Mechanism of Cement Paste with Different Particle Sizes of Bottom Ash as Partial Replacement in Portland Cement

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Manufacturing of Portland cement consists of high volume of natural aggregates which depleted rapidly in today construction field. New substitutable material such as bottom ash replace and target for comparable properties with hydraulic or pozzolanic properties as Portland cement. This study investigates the replacement of different sizes of bottom ash into Portland cement by reducing the content of Portland cement and examined the mechanism between bottom ash (BA) and Portland cement. A cement composite developed by 10% replacement with 1, 7, 14, and 28 days of curing and exhibited excellent mechanical strength on day 28 (34.23 MPa) with 63 μm BA. The porous structure of BA results in lower density as the fineness particles size contains high specific surface area and consume high quantity of water. The morphology, mineralogical, and ternary phase analysis showed that pozzolanic reaction of bottom ash does not alter but complements and integrates the cement hydration process which facilitate effectively the potential of bottom ash to act as construction material.

Keywords: Bottom ash, Portland cement, pozzolanic properties, ternary phase analysis

Manufacturing of Portland cement consists of high volume of natural aggregates which depleted rapidly in today construction field [1]. Hence, it is critical and necessary to discover a new substitutable material to replace and target for comparable properties as Portland cement with sustainable building material especially those materials that have hydraulic or pozzolanic properties. Coal combustion products increasing problem for landfilled with limited land and environment protection issues have raised concern throughout the world and this leads alternative way to recycle and produce value added product. Recycling the ash in cement is an environmentally friendly solution for cement industry [2]. Coal combustion products mainly composed of fly ash, bottom ash, boiler slags, and fuel gas desulfurization residues where have been used as construction material for over 2000 years that assessed by U.S. Environmental Protection Agency [3, 4].

Although there are many researchers and research towards reuse BA in varies area, actual utilization is still under 50wt% of total production [4]. The most commonly used of BA in construction field as fine and coarse aggregates, lightweight aggregates, road construction, embankments, building materials industry, raw material for ceramic industry and also other purpose in open cast mining or marine applications [5-7]. BA from coal combustion presents a high variability of its properties, thus understanding the chemical composition and pozzolanic reactivity of BA is needed.

Chemically bottom ash is composed of silica, iron, calcium, and alumina with small amount of magnesium, and sulphate which the constituent is quite similar to Portland cement but still depending on the incineration of coal combustion [8]. In some cases, the high reactivity of bottom ash could cause some long term detrimental effects on the properties of final product [9], the physical properties of bottom ash is coarser particle with irregular and rough structures [10]. The chemical and physical properties make it suitable to replace in construction engineering application. However, the pozzolanic properties of bottom ash are highly dependent on physical characteristic of the original coal [11, 12]. For this reason, it is significant to understand the mechanism of hydration products of bottom ash in Portland cement as replacement.

In this study, we fabricated new cement composite by mixing BA with OPC with direct replacement of BA into Portland cement. The aim of this study is to find out the chemical composition and ability of three different sizes (63, 75, and 150 μm) bottom ash hardened cement paste that prepared with 10 wt% replacement after 1, 7, 14 and 28 days of curing to describe the hydration process. By using the ternary techniques, there are furthered understanding on how the bottom ash replace into Portland cement effectively.

Experimental part

Materials and method

An experimental procedure was performed to determine the effect of different sizes of bottom ash of curing at 1, 7, 14, and 28 days by 10% replacement of bottom ash into Portland cement. Mix was prepared by partial substitution of 10% BA with 90% of Portland cement. Portland cement used in this study is manufactured by Cement Industries of Malaysia Berhad (CIMA) and under an effective system of testing, control, and monitoring and complies with MS EN 197-1:2007. Whereas bottom ash is purchased from Manjung Coal-Fired Power Station, Lumut, Perak, Malaysia. Before utilization of different sizes bottom ash, additional grinding and sieving into 63, 75, and 150 μm is necessary in order to use as replacement and increase the specific area thereby reduce inter-particle friction while enhancing the chemical reactivity [5]. These BA had almost identical chemical components and only differs in particle size.

Portland cement with 10 wt% of BA was prepared by mixing the original cement powder with different sizes of 63, 75, and 150 μm. The samples of size 50 x 50 x 50 mm^3 were fabricated. All samples will be demolded after 1 day

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Table 1
CHEMICAL COMPOSITION OF PORTLAND CEMENT, BOTTOM ASH, AND BA COMPOSITE CEMENT

Chemical compound	OPC (%)	BA (%)	BA composite cement (%)(63 µm)	BA composite cement (%)(75 µm)	BA composite cement (%)(150 µm)
SiO_2	14.00	38.10	15.60	16.30	16.70
Al_2O_3	3.00	10.90	3.50	3.40	3.10
Fe_2O_3	4.47	20.88	5.89	5.59	5.82
SO_3	3.63	4.61	2.83	2.81	2.81
CaO	71.33	19.50	70.65	70.49	69.75
TiO_2	0.31	1.16	0.34	0.45	0.44
CuO	0.03	0.05	0.04	0.04	0.04

under ambient condition and the hydration process of this cement composite was maintained by soaking in water for 1, 7, 14 and 28 days to investigate the effect of curing. The standard water/cement ratio 0.5 was used. Physical, chemical, and mineralogical characterization was performed to achieve this research goal. One control sample was prepared using 100% Portland cement as references.

Compressive strength was conducted using universal testing machine at 1, 7, 14 and 28 days to determine the strength and 28 days as a standard testing age accordance with ASTM C109 where 3 samples were tested for the accuracy at age of curing time. Compressive strength is the most important design parameter for cement industry. Density of the BA composite cement samples were conducted by using equation 1. The weight of the samples before and after curing in water tank was measured at each sample.

$$\lambda = \frac{M\ after\ curing - M\ before\ curing}{Volume} \qquad (1)$$

Mineralogical investigation XRD was conducted on Portland cement itself and BA cement composite to highlight the presence of hydration of cement phase. Data were recorded with peak position and intensity where determined according to database in reference data. Scanning Electron Microscopy (SEM) resolving the microstructure and topography using fractured pieces of mortar that generated from compressive strength samples.

The characterization of chemical composition was performed using X-ray Fluorescent (XRF) spectroscopy. The chemical composition of different particle sizes of BA cement composite and Portland cement was measured and the result was shown in table 1. According to the ASTM C618 [11], the result in this study shows that it is class C BA as the sum of the $SiO_2 + Al_2O_3 + Fe_2O_3$ is 69.88% and suitable to reuse as cement materials. Class C BA classified as material that has self-cementing properties and high calcium content as well as pozzolanic properties. It can be observed that the addition of BA has small effect on the chemical composition of Portland cement which means that BA cement composite should have similar mineral phase composition [12, 13].

Results and discussions

Compressive strength

Figure 1 shows the different in strength properties of 10wt% BA cement composite containing different sizes of BA with various curing time. Correspondingly, compressive strength of all the samples increased with increasing curing time. The result has a quite similar compressive strength according to BA sizes but the compressive strength of OPC is higher than the BA cement composite. These results agree with relevant research result in the literature [15, 16]. This was due to macroscopic voids and porous structure of BA particles that delay the hydration process of cement in weakening the mechanical strength of BA cement composite [7, 14]. Generally, compressive strength of Portland cement is determining by the size particles. The largest the size of BA may be seen more effect and make the mechanical strength even weaker whereas fine particles could promote hydration efficiently.

The strength of Portland cement was 9.8% higher than 63 µm BA composite cement for day 1 while 6.35% difference attained after 28 days. Anyway, the compressive strength between BA composite cement revealed that the interfacial binding strength between 63µm BA (34.23 MPa) with cement was 2.37 and 2.83% much higher than that between 75 µm BA (33.42 MPa) and 150 µm BA (33.26 MPa) for 28 days which make the finer BA particles have much effective in the mechanical strength. Fineness of particle size could lead to higher effective substitution as finer particle size will facilitate hydration process and ensure the early age strength acquirement and also act filling material to reduce the porosity and voids that presented in

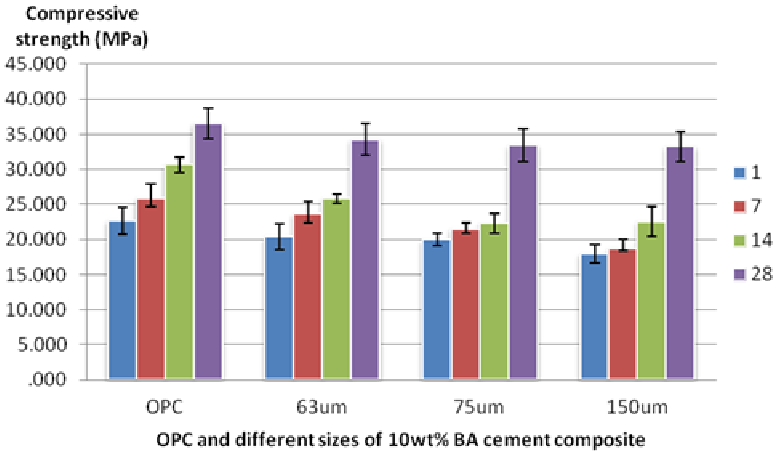

Fig. 1. Compressive strength of 10 wt% BA cement composites with 63, 75, 150 µm at different curing time

the samples. The reduction in strength may be due to lack of water as BA is porous material and thus needs to absorb more water that result in limits the pozzolanic effect of BA cement composite [17].

The fineness of BA might increase the pozzolanic activity of BA in composite cement even though the strength of BA composite cement was lower than 100% OPC but the intension of using various size of bottom ash was prior to minimize the inhomogeneity of the mixes and increase the specific surface area of the material [9]. Therefore, it is very depending on the particle size of bottom ash as the particle size of bottom ash decreases there is an increment on strength. The opinion that increased fineness can improve strength is always right and this might due to several reasons such as pre-hydration problems of fineness or amount of water required for setting. According to Celik [18], increasing of fine particle might result in higher early strength yet may cause problem during setting such as undesirable volume changes.

Density

The data for density in the effect of particle size and curing time of BA cement composite is shown in figure 2. The rough surface of BA have led to difference in density between control sample and BA cement composite samples. The density of BA cement composite increased as the size of bottom ash particle increase from 63μm to 150μm. According to Brown, fine aggregate with high specific area could have high demand of water which results in low density of concrete [18, 19, 20]. In this study, fineness size particle (63μm) contains high specific surface area and expected to consume much quantity of water as BA is known to be porous material. Porosity of BA in Portland cement increases and its required high demand of water during cementation process [21, 22]. Due to high porosity of BA, results on lower density and higher porosity relative to control mortar will achieved. Hence lower value of density is achieved for 63μm (53.33g/cm^3) compared to 75μm (68.67g/cm^3) and 150μm (79.45g/cm^3) as smaller particles with high specific surface area resulted in increased water consumption.

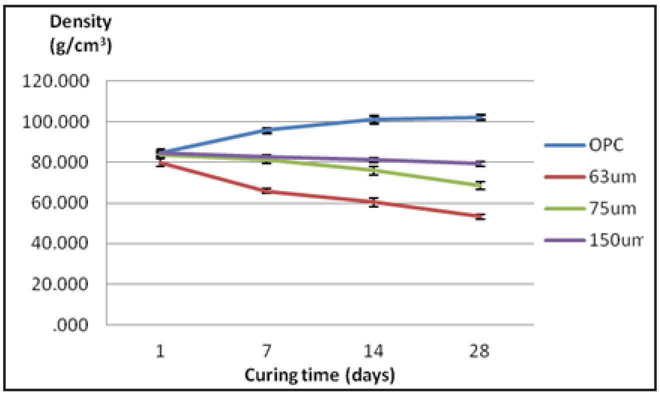

Fig. 2 Density of OPC and 10 wt% BA cement composite with curing time

All the samples require curing in order to promoting the cement hydration for development of strength, durability and other mechanical characteristic. The chemical reaction between cement and water continue as cement remain in wet condition, therefore more hydration products produced in the situation of wet curing and this leads to increase in density [23]. For 100% OPC, the value of density for day 1 was 84.67g/cm^3 and day 28 was 102.45g/cm^3 and this means that curing time does affect the density of BA composite cement where the longer the curing time, the lower value of density for all the sizes of BA. The replacement of BA reflected that the density was declined in values. Due to fine BA is in high demand of water, these fine particles will fill the voids in between cement particles and results in decreased the density of samples. This attributed to the increasing in porosity with BA contents as BA is large particle with non-uniform shape. Besides that, Carrasco also stated in his finding that the lower of density with increment of BA contents can be attributed to low specific gravity and high water absorption rate by BA through the hydration process of Portland cement [24].

Morphology Analysis

The SEM analysis on microstructure was under 1000x and 3000x magnification. As observed in the micrograph, there is reaction between bottom ash with Portland cement and the spherical particle is the bottom ash particles. From the micrograph below, more understanding regarding to the formation of bottom ash in Portland cement through distribution of agglomerate structure.

Figure 3 represents the microstructure of Portland cement mortar before any bottom ash replacement under day 1 (fig. 3a and b) and day 28 (fig. 3c and d). It can be found that there is no empty space exists and phase separation occurs and therefore the phases are combined and interlocking on the boundary layer between each other [25]. The results on 10% replacement of bottom ash in Portland cement mortar which cures in water at room temperature for day 1 and day 28 are shown in figures 4, 5 and 6 respectively. The reacted bottom ash particles increase as the curing time increased regardless influenced of particle size of bottom ash.

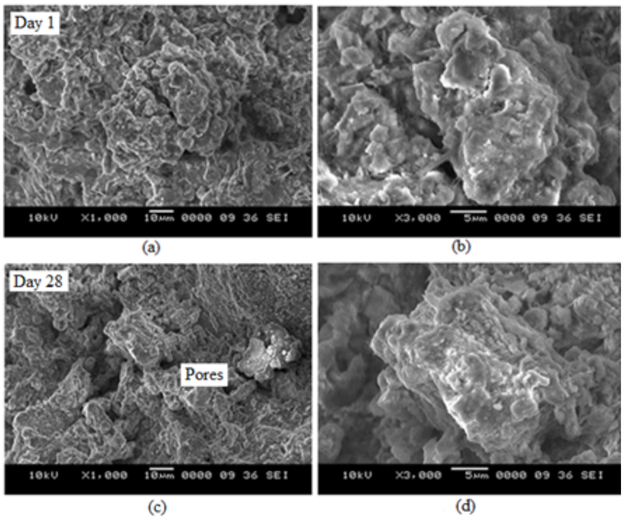

Fig. 3. Portland cement day 1& 28

The microstructure of the BA cement composite for day 1 shows the much appearance of unreacted sphere shape bottom ash and the microstructure of day 28 (figs. 4-6, c and d) become more compacted compared to day 1 (figs. 4-6 a and b). The reacted area of BA cement composite improved as the curing duration increased where attributed to the formation of C-S-H. This type of C-S-H is generally seen in hydration process and it is also believe that the addition of bottom ash lowers the alkalinity of the solution which might cause the delay of hydration process. These C-S-H formations probably change the network structure of bottom ash from irregular to distributed structure. The SEM image depicts the formation of hydration product with nucleation of C-S-H with some of the area with compacted whereas other part develop fibrillar structures of C-S-H and needles indicating different degree of hydration process [26]. The high density of fibrillar structures formation

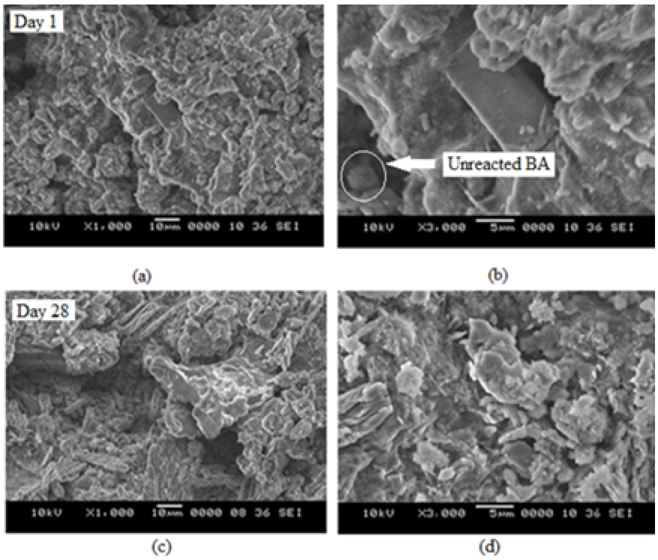

Fig. 4. 4.63µm with 10% Bottom ash for day 1 and 28

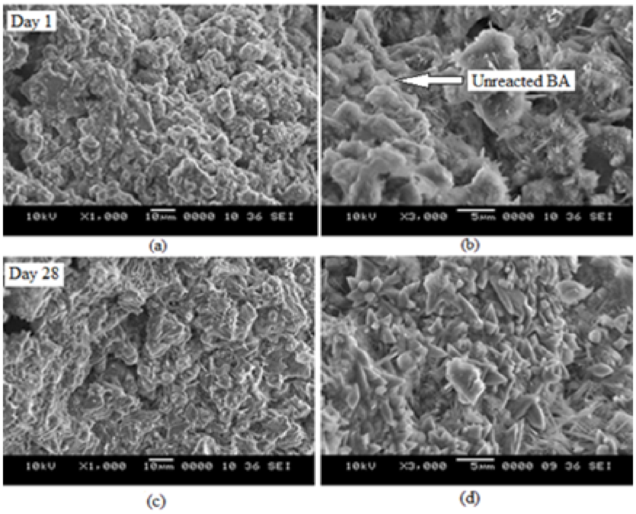

Fig. 5. 75µm with 10% Bottom ash for day 1 and 28

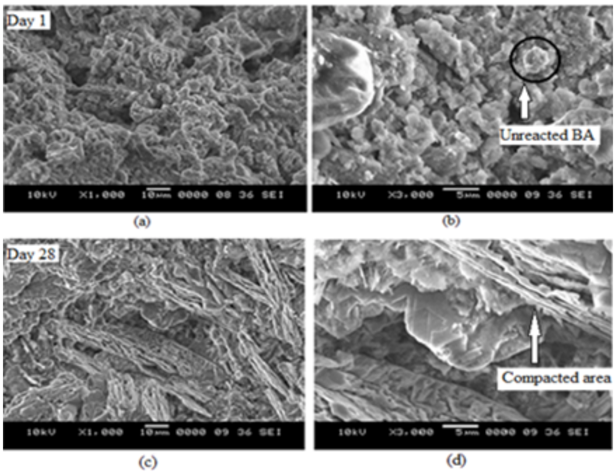

Fig. 6. 150 µm with 10% Bottom ash for day 1 and 28

contributed to the strength development in BA cement composite. The portlandite produced by the hydration of calcium silicates in cement reacts with silica and alumina in the pozzolanic material and produces C-S-H gels which grew into capillary spaces [26- 28].

Generally, the larger particles are more susceptible to breakage than the fineness ones. This may be attributed to the fact that there is a higher probability of finding a flaw of critical size in larger particles than in their smaller counterparts. Larger particles are also more likely to be cracked during the hydration process. Anyway, particle size of bottom ash did not showed any dramatically changes towards the structure in the microstructure. From the micrograph below, more understanding regarding to the formation of bottom ash in Portland cement through distribution of agglomerate structure. However, the differences in the microstructure features between the specimens without BA were not obvious.

Mineralogical Analysis

The mineralogical analysis of 28 days with 10% replacement is shown in figure 7 where both of Portland cement and BA cement composite was differing substantially. BA cement composite samples generated new peak at 2Ÿ angle of 19° which represent crystalline of Portlandite ($Ca(OH)_2$). These showing that BA reacts with Portland cement and form hydration product from pozzolanic reaction of admixtures with $Ca(OH)_2$. 29° represents C_3S and C_2S. The amount of C_3S and C_2S was declined in intensity compared to Portland cement as the hydration of calcium silicate phases of BA composite cement was delay due to slow pozzolanic reaction of BA [29, 30].

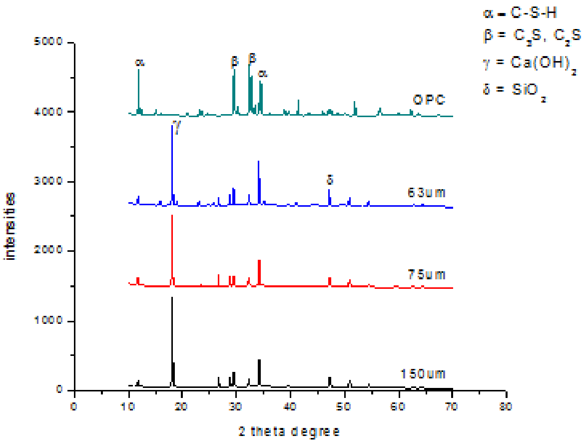

Fig. 7. X- ray diffraction of Portland cement and BA cement composite at several sizes at 28 days with 10% replacement.

Reaction of BA with Portlandite will generate more C-S-H gels where pozzolanic reactions could improve material strength by formation of addition binding phase (C-S-H) which this component is the proved and common phase for hydration products of cement phase. OPC typical contain primarily of tricalcium and dicalcium silicate phase when exposed to water, calcium silicate form calcium hydroxide and calcium silicate hydrate (C-S-H) gel as shown in equation 2. That is source of compressive strength in concrete where 9 and 35° been identified as C-S-H peak where it is more visible in Portland cement [29].

$$C_3S + H_2O \rightarrow Ca(OH)_2 + C\text{-}S\text{-}H \qquad (2)$$

When Portland cement is replaced by a portion of BA, the Portlandite produced reacts with the silica or the silicates in the ash in the presence of water to create products similar to those obtained in the hydration of the cement. These showing that bottom ash react with Portland cement and form hydration compound whereas the rest of the peaks did not show any distinctive differences. As the hydration process continues, crystallinity were increased which indicated that the formation of C-S-H also increased. The replacement of BA into Portland cement had not much effect on the mineralogical phase composition while the major component was almost similar. This clearly implying that

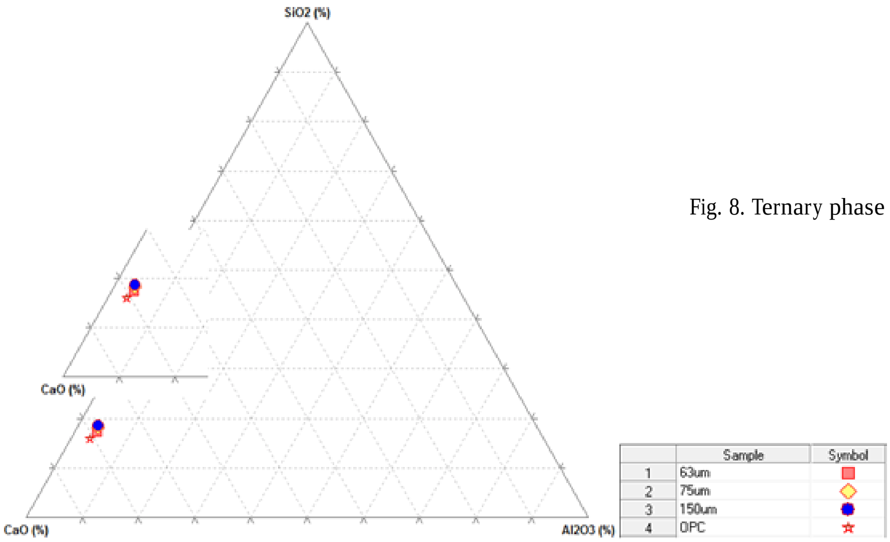

Fig. 8. Ternary phase diagram of OPC-BA for 28 days of curing with 10% replacement

all BA sample suitable to use as alternative material for Portland cement.

Ternary Phase Analysis

Class C bottom ash is pozzolanic material and the primary composition is in siliceous. It does not have any cementitious properties even in the presence of water. However, when a pozzolanic material exposed to water and calcium, it will hydrates and indicate the capability of bottom ash to performs cementitous properties which could enhanced the particle bonding effect [31, 32].

A major concern on this study is the knowledge of the chemical transformation occurring during the hydration process. Portland cement is composed of four primary oxides which are tricalcium silicate (C_3S), dicalcium silicate (C_2S), tricalcium aluminate (C_3A), and tetracalcium aluminiferrite (C_4AF) [27]. During the hydration process, C_3S and C_2S react with water to form calcium-silicate-hydrate (C-S-H) gels and calcium hydroxide where these gels provide the strength to mortars or concretes [33, 34, 35]. Anyway, the hydration products from four oxides is $Ca(OH)_2$ will react with silica of bottom ash and form more C-S-H. In this study, the addition of BA increased the amount of SiO_2 in producing calcium silicate hydrate ($(CaO)x.(SiO_2)y.(H_2O)z$, C-S-H) but the pozzolanic effect of bottom ash also promoting the consumption of calcium hydroxide ($Ca(OH)_2$,CH) for the formation of C-S-H [2, 30].

The ternary phase diagram of CaO-Al_2O_3-SiO_2 for OPC-BA and typical composition range of OPC of 28 days of curing is shown in Figure 8. In this ternary phase diagram, BA cement composite is analogy with the common pozzolanic material which similar to Portland cement. For all the sizes of BA cement composite, they were very close to calcium zone and shifted to CaO side. CaO consider as the most prominent constituent in hybrid cement [36, 37]. This indicated that the BA cement composite has high calcium oxide content in pure system of cementitous materials where high CaO content activated the consequent production of C-S-H compound. This phase is present as active constituents of Portland cement and usually contributes to the mechanical properties. Besides that, an increase in particle size of BA affected the degree of hydration by allowing more free water in the gel pores which indicated that BA may not have completely reacted.

The addition of BA causes lowering in Ca contents and hydration product which also decreased the rate of hydration. This situation indicated as cement dilution where could be explained from the dilution effect that origin by BA [38]. In this case, the C-S-H is expected to produce in small amount and unable to exhibit the properties as Portland cement. This result indicated that the cementitious activity of BA is lower and its addition to cement leads some retardation of cement hydration.

Conclusions

In this paper, it provides an insight into the effective replacement of BA into Portland cement at different stages of curing. From the result presented, it can be concluded that the pozzolanic properties displayed by BA were very weak and always lower than Portland cement in all the samples regardless of curing time. Our findings provide a simple yet effective way for BA to replace into Portland cement as well as achieving a comparable compressive strength with Portland cement. As the amount of BA increased, the compressive strength decreased. The optimum compressive strength (34.23 MPa) was achieved by 63 ìm BA with 10% replacement in 28 days. Although only small amount of BA can be added into Portland cement but the reuse can be an ultimate solution for disposal and important on solving solution of landfilling problems where also reduce the cost of the construction purposes.

The density of the control was much better than BA cement composite where the rough surface of BA leads to difference in density. The density increased as the size of bottom ash particle increase with the density of 53.33g/cm^3 (63 µm) increased to 68.67 g/cm^3 (75 µm) and 79.45 g/cm^3 (150 µm). The fineness particles size contains high specific surface area that resulted in lower density as BA is porous material which consume high quantity of water.

The day 1 microstructure of the BA cement composite shows much appearance of unreacted sphere shape bottom ash whereas the microstructure of day 28 become more compacted. The reacted bottom ash particles increase as the curing time increased regardless influenced of particle size of bottom ash. The reacted area of BA cement composite improved as the curing duration increased where attributed to the formation of C-S-H.

The mineralogical analysis of BA cement composite showing that BA reacts with Portland cement and form hydration product from pozzolanic reaction of admixtures with $Ca(OH)_2$. Reaction of BA with Portlandite generate C-S-H gels that could improve material strength by formation of addition binding phase which this component is the proved and common phase for hydration products of cement phase.

For phase analysis, BA cement composites were shifted to CaO side where this consider as the most prominent constituent in hybrid cement. This phase is present as active constituents of Portland cement and usually contributes to the mechanical properties. The addition of BA causes lowering in Ca contents and also decreased the rate of hydration. This dilution effect indicated that the cementitious activity of BA is lower and its addition to cement leads some retardation of cement hydration.

Our findings proved a simple way to recycle BA and reduced substantial amount of pollution in environment standpoint where the interface between BA and OPC achieved the requirement of compressive strength without compromising the quality of final product. A better scientific understanding and significant future research is needed especially on the behavior of pozzolanic reaction.

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