CHARACTERIZATION OF A TREATED PALM OIL FUEL ASH

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ABSTRACT
Palm oil fuel ash (POFA) has been known to possess a pozzolanic property. The abundance of POFA as an agricultural waste makes it a promising candidate to be used as a supplementary cementation material in palm oil producing countries. This paper presents structural analysis and surface morphology of a treated Palm oil fuel ash (POFA). The untreated POFA was grounded for 1.5 hours in a ball mill to reduce the particle size and to improve reactivity. It was then heated at a temperature of 600°C for 1.5 hours in an electric furnace. X-ray fluorescence (XRF), X-ray diffraction (XRD) and scanning electron microscopy (SEM) were used to observe the surface and internal structure of the POFA. The results among other things revealed that the POFA consists mainly of silica (SiO2) with crystalline structure. Microscopic examination showed that the POFA has a porous cellular structure and consists of irregular-shaped particles. This study implies that POFA is a good candidate for various applications by ceramic industries.

Keywords: POFA, Morphology, SEM, XRD, XRF

INTRODUCTION
Palm oil fuel ash (POFA) is a by-product from biomass thermal power plants whereby oil palm residues are burnt to generate electricity. Malaysia is one of the largest producer of palm oil with around 41% of the total world supply in the year 2009–2010 (Awal and Hussin, 1997; Sata et al, 2004; Chindaprasit et al, 2007; Chandara et al, 2010; Tangchirapat and Jaturapitakkul, 2010; Sata et al, 2010; Altwair et al, 2011). In Thailand for example, it had been estimated that 2.1 million tons of biomass was used as fuel in 2004, producing about 100,000 tons (5%) of biomass ash (Sata et al, 2004; Tangchirapat and Jaturapitakkul, 2010). Since palm oil is one of the major raw materials used to produce bio-diesel, it is likely that the production of POFA will increase every year. Very little of the POFA produced is actually used. While some of it serves as low-value material for backfill or fertilizers, most of the POFA is disposed as waste in landfills, causing environmental and other problems such as health related problems (Chindaprasit et al, 2008; Frias et al, 2008; Ismail et al, 2010; Awal and Hussin, 2011; Sata et al, 2010; Kroehong et al, 2011).

POFA is greyish in colour, becoming dark with increasing proportions of unburnt carbon (Altwair et al, 2011; Bartell et al, 2010; Li, 2011; Steenland et al, 2010). Its chemical composition indicates presence of high amount of silica, which is considered to possess high potentials of serving as cement and porcelain replacement. The large amount of silica freely obtained from this source provides cheap alternative of silica for many industrial uses (Awal and Hussin, 1997; Mannan and Ganapathy, 2004; Rukzon et al, 2009; Jaturapitakkul et al, 2011).

This paper reports the results of an investigation into the structural analysis of POFA heated at a temperature of 600°C using X-ray fluorescence (XRF), Scanning electron micrographs (SEM) and X-ray diffraction (XRD). The aim was to study the structure and surface morphology of POFA collected from United Oil Palm Industries Sdn. Bhd. located in Nibong Tebal, Penang, Malaysia and compares it with that obtained from Thailand.

MATERIALS AND METHODS
The following tests were conducted to characterize the POFA.

X-ray Fluorescence (XRF)
The removal of excess carbon and other unburnt organic materials contained in POFA is important to avoid their potential negative effect on finished product. Thus, the POFA was dried in an oven at 100°C for 24 hours and then sieved using a set of sievers (50 µm) to remove the particles coarser than 50 µm. The untreated POFA (Fig.1b) was then ground in a ball mill to reduce the particle size to improve reactivity. The milling time was approximately 1.5 hours at 200 rev/min. The untreated POFA was heated at a temperature of 600°C for 1.5 hours in an electric furnace. After the heat treatment, the colour of the POFA changed from light brown to greyish red (Fig.1a) when the unburnt residue was removed, after which it was subjected to the XRF analysis. The machine used for the analysis was XRF Bruker S4 Pioneer which was operated at 60 KVP and 50 mA.
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Scanning Electron Microscopy (SEM)
JOEL-JSM-6380 Instrument was used to study the morphology of the POFA which is available at Mechanical Laboratory, Universiti Tun Hussein Onn Malaysia. Small amount of POFA powder was poured on the carbon tape which is attached to the holder. Then the excess powder was blown off with air gun to ensure that only small pieces of the powder remain on the tape. After that, it was put into in the SEM chamber for an analysis. The SEM machine was operated at 10kV. A magnification of X100 was used to capture the photo of the sample.

X-Ray Diffraction (XRD)
The POFA samples were subjected to X-Ray Diffraction (XRD) analysis using an X-Ray Diffractometer to determine their silica structure. Prior to analysis, the ash samples were ground to a powder form by simple pounding using a mortar and pestle due to its brittle nature. The ground samples were analyzed by Cu Kα radiation with a scanning rate of 0.05° per second 40kV/20A, at 3° ≤ 2θ ≤ 90°. The X-Ray Diffractometer (Model Bruker D8 Advance) is available for use at the Faculty of Civil and Environmental Engineering, Universiti Tun Hussein Onn Malaysia.

RESULTS AND DISCUSSION
Scanning Electron Microscopy
Fig. 2 shows the SEM results of the treated POFA particles; the particles were irregular in shape and having porous texture. In addition, there was no agglomeration of POFA particles after the heat treatment (Fig. 2). The main component of the treated POFA is SiO₂ (Table 1), which indicates that the chemical composition of the treated POFA from Malaysia gives 66.91 wt% of SiO₂ compared to the one obtained from Thailand as reported by Kroehong et al. (2011) which gives 55.7 wt% of SiO₂. The second component of the POFA from Malaysia gives 6.44 wt% of Al₂O₃ compared to the POFA from Thailand which gives only 0.9 wt%.
Table 1: Chemical analysis of POFA (wt %)

<table>
<thead>
<tr>
<th>Chemical Composition</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>Fe₂O₃</th>
<th>CaO</th>
<th>K₂O</th>
<th>P₂O₅</th>
<th>MgO</th>
<th>SO₂</th>
<th>Na₂O</th>
<th>LOI</th>
</tr>
</thead>
<tbody>
<tr>
<td>POFA (Malaysia)</td>
<td>66.9</td>
<td>6.44</td>
<td>5.72</td>
<td>5.56</td>
<td>5.20</td>
<td>3.72</td>
<td>3.13</td>
<td>0.33</td>
<td>0.19</td>
<td>2.30</td>
</tr>
<tr>
<td>POFA (Thailand)</td>
<td>55.7</td>
<td>0.9</td>
<td>2.0</td>
<td>12.5</td>
<td>11.9</td>
<td>5.1</td>
<td>2.9</td>
<td>1.0</td>
<td>4.7</td>
<td></td>
</tr>
</tbody>
</table>

[Kroehong et al., 2011]

X-Ray Diffraction (XRD) Analysis

X-Ray Diffractometer is a mechanical device for obtaining X-ray intensities as a function of the angle between the incident and the diffracted beams. Fig. 3 shows the result of phase diagram (called a diffractogram) which indicates that the crystalline phases determined are:

- Q = Quartz
- C = Cristoballite
- G = Grossular \((Ca_3Al_2(SiO_4)(OH)_4)\)

Fig. 3 shows that the peaks increased after treatment. Hence, the treated POFA is expected to give more compressive strength because of the silica content.

Fig. 3: X-ray diffraction (XRD) spectra for the POFA

Figure 4 shows the particle size of POFA. The lowest and the highest percentages which are \((d_{10})\) and \((d_{60})\) of the POFA particle size are 1.31 μm and 15.36 μm, respectively. The average particle \((d_{30})\) size is 5.40 μm.
CONCLUSION
POFA is a valuable natural resource not only as a good source of silica, but also as a source of lignocellulosic material which can be potentially used to produce a range of valuable products. However, product development will require greater understanding of the POFA. The information provided here could form both a useful background on the compositional and morphological characteristics of the POFA surface as well as its internal tissues. Therefore the extension of knowledge on structural analysis and surface morphology of this POFA is very important for the determination of which type of POFA to be used by the industries.

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