High-calcium fly ash recovery from wet-stored condition and its properties

Phattarathicha Tepsri, Angkana Chumphu and Sorachon Yoriya
National Metal and Materials Technology Center (MTEC), National Science and Technology Development Agency (NSTDA), 114 Thailand Science Park, Pahonyothin Rd., Khlong 1, Khlong Luang, Pathum Thani 12120, Thailand
E-mail: sorachy@mtecc.or.th

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Abstract
Fly ash can undergo changes in its properties during the wet-stored conditions, leading to reactivity variability consequently affecting its potential applications. Evaluation of the material properties is considerably essential. This work reports on an investigation of changes in physical and chemical properties of high calcium fly ash after recovering from the wet-stored conditions. Herein the high calcium lignite fly ash with calcium oxide constituent in a range of 20%–30% is the focus of this study. Important characteristics of fly ash have been studied, including physical properties, morphologies, chemical composition, and crystallographic phase transformation. Relation of oxide composition including silica, alumina, ferric oxide, and sulfur trioxide, to wet-storing period has been established to visualize the effect of short-term storage in water on changes in the content of mineral bearing species in fly ash. The strength activity indices of the recovered fly ash samples cured for 7 and 28 days has been presented. The significant amount of free lime and soluble calcium, aluminum, and sulfur-bearing minerals varying with storage times are believed to play their pronounced role governing compressive strength.

1. Introduction
Fly ash, one of the by-products from burning coal in the coal-fired power plant [1–5], is known as a raw material that has been extensively used in many applications such as Portland cement [6–9], geopolymer [10–12], asphalt concrete [13–16], and catalyst [17–22]. Particularly in cement and concrete applications, fly ash with unique characteristics has been found to improve workability [6–9], mechanical strength [10, 11, 23, 24], cohesiveness [6–9, 25], and shrink-swell phenomenon [26, 27]. Adding fly ash in concrete mixture could reduce permeability [6–9, 28], temperature rising [6, 23, 29, 30], heat of hydration [23, 29, 30], need of drying agent [6, 7, 31], bleeding [6, 7, 32], and abrasion resistance [6, 7, 9, 10]. Fly ash is pozzolanic material consisting alumino-silicate glasses with varying amounts of mullite, crystalline quartz, hematite, and magnetite. Physical and chemical properties of fly ash are basically dependent upon the coal source that consequently determines the oxide composition. ASTM C618 categorizes fly ash into two classes – class F and class C, typically depending upon their coal source and the oxide composition. Most fly ashes contain a major constituent of silica (SiO$_2$), alumina (Al$_2$O$_3$), and ferric oxide (Fe$_2$O$_3$), as one with this composition in the range of 50%–70% is classified as class C, while another with the composition more than 70% is class F [1, 33–35]. Further, high-calcium class C fly ash typically contains a significant amount of calcium oxide (CaO) up to 40%, relatively higher than the low-calcium class F fly ash, which is generally found to contain less than 10% CaO content.

Fly ash properties determine the chemical reactions; the high-calcium class C fly ash possesses predominant cementitious property and participates in pozzolanic reaction in the hydration process. The reactivity of silica in the hydration of fly ash-cement blends was found to hold a predominant role forming additional cementitious compounds with binding properties [36]. The increase in fineness (% retained on sieve #325) reflecting the reactivity of fly ash was found to improve the strength of mortar [36–38]. The calcium-bearing glass by its nature in the high-calcium (>20%) class C fly ash was found to be more reactive to water than that in the low-calcium
(<8%) class F fly ash [35]. The calcium content has been found as an indicator how fly ash to behave in concrete, while the other compounds such as sulfur trioxide (SO₃), alkali like sodium oxide (Na₂O) and potassium oxide (K₂O) could also affect the performance of fly ash [39]. In concrete, variation in the ash composition is significance to affect the hydration behavior and the performance of this cementitious material in the mixtures. The high CaO fly ash is known to rapidly harden when mixing with water, practically rather difficult to be reused as a pozzolan after experiencing wet condition. The fly ash after weathering condition has resulted in changes in physical, chemical, and mineralogical properties in terms of the formation of secondary mineral phases and reduction of soluble salt content [40]. Reusing the low CaO fly ash seems to be possibly practical that preexistence of water in fly ash after experiencing wet conditions was found to improve the properties of mortar as mixing the 2% CaO containing fly ash with cement [41]. To our knowledge, material properties and composition of fly ash with relatively higher calcium influencing its performance in concrete has been rarely reported.

This work investigate changes in high calcium fly ash properties after recovering from wet-stored conditions. With a focus on the 20%–31% CaO constituent, this study has employed class C fly ash produced from Mae Moh power plant, Thailand. This power plant generates over 3 million tons of lignite fly ash in these recent years, as two million tons are beneficiated in concrete application and the rest is disposed mostly in wet disposal method. In practice the storage period for the unused fly ash can range from few days occasionally by mixing with water in the temporary storage area to months or decades in the permanent disposal area. Nonetheless, with the currently large amount of unused fly ash and the limit of dumping area concerned, the idea of utilization of the short-term dumped fly ash has become the focus of the power plant. Since there is no adequately evidential support for the temporary dumped fly ash on feasibility to reuse or utilize it, associated with the change in pozzolanic properties still in doubt. Understanding the changes in physical, chemical, and morphological properties of fly ash is considerably needed in evaluating the recovery process for fly ash after experiencing short-termed wet storage. Those properties and the phase transformation of fly ash have been brought in relation to the mechanical compressive strength of concrete via the study of specification in Portland-cement concrete as a function of wet-storing period. This work aims to further provide an additional merit in beneficial use of fly ash in concrete application.

2. Materials and methods

Three types of class C fly ashes with calcium oxide (CaO) ranging between 20%–31% were collected from Mae Moh thermal power plant, Thailand. Those samples with different CaO contents were named herein as FA₁, FA₂, and FA₃. The fresh or non-soaked fly ash is to be referred as F-FA, and the chemical composition of those samples is shown in table 2. The wet-stored condition was performed by mixing fly ash and distilled water in a fixed ratio of 1:10, then stirring for one minute, and soaking at different periods, which are 4, 24, 48 and 72 h, hereafter to be referred as 4H, 24H, 48H, and 72H, respectively. After soaking, the float part and water were drained out, whereas the sink portion was collected and oven-dried at 105 °C [42, 43] for seven hours. Then moisture content was examined by weight loss method. Those samples after drying were further examined for their characteristic properties including physical properties, chemical composition, and specification in Portland cement concrete.

The physical properties were investigated by laser particle size analyzer (Malvern Mastersizer 2000) for particle size distribution and ultrapycometer (Ultrapycometer 1200e) in helium atmosphere for density. The morphology and elemental analysis were characterized by scanning electron microscope (SEM) and Energy Dispersive x-ray Spectroscopy (EDX; Hitachi & Edax S-3400N). The oxide composition were analyzed by x-ray fluorescence spectrometer (Philips PW 2404) with Rh source. The samples were run twice and a set of the results have been selected to report in table 2 and figure 6; the max. standard deviation found for those oxides is 0.07. The mineral phases and oxide composition were analyzed by x-ray diffraction (X’Pert Pro) and x-ray fluorescence (PANALytical PW2404), respectively. For specification in portland cement concrete, mortars were prepared using sand to binder ratio of 2.75 by weight, as binder is a mixture of cement and a fixed 20 wt% fly ash. This study kept the flow of mortar under a percent of control at 110 ± 5, as water to binder (w/c) ratio was optimized to achieve this flow requirement. The standard flow test was performed using a standard conical frustum shape of mortar with a diameter of four directions. The mortar specimens were placed on a flow table and dropped 25 times within 15 s, with the test procedure following ASTM C230. 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 (ASTM C311) [44].

\[
\text{Water requirement, percentage of control } = (X/Y) \times 100
\]
Where X is water required (ml) for the test mixture of fly ash to be 110 ± 5 of control flow, and Y is water required (ml) of cement for each condition. The size of mortar specimen was kept constant at 50 mm × 50 mm × 50 mm. The specimens were placed in moist closet for 24 h prior to curing in water for 7 and 28 days before the compressive strength testing (ADR-Auto V2.0 250/25). The strength activity index of Portland cement prepared regarding ASTM C 311 was calculated by this expression.

\[
\text{Strength activity index with Portland cement} = \left( \frac{A}{B} \right) \times 100
\]

Where A is average compressive strength of test mixture fly ash cubes (MPa), and B is average compressive strength of cement control mix cubes (MPa).

3. Results and discussion

3.1. Physical properties

The chemical and mineral compositions in fly ash varies depending upon the type of coal used in the power station, yielding difference in color of fly ash. The fresh fly ash exhibits the reddish brown color, which is a typical characteristic of class C fly ash containing relatively high iron oxide content [34]. All fly ash samples after being stored in wet conditions from 4 h to 72 h have changed their color from reddish brown to pale brown; with prolonged soaking period, the soaked samples have become pale brown as seen in figure 1. After oven drying, the moisture content of all samples were found to be less than 0.4% regarding the method stated in ASTM C618.

The results of density and particle size in volume mean diameter D [3, 4] are shown in table 1. Density of the fresh fly ash samples are 2.86 g/cc for FA1, 2.63 g/cc for FA2, 2.88 g/cc for FA3. Typically reported, density of class C fly ash lies in the range between 2.4 to 2.8 g/cc [2]. The prolonged period of wet storing up to 72 h has shown insignificant effect on the density of the samples, as apparently the density measured for both before and after wet-stored samples lies in the close range. The particle size distribution curves are shown in figure 2.
Table 1. Density and particle size of FA₁, FA₂, and FA₃ samples both before and after storing in wet conditions at 4, 24, 48, and 72 h.

| Condition | FA₁ | FA₂ | FA₃ |
|-----------|-----|-----|-----|
| Density (g/cc) | 2.86 ± 0.006 | 2.87 ± 0.009 | 2.88 ± 0.011 |
| Particle size (D [3, 4], μm) | 15.43 ± 2.28 | 13.51 ± 0.71 | 14.07 ± 0.18 |
| Density (g/cc) | 2.63 ± 0.003 | 2.61 ± 0.007 | 2.64 ± 0.011 |
| Particle size (D [3, 4], μm) | 27.94 ± 2.11 | 19.32 ± 2.60 | 19.34 ± 0.73 |
| Density (g/cc) | 2.88 ± 0.009 | 2.91 ± 0.004 | 2.99 ± 0.004 |
| Particle size (D [3, 4], μm) | 12.38 ± 0.59 | 15.33 ± 0.57 | 15.95 ± 2.65 |

Figure 2. Particle size distribution of (a) FA₁, (b) FA₂, and (c) FA₃ samples both before and after recovering from different wet-stored conditions at 4, 24, 48, and 72 h.
3.2. Morphology

Figure 3 show SEM micrographs of the fresh fly ash samples. The particles of fly ash samples are typically in spherical shape, with the size varying from less than one micron to more than one hundred microns. Precipitates on the particle surface could be observed for fly ash after experiencing the wet storage; see figure 4. Those precipitates were found to be in the forms of fibrous matrix and aggregates with multi-dimensional shape, probably a result of crystalline calcite (CaCO₃) and portlandite transformation; the chemical reactions are expressed as follows [45–47].

\[
\begin{align*}
\text{CaO} + \text{H}_2\text{O} & \rightarrow \text{Ca(OH)}_2 \\
\text{Ca(OH)}_2 + \text{CO}_2 & \rightarrow \text{CaCO}_3
\end{align*}
\]

In addition, some hollow spheres and irregular shaped carbon from incomplete combustion and magnetic iron particles were also found in the heterogeneous mixture fly ash, which typically contains these characteristic types of contaminant of various size, shape, and chemical composition [48–51]. The morphologies of such hollow spheres are displayed in figure 5. In addition, the solutions produced from all storage conditions have pH higher than 12 after fly ash samples have come in contact with water. It has been believed that such high pH solution contains significant amounts of soluble species like calcium, aluminium, and sulfur ions, thereby contributing their substantial source for ettringite formation [52].

3.3. Chemical composition

Chemical composition of the wet-stored fly ash samples was determined by x-ray fluorescence (XRF) spectrometry; those oxide compositions are listed in table 2. Considering the composition of all conditions of sample, the major oxide components were found to include SiO₂, Al₂O₃, and Fe₂O₃. The sum of these oxides for all conditions lies in the range between 50%–70%, confirming their characteristic composition for class C fly ash according to the ASTM C618 requirement as the area highlighted in figure 6. From table 2, calcium oxide (CaO) is significant in content, while the other components found are sulfur trioxide (SO₃), potassium oxide (K₂O), sodium oxide (Na₂O) and magnesium oxide (MgO), also with a trace amount of titanium oxide (TiO₂) and phosphorus oxide (P₂O₅). The chemical compositions of fly ash mainly depend on the chemical composition of the coal [53]. In lignite coal, the composition of SiO₂, Al₂O₃, Fe₂O₃, and CaO is in the range of 15%–45%, 20%–25%, 4%–15%, and 15%–40%, respectively.
Figure 5. SEM micrographs showing (a) cross-sectional wall thickness and (b) precipitates on the surface of hollow spheres found mixing with FA3 sample after soaking in water for 48 h.

Table 2. Chemical composition of FA1, FA2, and FA3 samples both before and after storing in wet conditions at 4, 24, 48, and 72 h.

| Condition | SiO2 (%) | Al2O3 (%) | Fe2O3 (%) | CaO (%) | SO3 (%) | K2O (%) | Na2O (%) | MgO (%) | TiO2 (%) | P2O5 (%) |
|-----------|----------|-----------|-----------|---------|---------|---------|----------|---------|----------|----------|
| FA1       | 30.57    | 17.32     | 15.98     | 23.16   | 5.45    | 2.16    | 1.55     | 2.58    | 0.44     | 0.24     |
| FA2       | 29.46    | 15.91     | 15.83     | 22.68   | 8.40    | 2.14    | 1.78     | 2.47    | 0.45     | 0.28     |
| FA3       | 22.78    | 11.09     | 15.89     | 31.11   | 11.09   | 1.89    | 2.23     | 2.54    | 0.36     | 0.38     |
| FA1       | 31.05    | 17.76     | 16.21     | 22.59   | 5.02    | 2.1     | 1.35     | 2.6     | 0.45     | 0.28     |
| FA2       | 29.73    | 16.10     | 15.90     | 23.22   | 7.62    | 2.08    | 1.48     | 2.51    | 0.46     | 0.30     |
| FA3       | 23.53    | 12.18     | 15.86     | 31.45   | 9.64    | 1.66    | 1.66     | 2.66    | 0.36     | 0.41     |
| FA1       | 30.64    | 17.47     | 16.5      | 22.68   | 5.23    | 2.22    | 1.37     | 2.54    | 0.46     | 0.26     |
| FA2       | 30.07    | 16.28     | 15.84     | 22.91   | 7.45    | 2.11    | 1.51     | 2.52    | 0.44     | 0.28     |
| FA3       | 21.73    | 11.11     | 16.87     | 32.27   | 10.91   | 1.67    | 1.54     | 2.51    | 0.35     | 0.40     |
| FA1       | 30.83    | 17.55     | 16.39     | 22.67   | 5.24    | 2.17    | 1.3      | 2.50    | 0.45     | 0.25     |
| FA2       | 29.32    | 15.82     | 16.38     | 23.20   | 7.89    | 2.14    | 1.40     | 2.48    | 0.45     | 0.31     |
| FA3       | 22.69    | 11.97     | 16.34     | 31.70   | 10.18   | 1.63    | 1.45     | 2.60    | 0.37     | 0.40     |
| FA1       | 30.36    | 17.23     | 16.50     | 23.33   | 5.32    | 2.14    | 1.27     | 2.53    | 0.45     | 0.27     |
| FA2       | 29.40    | 15.78     | 16.33     | 23.47   | 7.68    | 2.14    | 1.37     | 2.44    | 0.46     | 0.29     |
| FA3       | 22.59    | 11.56     | 16.78     | 31.85   | 10.20   | 1.65    | 1.39     | 2.35    | 0.38     | 0.41     |

Figure 6. Major oxide compositions of (a) FA1, (b) FA2, and (c) FA3 samples plotted against the soaking time in water.
Comparing FA2 to FA1, the higher free-lime FA2 (5.72%) has also affected the main composition of fly ash; i.e. the sum of SiO2, Al2O3, and Fe2O3 of FA2 is slightly lower than that of the lower free-lime FA1 (3.34%). For the FA3 (6.90% free lime), the composition of those three main oxides is comparatively lower than that of FA1 and FA2 due to such high CaO content contributing its part up to 31% in this type of fly ash. However in figure 6, the content of SiO2-Al2O3-Fe2O3 seems to be unaffected by the soaking period, as the same pattern is seen for all types of sample.

The high-calcium FA3 has shown the high SO3 content in comparison to those FA2 and FA1. It has been reported that the high calcium oxide content is an indicator of decreased sulfate resistance [35]. That is; the sulfur content in the ash could possibly increase in parallel with the increased CaO content, which is attributed to a result of the nature of coal resource and the calcium-sulfur reactions in flue gases of the furnace [52]. In addition, the soaking condition has resulted in a slightly decreased tendency of the SO3 composition with time.

3.4. Mineralogical compositions

Mineralogical study has been carried out to identify the crystalline phases present in the fly ash samples. Figures 7(a)–(c) is a result of x-ray diffraction patterns for FA1, FA2, and FA3, showing a comparison of the fresh (F-FA) to the soaked samples at different storing times (4, 24, 48, and 72 h). The major phases found for these samples include anhydrite (CaSO4), quartz (SiO2), lime (CaO), magnetite (Fe3O4), mullite (3Al2O3·2SiO2), hematite (Fe2O3), calcite (CaCO3), and portlandite (Ca(OH)2), as anhydrite, quartz, and lime are the main crystalline mineral phases typically existing in class C fly ash [1, 2, 52]. The peaks of anhydrite, quartz, mullite and lime appear at 2θ = 25.442, 26.2, 35.1, and 37.346, respectively. The peaks of anhydrite, quartz, mullite, and lime for the FA3 and FA2 samples could be seen more clearly than those for the FA1.
The intensity of anhydrite, quartz, mullite, and lime phases obviously decreases with the contacting time that fly ash was soaked in water. For the recovered samples, the intensity of these main peaks is comparatively smaller than that of the fresh sample, indicating the poorly crystalized minerals presenting, to some extent, in smaller concentrations. This is probably due to the high-calcium fly ash containing significant amounts of calcium, aluminium, and sulfur-bearing minerals, which is likely to be more soluble than the calcium aluminate glass containing low-calcium fly ash [52].

As the XRD pattern seen in the figures 7(a)–(c), with prolonged soaking time, the peak of lime phase apparently disappeared. CaO in the high calcium fly ash could participate in producing cementitious compounds when reacting with water leading to the amorphous phase formation [45]. The amorphous phase in the high calcium class C fly ash is considerably more reactive than that in the low calcium fly ash [54]. Such amorphous form of calcium compound is usually the main constituent involving the chemical reactions associated with fly ash utilization such as in cement and concrete industry [55]. The pozzolanic activity of fly ash could be affected by calcium in the amorphous form, rather than by either free lime or crystalline CaO [54].

3.5. Specification in Portland cement

3.5.1. Water requirement

In this work, the flow test was controlled at 110 ± 5, as water to binder (w/b) was optimized to achieve this flow requirement. In this study, the w/b ratios optimized for the mixture with FA1 are 0.67 (F-FA), 0.665 (4H), 0.665 (24H), 0.665 (24H), 0.665 (72H), and 0.675 for cement solely. While the mixtures with FA2 and FA3 needed a slightly higher amount of water. The w/b ratios found for the higher %free lime FA2 and FA3 are 0.7, with this larger water demand resulting in higher %water requirement than the lower free lime fly ash; see figure 8. While in the mixtures with low free lime fly ash ranging of 0.83%–2.11%, free lime was found to show no effect on water requirement of the mixtures, tested according to ASTM 311 [5].

As a function of the wet-storing condition, the amount of water uptake for all types of fly ash samples appears to be insignificantly different. Water requirement of all the prepared conditions, both fresh and soaked ones, in this study was lower than 105, the maximum percentage of water requirement regarding the ASTM C618. Compared to cement, both the fresh and the soaked fly ash samples required lower amount of water. Containing in cement concrete, fly ash with spherical shape promotes flowability offering good dispersion in the mixture [56]; as a result, water demand is reduced [41, 48, 57].

3.5.2. Strength activity index

Figure 9 illustrates a comparison of strength activity index of mortars containing fly ash of different conditions with respect to cement control. The strength activity indices of all fly ash containing mortars are higher than 75% of the cement control, meeting the ASTM C618 requirement. It is evident that the high %CaO fly ash has shown its pronounced effect on the strength activity index. The strength activity indices at 7 and 28 days of the FA3, for both fresh and soaked conditions, are higher than those of the FA1 and FA2 samples. The strength activity indices at 7 days of those fly ash samples lie in the range between 75%–95% of cement control. The prolonged curing time at 28 days has led to the enhanced strength activity indices of all specimens locating in the closer regime nearly the indices of cement control. It has been believed that high CaO fly ash could improve the strength activity index at early age of mortar due to the high amount of cementitious materials facilitating the pozzolanic
reactions [3, 33, 45], consequently reflecting the increase in compressive strength [41, 57, 58]. Illustrated in figure 9(b), the relative drop in strength with respect to those experiencing shorter wetting periods for the sample FA-72H at 28-day curing could probably be due to the late ettringite formation of the high-calcium (>20% CaO) fly ash associated with the less significance of free lime content in the samples affecting the compressive strength, hence making concrete potentially susceptible to expansion [5, 35]. Fineness (%sieve #325 retention) of fly ash is known a parameter to play a predominant role governing the compressive strength; the more fineness of particles exhibits a faster rate of early hydration [59]. At 7 days, the strength activity indices of mortar samples containing the FA3 sample are higher than those of the FA1 and FA2 samples. With relatively smaller particle size of the FA3 sample as shown in table 1, it is believed to be a beneficial effect on the compressive strength, to this point of view, in a combination with its high %CaO.

4. Conclusions

This research work reported on the properties of high calcium fly ash recovered from different wet-stored conditions. Varying from 4 h to 72 h, color of fly ash was observed to deviate from reddish brown to pale brown, presumably to be due to dissolution of ion content from the ash sample. The density values of the fresh and the soaked fly ash samples lie in a range of 2.61–2.99 g/cc, and the average particle sizes are approximately 13–20 μm. The soaking conditions led to the more precipitates depositing on the fly ash surface. The chemical composition by XRF resulted in the oxide compositions, including the main components of SiO2, Al2O3, Fe2O3, CaO, and the slight contents of SO3, K2O, Na2O, MgO, TiO2, P2O5. The chemical composition of the soaked samples has been compared to that of the non-soaked sample as a function of the wet-stored period. The SO3 content was found in the range of 5%–11%, tending to relate to calcium oxide composition and depending upon the nature of fly ash raw material. Anhydrite, quartz, mullite, and lime phases were identified to contain in the fly ash samples, with prolonged soaking leading to the low intensity of the crystalline peaks. For all types of fly ash, water requirement of all wet-storing conditions was found to be insignificant different. The strength activity indices of all fly ash containing mortars was found to be higher than 75% of the cement control. The fly ash
samples with such high contents of CaO could improve the strength activity index at curing age of mortar at 7 and 28 days; the significant amount of free lime and soluble calcium, aluminum, and sulfur-bearing minerals found varying with storage times are believed to play their pronounced role governing compressive strength. There was a previous report on utilization of 4.51% free lime of wet fly ash from the same source in Thailand [5] developed in the cementitious application. Our finding has presented an extended window in view of the possible use of fly ash, with its free lime content up to 6.90%, after recovering from the wet-stored conditions as the mineral admixture in concrete in which the important properties of the mixtures were found to satisfy the relevant standards.

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ORCID iDs

Sorachon Yoriya @ https://orcid.org/0000-0002-8890-5407

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