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Effect of Preliminary Calcinations on the Properties of Boiler Ash for Geopolymer Composite

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Abstract: This study concentrates on the effect of calcinations towards producing geopolymer paste. Boiler ash was one of the major wastes from the palm oil processing industry where it was widely available in Malaysia. The raw boiler ash (BA1) was calcined at two different temperatures which were 800 ºC (BA2) and 1000 ºC (BA3) for 1 hr. Then, boiler ash was mixed with alkaline activator consists of sodium hydroxide (NaOH) and sodium silicate. The ratio of solid/liquid and sodium silicate/NaOH was 1.5 and 2.5 for all mixtures. Chemical composition, morphology of calcined boiler ash and the compressive strength of geopolymer paste were investigated. From XRF analysis, the chemical composition has been altered by the calcinations process where the chemical compositions tend to fluctuating. Besides that, the morphology of boiler ash also changed due to calcinations process. The maximum compressive strength of geopolymer was 19.4 MPa contributed by BA2. When the calcinations temperature increased until 1000 ºC, the compressive strength was decreased. It can be concluded that, the calcinations temperature influence the chemical composition, morphology of particles and also the strength of hardened geopolymer.

Key words: Geopolymer, Boiler ash, Calcinations, Compressive strength.

INTRODUCTION

Malaysia is one of the largest countries that had exported palm oil all over the region where up to 2009 there are 4.7 million hectare of palm oil estate (Program Transformasi Ekonomi, Hala Tuju Untuk Malaysia, 2009). With the large production of palm oil, many waste has been created from this industry such as empty fruit bunch (EFB), palm press fibre (PFF), palm kernel shell (PKS), palm kernel cake (PKC), decanter cake (DC), palm oil mill effluent (POME), palm oil mill sludge (POMS), boiler ash and also palm oil fuel ash (POFA) (Embrandiri, *et al*, 2012).Boiler ash was obtained from burning the palm fibre and kernel shells in the boiler where it consists of clinker and ashes (Subramaniam, *et al,* 2008). The production of boiler ash was estimated to be over 4 million tones/ year (Boey, *et al*, 2011). Usually the boiler ash was used for land application such as roads in the palm oil mill (Vijaya, *et al*, 2008). Nowadays, many waste materials have been utilized to produce more beneficial product such as geopolymer.

Geopolymer can be defined as inorganic polymers based on aluminosilicates and can be produced by synthesizing pozzolanic compounds or aluminosilicate source materials with highly alkaline solutions (Kong, *et al*, 2007). The strength of geopolymer was comparable or better properties than conventional cementitious binder where it produce less emission of greenhouse gases (Temuujin and Van Riessen, 2009). The common materials uses as geopolymer were fly ash, calcined kaolin (metakaolin), and ground granulated blast furnace slag (ggbs) because these materials were rich in alumino-silicate. The applications of geopolymer were included cement and concrete (Hardjito, *et al*, 2004; Liew, *et al*, 2012; Bakharev, 2005; Duxson, *et al*, 2007), coating application (Zhang, *et al*, 2010; Zhang, *et al*, 2010; Zhang, *et al*, 2012; Zarina, *et al*, 2013) and also fire resistance purpose (Temuujin, *et al*, 2010; Temuujin, *et al*, 2009).

A few researchers (Temuujin and Van Riessen, 2009; Elimbi, *et al*, 2011) have studied about the effect of calcinations of fly ash and kaolin to the strength of the geopolymer where it was found that by calcinations of fly ash at temperature 500 ºC and 800 ºC has lowering the reactivity of the fly ash and reduction in strength (Temuujin and Van Riessen, 2009).

However, in the case of kaolin the compressive strength of calcined kaolin in temperature between 500 ºC and 700 ºC has increase the geopolymer strength but reduction in strength was observed at temperature above the 700 ºC (van Jaarsveld, *et al*, 2003). The current study investigates the effect of palm oil boiler ash calcinations to the strength of geopolymer pastes.

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MATERIALS AND METHODS

Materials:

The raw boiler ash was obtained from palm oil processing factory in Penang, Malaysia and the sample of boiler ash as shown in Figure 1 below. Since the boiler ash consist of larger particle of kernels, unburn palm oil nutshells and clinkers; hence it required grinding process to refine the particles sizes. After that, the boiler ash was sieved passing through 100 µm sieve to remove coarser particles. Subsequently, the boiler ash was heated at 800 ºC and 1000 ºC for 1 hr to increase the reactivity of the boiler ash. The chemical composition of boiler ash was analyzed using X-ray Fluorescent (XRF) and showed in Table 1.

Fig. 1: Palm oil boiler ash.

Sodium hydroxide (NaOH) and sodium silicate were used as alkaline activator to synthesis the boiler ash. The NaOH pellets with 99% purity was made in Taiwan with brand name of Formosoda-P was used to produce 12 M NaOH solution by dilute the NaOH pellets with distilled water. Meanwhile, a technical grade of sodium silicate solution was supplied by South Pacific Chemical Industries Sdn. Bhd. (SPCI), Malaysia. The sodium silicate solution consists of SiO_2 = 30.1%, Na_2O = 9.4% and H2O = 60.5 % (SiO_2/Na_2O = 3.2), specific gravity at 20 ºC = 1.4 kg/cm^3 and viscosity = 0.4 Pa s.

Mix Design and Samples Preparation:

The ratio of solid/liquid (S/L) and sodium silicate/ NaOH used in this study were constant for all mixtures. The geopolymer paste was produced by mixing the boiler ash with alkaline activator for a few minutes using mechanical mixer until homogeneous mixture was obtained and the mix design was listed in Table 2 below. After that, the geopolymer mixture were placed in 50mm x 50mm x 50mm mould and cured at 80 ºC for 24 hr. The compressive strength was tested on three hardened geopolymer samples at 28 days and the average strength was calculated.

RESULT AND DISCUSSION

Chemical Composition:

The analysis from XRF showed that BA1 rich in silica oxide (SiO_2), followed by calcium oxide (CaO), ferum oxide (Fe_2O_3), and potassium oxide (K_2O) as in Table 1. The content of SiO_2 was reduced for BA2 (35.1 %) but increase in BA3 (41.2%). However, the content of Al_2O_3 was disappearing in BA3 possibly due to excessive heat. The content of CaO which influence the properties of fresh geopolymer mixture and also hardened geopolymer increased in BA2 (32.9 %) but decrease in BA3 (20.8%) (Xu, *et al*, 2004). Hence, calcinations temperature has influence the chemical composition of boiler ash.

Table 1: Chemical composition of boiler ash

Compositions (wt. %)	Boiler ash (BA1)	800ºC calcined boiler ash (BA2)	1000ºC calcined boiler ash (BA3)
SiO_2	40.60	35.1	41.2
Al_2O_3	3.71	2.6	-
Fe_2O_3	15.74	8.6	15.55
CaO	19.60	32.9	20.8
MgO	1.30	1.7	1.7
P_2O_5	2.73	3.15	3.39
K_2O	13.80	13.5	14.9
SO_3	0.44	0.86	0.42
TiO_2	0.35	0.33	0.37
MnO	0.28	0.33	0.32

Table 2: Mix design of boiler ash

Samples	S/L ratio	Sodium silicate/NaOH ratio
Raw boiler ash (BA1)	1.5	2.5
800ºC calcined boiler ash (BA2)	1.5	2.5
1000ºC calcined boiler ash (BA3)	1.5	2.5

Compressive Strength:

The compressive strength of geopolymer was tested at 28 days and the result was shown in Fig. 2 below. It displayed geopolymer sample from BA2 produced the maximum compressive strength which is 19.4 MPa. Meanwhile, the lowest compressive strength obtained is 7.4 MPa from BA3. The compressive strength was increased when the boiler ash was calcined at 800 ºC then drops when the calcined temperature increased up to 1000 ºC.

Morphology Analysis:

Figures 3 (a-c) showed the closed up of the BA1, BA2 and BA3 particle when observed using Scanning Electron Microscope (SEM). BA1 displayed solid particles, irregular surface with flaky like feature as label in Figure 2 (a). Nevertheless, the morphology of BA2 showed some hollow structure in the particle and smaller solid particles also present. For BA3 most of the larger particle where consists of smaller particles that have coagulated on the surface.

Besides that, the morphology of the boiler ash particles also influences the mixing process of geopolymer samples. Among three types of boiler ash, BA3 was the hardest to stir even though the ratio of S/L was the same for all samples. This can be contributed by shape of the particles where the mixture tends to coagulated, hard to mix and insufficient liquid.

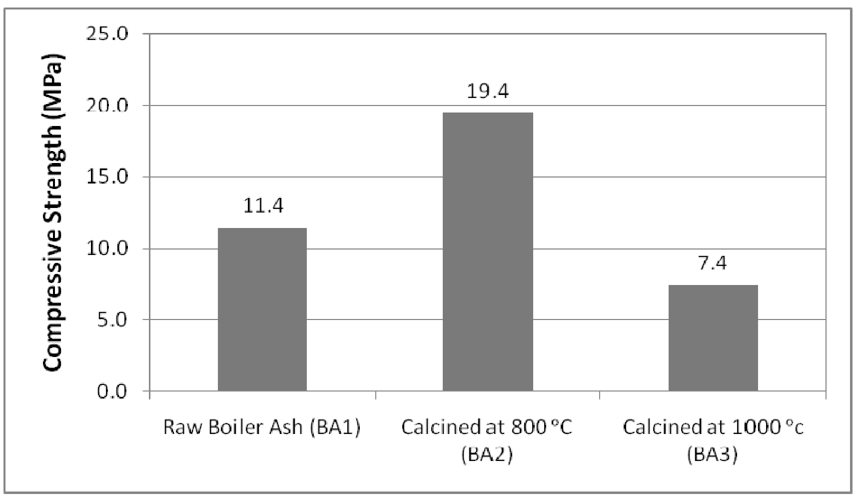
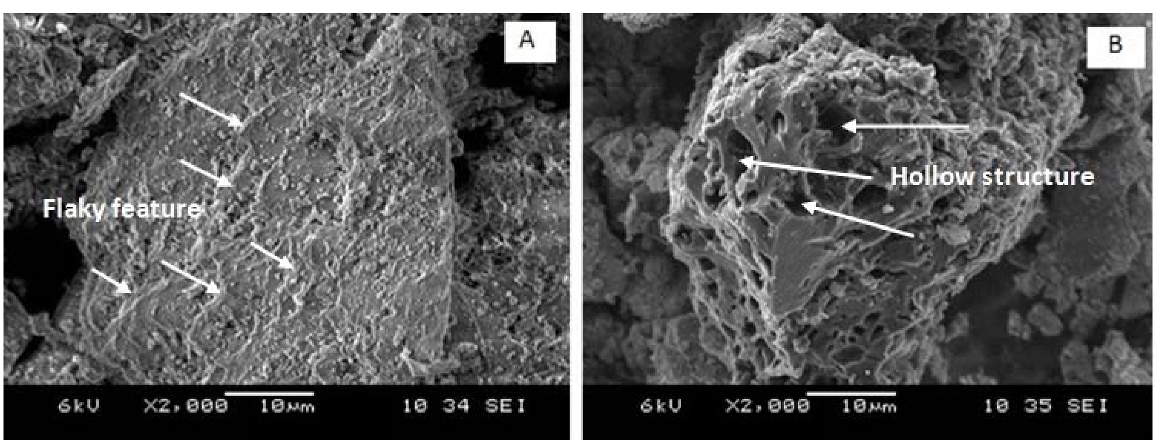


Fig. 2: Compressive strength of geopolymer at 28 days.



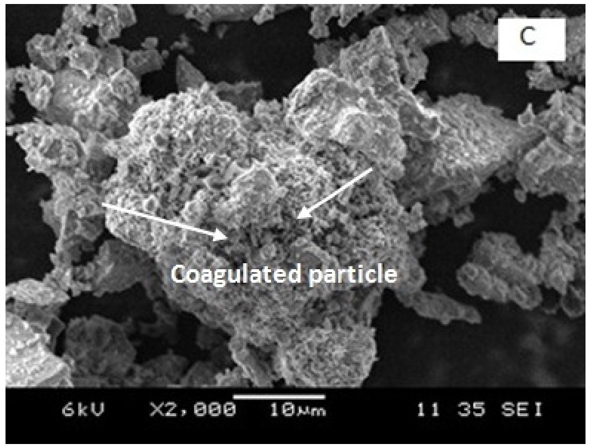


Fig. 3: Micrograph of (a) raw boiler ash, BA1 (b) calcined at 800 ºC, BA2 and (c) calcined at 1000 ºC, BA3

Conclusions:

From this study, a few conclusions were made as below:

a) The chemical composition of boiler ash was fluctuating when it heated up to 800 ºC (BA2) and 1000 ºC (BA3).

b) The heat treatment has changed the morphology of boiler ash as observed under SEM. The BA1 showed solid particles with flaky surface, while the heat treatment has changed the particles of BA2 become hollow and BA3 was coagulated with small particles of boiler ash.

c) The compressive strength of geopolymer paste increased with calcinations temperature up to 800 ºC (BA2) but drops at 1000 ºC (BA3).

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