Effect of Molarity of HCl on Production of Silica (SiO$_2$) From Palm Oil Fuel Ash (POFA)

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ABSTRACT. Palm oil fuels ash (POFA) is a by-product from palm oil industries that constitutes of different compounds such as silica (SiO$_2$), alumina (Al$_2$O$_3$) and many others, its abundances and improper disposal in waterways and as a landfill cause environmental pollution and hence need for its modification and usage. This paper presents an alternative way of producing silica from POFA. Untreated POFA was dried in an oven for 24 hours at a temperature of 110 °C and ground in a ball mill machine for 12 hours at a speed of 250 rev/s. After sieving, the powder was dispersed in 1 mole, 2 mole and 3 mole of HCl. An increase in the composition of SiO$_2$ was observed using X-ray Fluorescence (XRF) analysis due to the increase in the molarity of acid, X-ray Diffraction analysis (XRD) and Scanning Electron Microscopy (SEM) shows that POFA has a crystalline and porous cellular structure. This shows that acidic wash treatment is good and an improved method for the production of silica from POFA.

Keywords: Silica, POFA, Effect of molarity, Acidic wash treatment, Microstructure;

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1. INTRODUCTION

In countries like Malaysia and Thailand palm oil industries are some of the major agro-industries. To extract crude palm oil, the industries uses waste removed from fresh fruit of palm oil such as fiber, shell and empty fruit bunches to heat up the turbine and generate electricity for palm oil extraction [1]. After extracting crude palm oil, Palm oil fuel ash (POFA) is produced and disposed in waterways and as landfill of no economic value and causes environmentally unfriendly situation [2-6].

POFA has a pozzolanic properties therefore can be used as partial cement replacement without sacrificing for either the mechanical or physical properties of cement [4,7,8]. The only feasible solution to reduce future environmental problems that may be caused by disposal of POFA is to use it as replacement of cement in concrete or quartz in ceramic industry [9]. Malaysia is among the largest producers of palm oil, thus, facing environmental problems by disposing palm oil fuel ash. In 2007, it was estimated that 3 million tons of POFA was produced and this figures are expected to increase annually as the palm oil plantations increased [10]. Research by Altwair [11] shows that, to lower the cost of production of Portland cement and also improve the durability and engineering properties of concrete, partial replacement of cement with POFA
is necessary. Use of POFA not only contribute to a healthier and sustainable environment but also increasing the ecological properties of concrete.

Waste by-products such as fly ash, rice husk ash, silica fume and POFA were used by several researchers as either replacement or filler to produce concretes due to their potentials [12] but only few used these wastes as replacement of quartz, feldspar or clay. This paper aimed at presenting a different method of POFA treatment using hydrochloric acid (HCl) for the optimum production of silica.

2. MATERIALS AND METHODS

POFA usually contained unburnt materials; therefore removal of these excess carbons is necessary as to increase its reactivity. Raw POFA was dried in an oven for 24 hours at a temperature of 110 °C, to obtain a fineness material the powder was then ground in a ball mill machine for 12 hours at a speed of 250 rev/s. The powder is then sieved with a set of sieve for particle size less than 50 µm. The powder undergoes different types of acidic treatment in order to have an optimum amount of silica from the upcoming characterization. POFA powder was then divided into 3 and was dispersed in 1 mole, 2 moles and 3 mole of HCl and an electric shaker was used to shake it for 30 minute. A Whatman ashless filter paper was used to filter the solution and separate the treated POFA from the acidic solution. The powder was then dried again to remove the acid moisture in an oven for 24 hours at temperature of 110 °C and then grind using ball mill machine for 90 min and then sieve to remove the agglomeration and obtain a fine powder. Energy dispersive X-ray and X-ray florescence analysis (XRF) were used to analyze the elemental and chemical compositions of POFA, to determine the morphology and phase of the POFA powder scanning electron microscopy and X-ray diffraction analysis were used.

3. RESULTS AND DISCUSSION

The chemical composition of POFA was determined using XRF, the machine was operated at 60 KVP and 50 mA. The result shows the presence of SiO$_2$ and many other compounds, it is clear that SiO$_2$ is the major composition then followed by other compounds such as CaO, K$_2$O, P$_2$O$_5$, Fe$_2$O$_3$ and Al$_2$O$_3$ as seen in Table 1.

The result also shows that acidic wash treatment is efficient and enhances the production of silica from POFA as the amount of silica increased from 54.33 wt.%, 56.17 wt.% and 59.77 wt.% by 1 mole, 2 mole and 3 mole acid treatment, respectively as shown in Table 1. This shows that acidic wash treatment is a good treatment for the optimum production of silica (SiO$_2$) from POFA. Table 1 below shows chemical composition of POFA using XRF.

The morphological structure was analyzed using JOEL-JSM 6380 Scanning Electron Microscopy (SEM) operated at 15 KV. A 1000X magnification was used to capture the image of the POFA powder, from Fig. 1 it can be deduce that POFA has spongy and porous nature with irregular shapes. It can be seen clearly from the micrographs that due to the acidic wash treatment the powder agglomerates and form porous structure and become irregular. After the acidic wash treatment, the particle compacted together and form amorphous-like structure, the picture revealed that POFA treated with 2 mole and 3 mole of HCl become more stacked and packed together than that treated with 1mole of HCl acid and this may be as a result of the reaction between acid and chemical compounds present in palm oil fuel ash (POFA).

![Fig. 3 shows the XRD patterns of 1 mole, 2 mole and 3 mole HCl acid treatment, respectively. The result indicates the presence of quartz (Q) as the major phase and calcium silicide (CS), = aluminum phosphate (A) and manganese oxide (M) as minor phase for 1 mole HCl treatment. After the molarity of acid was increased to 2 mole the crystalline phases determined are](image)

Q as major phase and other minor phases. Similarly, for 3 mole the peak increase and the crystalline phase detected are Q as major phase and magnesium silicate as minor.
Table 1 Chemical composition of POFA

| Composition of POFA | Different molarities of acid (HCl) (wt.%): | 1 mole | 2 mole | 3 mole |
|---------------------|-------------------------------------------|--------|--------|--------|
| SiO₂                |                                           | 54.33  | 56.17  | 59.77  |
| C                   |                                           | 6.22   | 7.43   | 8.34   |
| CaO                 |                                           | 5.63   | 6.47   | 4.69   |
| K₂O                 |                                           | 5.63   | 5.86   | 4.65   |
| P₂O₅                |                                           | 6.65   | 5.73   | 3.20   |
| Fe₂O₃               |                                           | 5.20   | 5.16   | 4.37   |
| Al₂O₃               |                                           | 4.74   | 4.71   | 3.36   |
| Cl                  |                                           | 8.25   | 4.61   | 8.21   |
| MgO                 |                                           | 1.88   | 2.32   | 1.56   |
| SO₃                 |                                           | 0.91   | 0.94   | 1.34   |
| TiO₂                |                                           | 0.30   | 0.29   | 0.24   |
| MnO                 |                                           | 0.06   | 0.08   | 0.05   |
| Cr₂O₃               |                                           | 0.03   | 0.06   | 0.05   |
| ZrO₂                |                                           | 0.03   | 0.05   | 0.04   |
| CuO                 |                                           | 0.04   | 0.04   | 0.05   |
| SrO                 |                                           | 0.02   | 0.03   | 0.02   |
| ZnO                 |                                           | 0.02   | 0.02   | 0.02   |
| Na₂O                |                                           | 0.01   | 0.01   | 0.01   |
| Rb₂O                |                                           | 0.02   | 0.01   | 0.01   |
| NiO                 |                                           | 0.01   | 0.01   | 0.01   |

Fig. 1 SEM micrographs of (a) 1 mole (b) 2 mole and (c) 3 mole HCl acid treatment

Non-carbon EDX analysis of POFA treated with 1 mole, 2 mole and 3 mole of HCl acid reveals that (with assumption that all Si come in form of SiO₂ as reported by Kalapathy et al. [13]). SiO₂ has the highest elemental composition then the remaining elements. Fig. 2 shows the EDX of (a) 1 mole, (b) 2 mole and (c) 3 mole HCl acid treatment.
Fig. 2 EDX of (a) 1 mole, (b) 2 mole and (c) 3 mole HCl acid treatment

Fig. 3 XRD Pattern of 1mole, 2mole and 3mole HCl acid treatment
4. SUMMARY

The amount of silica increased as the molarity of acid wash increased, the XRF result indicated that acidic wash treatment is good for the production of silica (SiO$_2$) from palm oil fuel ash (POFA). The SiO$_2$ peaks show an incredible increase from 1 mole to 3 mole with increase from 54.33 wt.% to 59.77 wt.% This study proved that HCl wash treatment is suitable for the optimization of silica production.

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