Effect of Soaking Period on High CaO Fly Ash Properties for Utilization in Cement Application

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Abstract — This work reports a study of fly ash with CaO higher 20%, emphasizing on the effect of soaking condition on fly ash properties. Properties of soaked fly ashes after drying have been characterized in comparison to those of the fresh unsoaked fly ash. Those properties include physical, chemical, morphological and mineralogical properties, and specification in portland-cement concrete. Morphologies of the soaked fly ash remains unchanged, the spherical shape. Phases of both soaked and unsoaked fly ashes have been studied, consisting of anhydrite, quartz, magnetite, hematite, lime, portlandite, and calcite. The strength activity indices at 7 and 28 days of dried fly ashes after soaking are higher than 75% of cement mortar and higher than that of the fresh fly ash. The achieved result has demonstrated important evidence that fly ash after experiencing wet condition and undergoing a particular recovery process could be reused and utilized in their application of cement.

Keywords — High CaO fly ash, Unsoaked fly ash, Soaked fly ash, Properties, Strength activity index

I. Introduction

COAL is the most widely used source to generate electricity. Its combustion produces fly ash that is one of the by-product of combustion [1]-[5]. Fly ash is a pozzolanic and cementitious material in the presence of lime (CaO or $Ca(OH)_2$). Upon the chemical composition, fly ash has been classified into two classes; class F and class C, according to ASTM C 618 [2], [6], [7]. For example, fly ash with CaO greater than 20% is classified as class C [1], [6]-[8]. Such high CaO, class C fly ash has been used in concrete industry because it modifies the properties of concrete in both fresh and hardened states. Those improved properties are such as workability, cohesiveness, early strength, drying shrinkage, temperature rising, pumpability and abrasion resistance [1], [2], [9]-[23]. The high CaO fly ash further affects mechanical properties in terms of expansion and durability of concrete, also sulfate resistances due to high free lime and sulfur content [5], [23]-[28].

Pozzolanic reaction between portland cement with fly ash requires $Ca(OH)_2$, a result from hydration reaction of portland cement, to react with pozzolanic materials that eventually form strength-producing products [6], [17], [29]. The low calcium fly ash is widely used to replace cement in concrete [30], [31] while diversely the high CaO fly ash is known to rapidly harden when mixing with water, practically impossible to be used as a pozzolan [3], [32]-[34]. Thereby, drying and grinding processes are essentially required for such hardened material before any further use. To this point of view, the high CaO fly ash is believed to be barely reused as a pozzolan as it experienced wet condition. Research concerning the change in properties of high calcium fly ash, for the regime of higher than 20%, in the condition of water mixing, to our knowledge, yet reported. Recently, Saengsoy [34] discussed the effect of moisture content and exposure period of wet fly ash on basic properties of mortar, explaining that preexistence of water in wet fly ash was believed to affect the properties of mortar by improving flow of mortar and strength activity. Nonetheless, this work was carried out using the low CaO of about 2 %.

Thus, this work presents a study on the effect of water on high CaO fly ash, CaO is higher than 20%. Effect of soaking period on fly ash properties was investigated; different soaking periods were simulated. After recovering, the soaked fly ash samples were tested their physical and chemical properties. When use as a cementitious material in concrete, the specification in portland-cement concrete was also evaluated to give insight into the terms of reuse possibility. These results could provide useful information for the commercial merit.

II. Materials and Experimental Methods

A. Sample Collections and Preparations

This study used high-calcium fly ash (Class C), higher than 20 % CaO. The high CaO fly ash was set to soak in water at different times: 4, 24, 48, and 72 hours. The fly ash samples soaked at 4, 24, 48, and 72 h soaking periods are herein called FA-4H, FA-24H, FA-48H, and FA-72H, respectively. While the fresh fly ash is called F-FA. Ratio of fly ash per distilled water was kept constant at 1:10. Then the samples were collected and oven-dried at 100-105 °C [35], [36] following by ball milling at 336 rpm for 4 hours. For the fresh fly ash after drying and ball milling, these conditions were labeled as FA-BM. The fresh and the soaked samples were analyzed their

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characteristic properties including physical properties, chemical properties, and specification in portland cement concrete. The characterization testing methods were carried out by applying ASTM standard specification for coal fly ash and raw or calcined natural pozzolan for use in concrete (C 618) [7].

B. Physical Properties

Density, particle size distribution, morphological, and elemental of all fly ash samples were determined by ultrapycnometer, laser particle size analyzer, scanning electron microscope (SEM), and Energy Dispersive X-ray Spectroscopy (EDS), respectively.

C. Chemical Properties

Moisture content percentage was investigated by weight loss method. For a given drying temperature at about 105 to 110°C [37], the weight of samples after drying was monitored comparing to the weight before drying. For pH measurement, the fly ash samples were mixed with distilled water in a 1:10 mass ratio, then stirred for 1 min and measured the pH value. Chemical and mineralogical compositions of the samples were determined by X-ray fluorescence (XRF) and X-ray diffraction (XRD), respectively.

D. Specification in Portland-Cement Concrete

Mortars were prepared with a sand to binder ratio of 2.75 by mass, with a 20 wt.% fly ash. This work kept the flow of mortars under control at 110±5, as water to binder (w/b) was varied and optimized to achieve the flow requirement. Then water requirement was calculated according to the ASTM Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete (C 311) [37]. The mortar samples are cured in water for 7 and 28 days prior to the compressive strength testing, as the strength activity index was calculated accordingly by applying ASTM C 311.

III. Result And Discussion

A. Physical Properties

The density of fly ash samples are shown in Table I. These values are in agreement with those reported by Naik and Singh [2], mentioning the density of class C fly ash generally falling in the range of 2.4 to 2.8 g/cc. Soaking in water show no effect on the density change of samples; densities of the soaked samples are insignificant different than density of the unsoaked sample.

TABLE I
Density and Particle size Of Fly Ashes Samples From Different Conditions

	Sample					
	F-FA	FA-BM	FA-4H	FA-24H	FA-48H	FA-72H
Density (g/cc)	2.63	2.60	2.61	2.63	2.64	2.64
Particle size (D[4,3]µm)	27.94	18.31	19.32	20.63	19.34	20.70

In Table I, particle size of FA-BM is obviously smaller the F-FA, confirming that ball milling is adequately effective to grind fly ash particles into the significantly reduced size. Particle size distribution (PSD) plots of those fly ash samples, both fresh and soaked conditions, are demonstrated in Fig.1

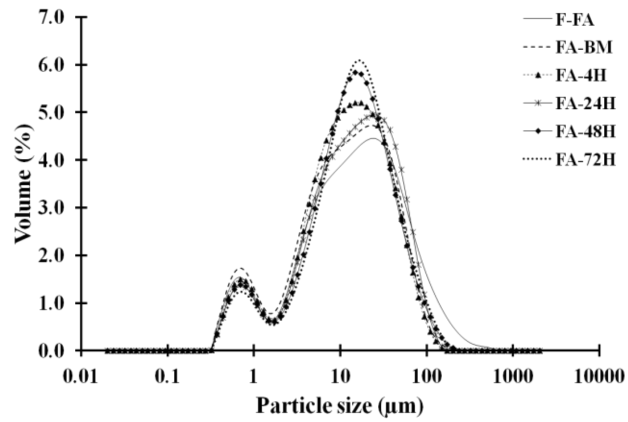

Fig. 1 Particle size distribution of fly ashes samples from different conditions

Fig. 2 shows SEM micrographs of the F-FA, FA-BM, FA-4H, FA-24H, FA-48H, and FA-72H. The typical characteristic of fly ash from all conditions was observed to be mainly a spherical shape. Comparing the fresh fly ash with and without ball milling, surface of the fly ash sample after ball milling became slightly rougher. While surface of the soaked fly ashes was found to be randomly covered with aggregates, confirmed by the EDS result that contain high calcium content. The aggregates could possibly be from fracture fragments of fly ash particles themselves, fibrous matrix of crystalline calcite, and crystalline portlandite transformation, which is expressed by these following Reactions [29].

$$CaO + H_2O \longrightarrow Ca(OH)_2 \quad (1)$$

$$Ca(OH)_2 + CO_2 \longrightarrow CaCO_3 \quad (2)$$

Beside the spherical shape, fly ash with different shapes and types could be found; e.g. hollow sphere, glassy particle, magnetic iron containing spherical particle, and irregular shape of carbon from incomplete combustion mixed with fly ash [17], [38]. This is typical for fly ash known as a heterogeneous mixture, composing particles of various types, shapes, sizes and chemical compositions [2].

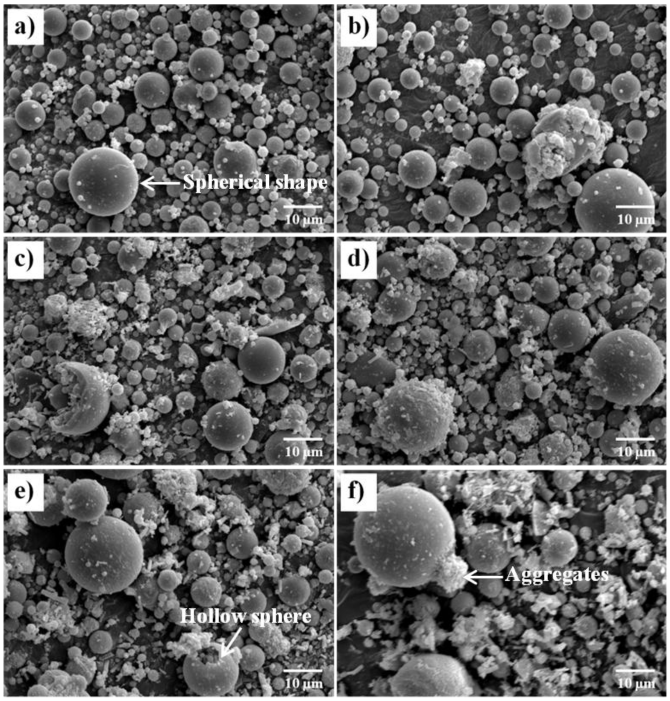

Fig. 2 SEM micrographs of a), b) are F-FA, FA-BM and c), d), e), f) are FA-4H, FA-24H, FA-48H, FA-72H

B. Chemical Properties

Chemical composition of fly ash samples was characterized by X-ray fluorescent. The results of chemical compositions of those samples are presented in Table II. Comparing the unsoaked and the soaked samples, the major compositions of SiO_2, Al_2O_3, Fe_2O_3, and CaO are in the close range of 29.4-30.1%, 15.8-16.3%, 15.6-16.4%, and 22.4-23.5%, respectively. The sum of these three main oxides including SiO_2, Al_2O_3, and Fe_2O_3 remains higher than 50%, meeting the class C fly ash standard. The SO_3 content was in the range of 7.5-8.4%, higher than the 5% standard as depicted by ASTM C618 requirement [7]. McCarthy et.al [39] mentioned that sulfur content could possibly increase in parallel with the increase in CaO content. This is attributed to, first, the reactions occurring between calcium and sulfur in the flue gases of the furnace, and second, the nature of coal resource. Moisture content was found to slightly vary, from 0.01 to 0.35%. While the approximate pH of 12 was obtained for all conditions, which is a characteristic result of class C fly ash produced from lignite coal of high-alkali performance [40].

Mineralogical study was carried out by X-ray diffraction (XRD). The phases of oxide components are demonstrated in Fig. 3. The major phases of unsoaked and soaked fly ash were identified as anhydrite ($CaSO_4$), quartz (SiO_2), lime (CaO), magnetite (Fe_3O_4), mullite ($3Al_2O_3 \cdot 2SiO_2$), hematite (Fe_2O_3), calcite ($CaCO_3$), and portlandite ($Ca(OH)_2$). Anhydrite, quartz, and lime are the main crystalline mineral phases existing in the class C fly ash [1], [2], [39]. Considering the lime phase at 2θ=37.346, the peak intensity for the soaked samples is much lower than that of the fresh sample, as the peak almost disappeared for the prolonged soaking period conditions. This could possibly be due to the oxide disappearing after reacting with water.

TABLE II
CHEMICAL COMPOSITION OF FLY ASHES SAMPLES FROM DIFFERENT CONDITIONS

wt.%	Sample					
	F-FA	FA-BM	FA-4H	FA-24H	FA-48H	FA-72H
SiO_2	29.46	30.09	29.73	30.07	29.32	29.4
Al_2O_3	15.91	16.3	16.1	16.28	15.82	15.78
Fe_2O_3	15.83	15.61	15.9	15.84	16.38	16.33
CaO	22.68	22.37	23.22	22.91	23.2	23.47
SO_3	8.4	7.95	7.62	7.45	7.89	7.68
K_2O	2.14	2.17	2.08	2.11	2.14	2.14
Na_2O	1.78	1.71	1.48	1.51	1.4	1.37
MgO	2.47	2.49	2.51	2.52	2.48	2.44
TiO_2	0.45	0.45	0.46	0.44	0.45	0.46
P_2O_5	0.28	0.27	0.3	0.28	0.31	0.29

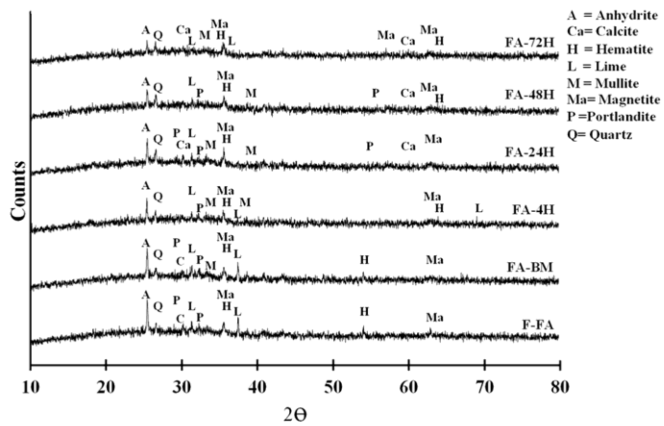

Fig. 3 X-ray diffractions pattern of fly ashes samples from different conditions

C. Specification in Portland-Cement Concrete

Fig. 4 presented percent of water requirement and flow table test of samples at different water to binder ratio (w/b). In this research, flow test was controlled at 110 ± 5; hence the obtained w/b of cement, F-FA, FA-BM, FA-4H, FA-24H, FA-48H, and FA-72H are 0.52, 0.50, 0.50, 0.505, 0.505, 0.51, and 0.52, respectively. As a result, the water requirement for F-FA, FA-BM, FA-4H, FA-24H, FA-48H, and FA-72H are 96.15, 96.15, 97.12, 97.12, 98.08, and 100 % of cement (100%) regarding the ASTM C618 requirement, as the max. % water requirement is 105. Both the fresh fly ash and the soaked fly ash samples required the lower amount of water comparing to cement. While the soaked samples required slightly higher % water uptake than the unsoaked sample.

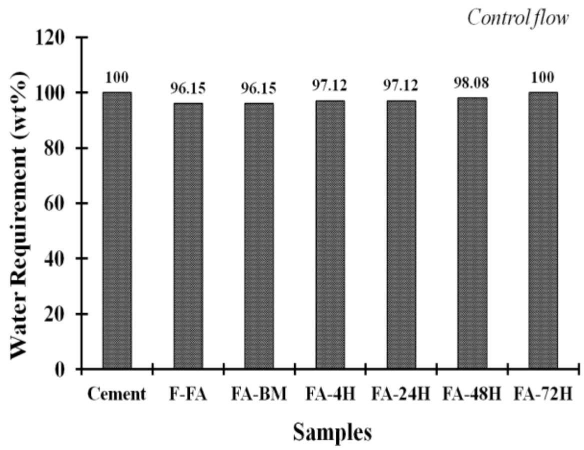

Fig. 4 Water requirement (wt.%) of fly ashes samples from different conditions at a control flow of 110±5

Fig. 5 shows strenth activity indices of mortars containing F-FA and the soaked samples. The strength activity indices at 7 and 28 days of soaked fly ash mortars are higher than that of the fresh fly ash mortars. The strength activity indices of all samples are higher than 75 % of cement according to ASTM C618 requirement, and locate in the close regime nearly the strength activity index of cement. The strength activity index of FA-BM sample is higher than that of the F-FA sample, indicating that fineness parameter has become a predominant role governing the compressive strength [41]. It has been believed that strength activity index of high CaO fly ash increases at early age of mortar due to the effect of higher amount of cementitious materials facilitating the pozzolanic reactions; the large amount of CaO could rapidly react with water then yielding $Ca(OH)_2$ at early period of mixing [3], [6], [29]. For the soaked conditions of high CaO fly ash, a certain amount of cementitious materials might have been produced while soaking, thus further enhancing the strength activity index of the soaked fly ash samples accordingly.

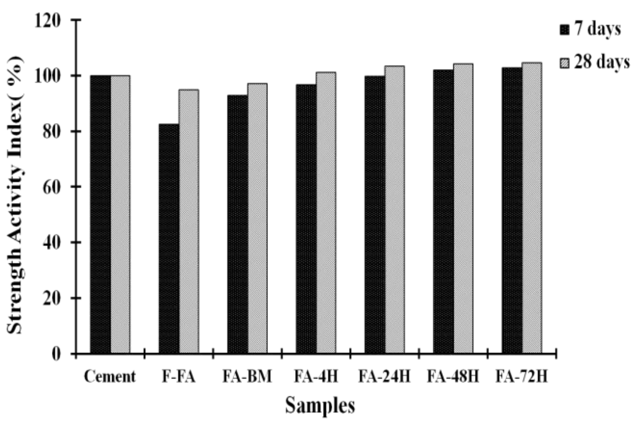

Fig. 5 Strength activity index of fly ashes samples from different conditions at 7 and 28 days

IV. Conclusion

Physical properties of the fresh and the soaked fly ash samples were investigated. Density and particle size distribution of the soaked fly ashes showed insignificant different with respect to the fresh fly ash. Morphology of fly ashes were found to be spherical shape, as surface of the soaked fly ashes became rougher than that of the fresh one due to aggregates from crystalline CaO. Chemical compositions of soaked fly ashes were characterized. The result indicated that the CaO content increased in parallel with the SO_3 content. Mineralogical study of all fly ash conditions showed the major phases of anhydrite, quartz, magnetite, hematite and lime. The lime intensity of the soaked fly ashes was found to be smaller than that of the unsoaked fly ash because of the phase change upon the reaction with water. In part of specification in portland cement concrete, strength activity index of the soaked fly ashes was found to be higher than that of the unsoaked fly ash, and the values of both conditions are more than 75% of cement at 7 days. For the 28-day conditions, the strength activity index for those samples showed the result in the nearly range. In addition, fineness showed its effective on improving the compressive strength.

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