill, Sarawak, Malaysia. The POFA is subjected to the 1st stage of grinding by using Los Angeles abrasion machine and sieved through 150 μm sieve to remove coarser particles. Next, POFA is burned at 500°C for 1 hour in a furnace to remove excessive unburned carbon. Then, it is subjected to 2nd stage of grinding by using electric powder grinder with 25000rpm to obtain micro fine sized which is ranging from 1-10 μm .

2.2 Experimental procedures

In order to determine the effectiveness of electric powder grinder, 2 samples were used which are; Sample A, 500 g of burned POFA and Sample B, 1000 g of burned POFA. All cement paste specimens were prepared with water to cement ratio of 0.35. One control sample and four samples were casted by replacing the cement with 10%, 20%, 30% and 40% of micro fine POFA (sample A). The test specimens were cured in water for 28 days.

The Particle Size Analyzer (PSA) was used to determine the physical size of burned POFA after 2nd stage of grinding. The morphology of cement paste was investigated by using Scanning Electron Microscopy (SEM) with Energy Dispersive Spectroscopy (EDS). Fourier Transforms Infrared Spectroscopy (FT-IR) analysis is carried out to

identify the pozzolanic characteristic in terms of hydration and identification of the composite group. Nitrogen sorption by using BET method is conducted to obtain the precise specific area of cement paste powder. Ordinary Portland Cement (OPC) and fly ash (FA) are also used as control samples.

3 Results and discussions

3.1 Particle Size Analysis (PSA)

3.1.1 Sample A

In this process, 500 g of burned POFA is grinded for 10 cycles which takes a duration of 30 minutes in total. After the 10th cycles of grinding are completed, the final amount of POFA collected is 490g. Therefore, 10g POFA loss is equivalent to 2% of the total amount of sample used. Micro fine particles of POFA is seen to slightly escaped from the tighten lid of the grinder during the grinding and while opening the lid after the grinding process.

3.1.2 Sample B

In this process, 1000 g of burned POFA is used and grinded for 15 cycles which takes a duration of 45 minutes in total. After the 15th cycles of grinding are completed, the final amount of POFA collected is 960 g. Therefore, 40 g POFA loss is equivalent to 4% of the total amount of sample used. The particle losses is also due to the same reason as in sample A. The d_{50} (μm) particle sizes of OPC and FA, 18.29 μm and 8.64 μm respectively are also determined to compare the different sizes of POFA, OPC and FA. Table 1.1 shows the particle sizes result for every cycle while, Figure 1.1 shows the Particle size d_{50} (μm) versus amount of cycles.

Figure 1.1: Particles Size at Diameter 50% vs Amount of cycles

Table 1.1 Particles Size Result for Samples A & B

Samples	A			B		
Cycles	Particle Size d_{50} (μm)	Mean Diameter (μm)	Specific surface (m^2/g)	Particle Size d_{50} (μm)	Mean Diameter (μm)	Specific surface (m^2/g)
0	29.09	34.51	1.117	29.09	34.51	1.117
1	15.97	21.38	2.296	16.78	19.67	1.555
2	8.49	12.10	5.782	10.04	14.24	5.058
3	6.10	9.07	6.415	9.39	12.30	1.705
4	4.58	7.04	8.627	7.88	11.59	4.766
5	4.16	6.27	7.135	7.86	11.72	3.736
6	3.41	4.35	6.071	7.36	11.47	5.093
7	2.94	3.70	13.288	7.13	11.03	3.125
8	2.87	3.15	5.798	6.98	11.74	2.775
9	2.67	2.90	17.893	6.98	10.14	3.927
10	2.23	2.41	15.871	6.46	10.77	5.286
11				6.22	9.37	3.700
12				5.46	9.32	4.138
13				5.34	7.95	7.834
14				5.02	7.52	4.530
15				4.94	7.38	2.583

Micro fine POFA which is ranging from 1-10 μm is obtained in both samples. For both Sample A and Sample B, the optimum cycle of grinding is at 6th and 12th cycles, respectively. This is because the diameter at 10%, 50%, 90% and mean diameter is already below 10 μm . By comparing the POFA diameter at 50% with OPC and FA, POFA with the highest amount of cycles has the smallest diameter. This factor influenced the surface area of the particle. Smaller particle size contributes to larger

surface area and lead to more pore capillaries that can be filled in concrete. Hence, the strength of the hardened concrete will be higher.

3.2 Fourier Transforms Infrared Spectroscopy (FT-IR)

Fourier transform infrared spectroscopy (FT-IR) analysis is conducted to identify the pozzolanic characteristics of harden cement paste

through absorption of infrared spectrum by the formation of main hydration products calcium hydroxide (CH) and calcium silica hydrate (C-S-H). A strong band ranging from 965 cm^{-1} to 975 cm^{-1} is due to Si-O asymmetric stretching vibration of tricalcium silicate, C_3S and /or dicalcium

silicate, C_2S which shows the formation of C-S-H [10]. Figure 1.2 shows the FTIR spectra of cement paste with POFA inclusion at (A) 0% replacement, (B) 10% replacement, (C) 20% replacement, (D) 30% replacement, (E) 40% replacement.

Figure 1.2: FTIR spectra of cement paste with POFA inclusion at (A) 0% replacement, (B) 10% replacement, (C) 20% replacement, (D) 30% replacement, (E) 40% replacement.

From Figure 1.2, the strongest absorption band centered at 972 cm^{-1} to 997 cm^{-1} . Cement paste with 0% replacement of POFA shows the peak at 948 cm^{-1} and 997 cm^{-1} which indicates the paste starts producing the C-S-H gel. As mention earlier

the strong band for C-S-H gel is at 965 cm^{-1} to 975 cm^{-1} . Thus, the spectra show the paste is producing a weak band of C-S-H. Cement paste with 10% replacement of POFA shows the peak at 967 cm^{-1} which shows the paste is producing a strong band

of C-S-H gel. Compare to the 0% replacement, the peak of 1109 cm^{-1} decrease to 1093 cm^{-1} . This indicates the extra amount of silica was added from the 10% of POFA replacement. The similar pattern was observed for 20% and 30% replacement. The absorption intensity of the corresponding peak increases with the percentage of replacement indicating the formation of more C-S-H in the cement paste due to the hydration of C_3S . Cement paste with 40% replacement shows a slight increment in peak which is 972 cm^{-1} . This is because of the large amount of POFA powder which takes longer time to contribute in hydration at later stage. Overall, cement paste with 10%, 20% and 30% replacement shows the strong band in the formation of the C-S-H gel.

3.3 Scanning Electron Microscopy (SEM) with Energy Dispersive Spectroscopy (EDS)

Scanning electron microscopy (SEM) is conducted to obtain the surface morphology of the cement paste while Energy Dispersive Spectroscopy (EDS) is carried out to obtain the chemical composition in the material. Figure 1.3 shows the surface morphology of the cement paste with POFA inclusion at (A) 0% replacement, (B) 10% replacement, (C) 20% replacement, (D) 30% replacement and (E) 40% replacement.

Figure 1.3: Surface morphology of the cement paste at (A) 0% replacement, (B) 10% replacement, (C) 20% replacement, (D) 30% replacement and (E) 40% replacement.

EDS analysis results can be obtained through mapping from the same point used for SEM. Table 1.2 shows the comparison of the Ca/Si ratio of cement paste with 0%, 10%, 20%, 30% and 40% of POFA replacement.

The results in Table 1.2 show that POFA tend to decrease in Ca/Si ratio which indicates

pozzolanicity characteristic. The results show that Ca/Si ratio is highest in 20% replacement of POFA, with 4.22 ratios. Ca/Si ratio will decrease with the increment of silica present in C-S-H gel [11]. Comparison with compressive strength is also determined. Figure 1.4 shows the compressive strength versus Ca/Si ratio for each paste.

Table 1.2: Comparison of Ca/Si ratio for each cement paste

Sample (28 Days)	Element (atomic %)		Ca/Si
	Ca	Si	
0%	37.19	10.03	3.71
10%	36.7	11.47	3.20
20%	44.67	10.59	4.22
30%	35.66	12.235	2.91
40%	31.6	18.613	1.70

Figure 1.4: Compressive strength vs Ca/Si ratio

From Figure 1.4, it is shown that cement paste with 20% replacement is high in term of compressive strength together with Ca/Si ratio. This indicates that there is suitable amount of calcium in cement which contributes to higher initial strengths of concrete. However, as the chemical reactions take place, cracks and shrinkage in concrete might be produced due to the greater heat developed. Therefore, too much Calcium oxide, CaO is one of the reasons that may lead to failure in concrete. POFA has less calcium content but high in silica. Only with the presence of sufficient calcium and moisture, pozzolanic reaction will takes place resulting in formation of C-S-H gel [6]. This reaction helps to generate

secondary C-S-H gel thus producing high strength concrete and makes concrete less destructible compare to OPC concrete. Hence, findings from EDS, supports findings from compressive test and FT-IR analysis.

3.4 Nitrogen sorption using BET_{N2} analysis

Nitrogen sorption BET_{N2} is conducted to obtain the precise specific area of cement paste. As shown in Table 1.3, the specific surface areas of POFA paste with inclusion of 10%, 20% and 30% are higher than 0% POFA replacement. This is because the micro fine sized POFA is used. Micro fine sized POFA has smaller particle sizes

compare with OPC which causes it to fill up the capillary pores in cement. Hence, this results in denser internal structure of concrete which could sustain higher capacity load. Since the specific area is greater, the contact surface between pozzolan materials and water is also larger. This

results to a better hydration process and increase the workability of concrete. The gaps between the particles of aggregates are able to be filled up with micro fine sized POFA which provides lubrication for aggregates to settle.

Table 1.3: Size of sample from BET_{N2} method

	0%	10%	20%	30%
Average Pore Radius (nm)	7.05	7.59	7.33	7.18
Total Pore volume (cc/g)	0.02	0.03	0.03	0.02
Specific Surface area (m ² /g)	52.87	69.39	65.00	63.97

4 Conclusion and Recommendation

Based on the results obtained from the experimental works on the production and characterization of micro fine sized palm oil fuel ash (POFA), the following conclusions can be made:

- I. A simple yet effective method for laboratory scale production of micro fine sized POFA has been developed using a 25000-rpm electric powder grinder. The efficiency of this method achieved up to 96% production of micro fine sized POFA ranging between 1-10 μm of median particle size (d_{50}) when the optimum grinding process is used.
- II. The microstructural analyses of the cement paste show formation of C-S-H gel as hydrated product is with the strongest band when 20% of the micro fine sized POFA replacement is utilized. Highest compressive strength together with Ca/Si ratio is also achieved indicating pozzolanic characteristics. BET_{N2} test gives average pore radius of 7.3nm with specific surface area of 65m²/g.

The results obtained indicate that the new approach using an electric powder grinder is successful in producing micro fine size POFA and this approach is envisaged applicable for used in the industry provided further modification is carried out in future work to increase the efficiency if this device.

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