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INFLUENCE OF WET STORAGE ON FLY ASH REACTIVITY AND PROCESSING FOR USE IN CONCRETE

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ABSTRACT

Wet stored fly ash is increasingly being considered as a cement component in concrete. However, the effect of these conditions on the materials' reactivity is uncertain. The research described, investigated this for wet laboratory-stored (10% moisture) and site stockpile fly ashes, using lime consumption (BS EN 196-5, Frattini) and activity index (BS EN 450-1) tests. Progressive reactivity losses occurred with laboratory storage up to 730 days. These were influenced by dry fly ash fineness and holding period; and suggest agglomerates/ products forming (by scanning electron microscopy) affect lime access to particle surfaces, with similar type behaviour for stockpile materials. Compressive (cube) strength reductions were also found between dry and wet stored fly ash concretes. Stockpile fly ash reactivity following laboratory- (drying/ball milling) and pilot-scale (flash drying/de-agglomerating, air classifying, micronizing and carbon removal) processing was then investigated. Exposure of sufficient reactive material using these methods appears important, with continual improvements in mortar/concrete strength generally noted with reducing fly ash particle size. To meet activity index requirements, fly ash sub 10 µm contents, with the Portland cement used, needed to exceed about 30%, irrespective of storage conditions/processing used. Enhanced concrete strengths were obtained with sub 10 µm contents above about 50%.

INTRODUCTION

Fly ash has found increasing use in concrete over the past fifty years, offering benefits including, enhanced fresh properties, long-term strength, and aspects of durability (Thomas et al., 2017). In addition, the material can reduce the environmental impact of concrete (Leese and Casey, 2019), contributing to sustainable construction. Recent changes in electricity generation in the UK, however, with the introduction of renewable energy technologies (DBEIS, 2020) and planned coal-fired power station closures (Cossutta et al., 2021), mean fly ash supply in future is uncertain. While operational plans may vary, similar developments are likely in other countries in the near future (Europe Beyond Coal, 2021). At the same time, new limits for emissions are also affecting Standard-compliant fly ash availability for concrete (Diaz-Loya et al., 2019).

There is, therefore, a growing interest in wet fly ash from stockpiles and other wet holding areas, frequently located close to power stations, where significant quantities may exist (UKQAA, 2020), and which has potential for recovery and use (De Paoli, 2016). In the early stages of storage, it has been noted for low lime stockpile fly ash that physico-chemical effects take place. Indeed, with water addition, surface charge/cohesive forces tend to develop (Clarke, 1992) causing agglomeration. Thereafter, chemical reactions can occur (Donahoe, 2006; Wirth et al., 2019) leading to, for example, sulfate- (Georgakopoulos et al., 2002) and carbonate-based (Eze et al., 2013) product formation, with pozzolanic processes also reported for the material, in the presence of free lime (Sear, 2001). Such effects are influenced by fly ash properties and storage conditions, as well as being time-dependent (McCarthy et al., 1999).

The literature indicates that wet storage of fly ash may also cause losses in reactivity. Laboratory studies on wet fly ash paste and concrete have given reductions in lime consumption and long-term strength, which mainly increase with holding period (McCarthy et al., 2000). Modifying the concrete mix is a possible means of overcoming this. Strength reductions have also been noted in wet fly ash mortar and concrete for material stored for 6 to 24 months in a landfill (Cheerarot and Jaturapitakkul, 2004) and that from a lagoon (in excess water) (Bapat et al., 2006). In these cases, grinding was applied to match a dry fly ash standard mix, or the material was used in a low strength concrete application. Slightly improved strength has been found with wet fly ash (1 month stored) compared to dry material by allowing for moisture present through the concrete water content and using a particular mixing procedure (Nguyen et al., 2018). Changes to fly ash, or how the material was used in concrete have therefore been generally followed to account for the effects of wet storage.

Related work also shown that material held under these conditions may be prone to variability (Tyra et al., 2003; McCarthy et al., 2013), containing particles covering a range of types and sizes, including unburnt components. In addition, wet fly ash is likely to be difficult to handle, e.g., for discharge from hoppers, during concrete manufacture. A suitable approach for using fly ash could then be drying and homogenising to meet Standard requirements (limits, e.g., to BS EN 450-1; BSI, 2012), thereby giving material with consistent properties for concrete applications, fitting with existing practice (e.g. plant for handling) and maintaining supply. The current study, therefore, investigated the factors influencing fly ash reactivity during wet storage and following processing to achieve suitable properties for the materials' use in concrete.

RESEARCH PROGRAMME

To examine wet stored fly ash reactivity, materials (i) moistened/held in the laboratory for extended periods under controlled conditions and (ii) obtained from stockpile sites at various UK power stations, were used. These enabled wet fly ash storage period to be considered and investigation of material moistened and kept under field conditions. Lime consumption experiments, following BS EN 196-5 (BSI, 2011a), used to assess pozzolanic cements and with potential for evaluating fly ash reactivity (McCarthy et al., 2017), and activity index (mortar) tests, as described in BS EN 450-1 (BSI, 2012), were carried out. Measurements of compressive (cube) strength on structural (strength class) concretes were also made. Details of the fly ashes and main tests used to examine reactivity are summarized in Table 1.

The study then examined processing of stockpile fly ash by grinding in the laboratory, one of several methods that could be applied during wet material recovery, to determine its effect on reactivity, again using lime consumption and activity index tests, as well as measurements of concrete strength. Material from a site stockpile was also processed at pilot-scale by various means (flash drying/de-agglomeration, air classifying, micronizing and triboelectric carbon separation) to examine the wider range of available methods at an increased scale, with reactivity similarly investigated as in the other parts of the study. The fly ashes used during the processing studies and main test methods adopted to assess reactivity are given in Table 2.

FLY ASHES AND OTHER TEST MATERIALS

A range of low lime fly ashes from various UK power stations was sourced for the study. These included (i) three dry materials derived from bituminous (DFA1 and DFA3) and anthracite (DFA5) coals for moistening/wet storage in the laboratory, and (ii) eight samples held at different stockpile sites. Key physical and chemical characteristics of the fly ashes were determined using Standard/in-house test methods (McCarthy et al., 2019a) with the data given in Tables 3 and 4. These indicate that the dry fly ashes (DFA1, 3 and 5) had fineness ranging from 6 to 34% (retained on a 45 μm sieve) and loss-on-ignition (LOI) from 5.6 to 13.6%; i.e., material within and exceeding the BS EN 450-1 (BSI, 2012) limit (9.0%), respectively. The bulk oxide and mineral compositions were typical of those for UK materials and the coal types used (Sear, 2001).

The stockpile fly ashes, SFA1 to SFA8, had moisture contents on receipt of between 6.0 and 21.1%, i.e. close to the typical 10 to 20% conditioning range (McCarthy et al., 1999). The fly ashes all had fineness > 40% (45 μm sieve retention), i.e., outwith BS EN 450-1 (BSI, 2012) requirements. Hence, they were relatively coarse, and comparable to earlier studies on fly ash from this type of storage (McCarthy et al., 2013). These materials also had similar composition and LOI ranges to the dry fly ashes referred to above. Details of their holding periods in the stockpiles were not known.

A Portland cement (PC, CEM I) of Strength Class 52.5N to BS EN 197-1 (BSI, 2011b) was used for the lime consumption, activity index and concrete strength tests. CEN Standard sand to BS EN 196-1 (BSI, 2016) and local sand and gravel in 0/4, 4/10 and 10/20 mm fractions (particle densities, 2630, 2600, 2610 kg/m^3; absorption - laboratory dry (condition the aggregates were used in) to saturated surface dry, 0.8, 1.4, 1.3%), meeting BS EN 12620 (BSI, 2002) were adopted for the mortars and concretes respectively. A polycarboxylate ether superplasticizing admixture (SP) to BS EN 934-2 (BSI, 2009) was included to control workability of the test concretes, at fixed water content.

FLY ASH MOISTENING AND PRE-TREATMENT

Wet stored laboratory fly ashes were prepared as described previously (McCarthy et al., 1999). This used tap water and moisture contents of 10% and 20% (the latter, selectively), found to give greatest agglomeration, with mixing carried out for 4 min in a 25 litre pan mixer. Similar behaviour was obtained between the moisture contents and hence only 10% is reported here. The material was sealed in plastic bags/air-tight containers and held at 20°C for up to 730 d, with samples taken periodically for testing. The eight stockpile fly ashes were delivered to the laboratory following excavation. All test materials had moisture contents measured, and were then oven dried at 105°C. Following cooling, they were re-sealed and kept at 20°C until use.

SAMPLE PREPARATION AND EXPERIMENTAL PROCEDURES

Particle Size Distribution and Specific Surface Area

Particle size distribution was measured using a Malvern Mastersizer 2000 LASER particle size analyser. This measures light scatter deflection from particles, applying Mie light scattering theory. Approximately 1.0 g of fly ash was introduced to the equipment for optimum obscurancy and sonicated in water with an ultrasonic lance for 2 min (which may give some de-agglomeration), prior to testing. The method records particles up to 1000 µm, with at least five measurements taken for each sample.

Specific surface area (SSA), by nitrogen (N_2) adsorption, was evaluated with a Quantachrome NOVA 3000e surface area size analyser. Approximately 3.0 to 5.0 g of fly ash were introduced to the equipment, degassed at 105 ± 5°C, before subjecting to N_2 adsorption/desorption cycles, with SSA determined by Brunauer–Emmett–Teller (BET) theory. A single sample, with selective confirmatory tests, was used for each material/storage condition.

Lime Consumption

Lime consumption (CaO consumed) was measured, based on BS EN 196-5 (BSI, 2011a), sometimes referred to as the Frattini test, following McCarthy et al., (2017). Samples comprising 15·0 g of PC and 5·0 g of fly ash (i.e. 25% level) were added to 100 ml of distilled/de-ionised water in a sealed container and shaken vigorously before storage at 40°C. Following 8 / 15 d, these were filtered and the liquid tested for hydroxyl ion (OH^-) and CaO concentrations. Neutralisation was carried out using dilute nitric acid (0·1 M), with a bead of potassium hydroxide added and titration by EDTA (disodium-dihydrate salt of ethylenediamine tetra-acetic acid) solution (0·025 M) and murexide indicator. An auto-titrator (Metrohm Titrino 719; 20 ml exchange unit/2 μl increment dispenser) with pH and photometric electrodes were used to determine the end point, with single/confirmatory tests again carried out.

The Standard requires plotting of OH^- against CaO concentrations at 8 / 15 d and comparisons with the lime saturation curve. The PC/fly ash combination passes, i.e. is a pozzolanic cement, if the point is below the curve at either age. The data were also considered as the difference in CaO level between PC and PC/fly ash with respect to that in PC (as a percentage, i.e. CaO consumed by the PC/fly ash combination (McCarthy et al., 2017)). The earlier research suggests dry fly ash of fineness between 10 to 30% (retained on a 45 μm sieve) should have CaO consumed quantities at 8 / 15 d of between approximately 20 - 35% / 30 - 45%. A negative value implies CaO of the PC/fly ash combination is higher than that of PC, i.e. there is no net lime reduction.

Activity Index

Activity index was determined following BS EN 450-1 (BSI, 2012). The mortars contained 450 g PC (337·5 g PC/112·5 g fly ash in the test mixes), 225 g water and 1350 g of CEN Standard sand, and were mixed, as described in BS EN 196-1 (BSI, 2016). Three prisms (40 mm × 40 mm × 160 mm) were cast from each batch. After storage under hessian/plastic sheeting for 24 hours, and water curing at 20°C, six compression tests (2 × 3 prisms) were made at 28 and 90 d. The activity index is the PC/fly ash to PC mortar strength ratio, as a percentage, at the two ages, with values $\geq$ 75% and 85% respectively required to pass. Variations were dealt with following the Standard, and the results at each age are the mean of five or six measurements.

Concrete Production and Cube Strength

Concrete was prepared in a laboratory pan mixer, following BS 1881-125 (BSI, 2013). The main test concretes comprised 231 kg/m^3 PC and 99 kg/m^3 fly ash, 165 l/m^3 free water, with a fine to total aggregate ratio of 0.42, and a target plastic density of 2375 kg/m^3. SP doses of between 0·45 and 0·55% by mass cement were used to achieve a slump of 130 ± 20 mm. The 100 mm cubes cast were similarly stored and cured to the activity index mortars, until testing. Compressive (cube) strength was measured following BS EN 12390-3 (BSI, 2019), on three specimens, at test ages up to 90 d.

RESULTS AND DISCUSSION

Wet Storage Effects on Reactivity and Concrete

Laboratory fly ash

The lime consumption results for laboratory wet stored fly ash (held for up to 730 d) are shown in Figure 1. These indicate that highest values and therefore reactivity were obtained for dry material, with increases mainly found at the later test time (15 d in the study), as typically noted with the method (Tironi et al., 2013; Kramar and Ducman, 2018). In addition, greatest lime consumption occurred with DFA3, with minor differences between the other fly ashes, i.e., generally agreeing with fineness effects reported previously (Dhir et al., 1998; Chindaprasirt et al., 2005). While there was some scatter, the trends with wet storage show gradual reductions in lime consumption, noted from only 1 d of moistening, with increasing storage period (and higher values at the later test age). DFA5, of average initial fineness, generally gave greater levels during this period, with lower reactivity for DFA1 (coarser) and DFA3 (finer) fly ash.

The data are shown at 8 and 15 d on OH^-/CaO charts (BS EN 196-5, BSI, 2011a) in Figure 2. At 8 d, dry fly ashes DFA3 and DFA5 passed (were below the lime saturation curve), while the coarser DFA1 achieved this at the later test age, with all PC/dry fly ash (25% level) combinations thereby classed as pozzolanic cements (BSI, 2011a). Of the wet fly ashes, some of the short-term DFA5 materials passed, with the other PC/fly ash combinations above the saturation curve at 8 d. While most combinations passed by 15 d, those (i) at some of the early storage ages for DFA3 and (ii) held for 730 d (all fly ashes) did not.

The behaviour observed above is comparable to the gradual coarsening of fly ash found during wet storage previously (McCarthy et al., 1999). To examine the effect of changes in fly ash under these conditions, lime consumption at 8 and 15 d is shown against fly ash median particle size in Figure 3. As noted above, dry fly ash gave gradual reductions in lime consumption with coarsening (higher median particle size). For wet stored fly ash, increases in median particle size occurred with holding period and were greatest with initially finer (dry) fly ash. More noticeable reductions in lime consumption were also found with increasing fly ash fineness, with similar effects at both test ages. These seem likely to relate to the different quantities of fine particles present, between materials, where agglomeration mainly occurs (McCarthy et al., 1999) and which also influence fly ash reactivity (Joshi and Kadu, 2012; Soutsos et al., 2016).

Activity index and lime consumption results are shown in Figure 4 left (28 and 8 d) and right (90 and 15 d) for dry and wet stored laboratory fly ashes, alongside median particle size and SSA by N_2 adsorption. For activity index all dry PC/fly ash combinations met BS EN 450-1 (BSI, 2012) limits of 75 and 85% at 28 and 90 d. Finer fly ash (DFA3) gave highest values with minor differences between the other materials. During wet storage, there were gradual reductions in this property (compared to dry fly ash) and these were mainly greater with fine fly ash (DFA3) at both test ages. Similar type effects were obtained between the two reactivity tests and in comparison, with median particle size. By 180 d wet storage, none of the fly ashes achieved 75% at 28 d, with two of these less than 85% at 90 d.

The results in Figure 4 also indicate increases in SSA, by N_2 adsorption, with storage period which were greatest for fine fly ash. These are likely to reflect agglomerate structures and products forming during wet storage (Donahoe, 2006; Nguyen et al., 2018) within the material and on particle surfaces as shown in the scanning electron microscopy (SEM) images (Hitachi S-4700 equipment; DFA1 and DFA3) in Figure 5. Previous research indicates that lime access to particles is likely to be an important factor influencing dry fly ash reactivity (Massazza, 1998). Other work notes reactions in lime/fly ash systems are influenced by (i) the material's pozzolanicity and (ii) diffusion of activating components through developing hydration products (Shi and Day, 2000) to reactive surfaces.

The reduced lime consumption and activity index noted after short holding periods and limited reaction time, suggest physico-chemical effects causing agglomerate formation (Clarke, 1992; Nguyen et al., 2018) and sulfate-based compound development, found on particles from early ages (McCarthy et al., 1999), may affect lime/particle surface contact. The continued agglomeration (Figure 5) and other chemical processes occurring with time in the material (Donahoe, 2006) are then likely to further increase these inhibiting effects. From the results, it seems that extended test periods enable lime to access particles, and for material to pass at the later ages (15 and 90 d), despite 'normal' PC hydration and fly ash reaction products also developing.

At the same time, fly ash surface chemistry changes may be expected with wet storage (Baltrus et al., 2002; Donahoe, 2006), including pH reductions (Eze et al., 2013; Wirth et al., 2019). With the common ion effect (Van Eijk and Brouwers, 2000), these may influence lime content in the concrete aqueous phase (Mertens et al., 2009), also affecting the process. The results, therefore, suggest wet storage affects fly ash reactivity, and the study progressed to examine material from stockpile sites and influences on concrete.

Stockpile fly ash

The results from tests on stockpile material are shown on OH^-/CaO charts in Figure 6. None of the fly ashes passed the test at 8 d, with all above the lime saturation curve. While some fly ashes, SFA3, SFA4 and SFA5 were below the curve by 15 d, the others were not. The data therefore show similarities between laboratory and stockpile stored materials, indicating that wet stored fly ash affects reactivity behaviour, but in the longer term, with extended test periods, migration of lime to reactive particle surfaces can occur. The random behaviour found for the stockpile fly ashes may relate to the effects noted in Figure 3, which indicate that both initial fly ash fineness and storage period affect reactivity. Given this, it may be deduced that materials passing the test were of average fineness when dry and had been held in their stockpiles for short periods of time.

Lime consumption and activity index of stockpile fly ashes are shown against median particle size in Figure 7. These data indicate increased lime consumption at the later test age, corresponding to the behaviour noted in Figure 6, with a general reduction found with increasing median particle size. The scatter in the results is likely to reflect the fact that lime consumption depends on the initial fineness of the material and storage period. For activity index, there were similarities with the lime consumption data, with increases between 28 and 90 d and a suggestion of median particle size influences on behaviour, particularly at the earlier age. In this case, highest values were obtained for SFA4, SFA5, SFA7 and SFA2, SFA5, SFA7 mortars at 28 and 90 days, with only SFA5 and SFA7 meeting BS EN 450-1 (BSI, 2012) limits (75 and 85%) at both ages.

It is possible that variations in developing hydrate structure between slurry and mortar (the former is likely to be more porous due to the higher w/c ratio) affect lime access to fly ash particles (McCarthy et al., 2017). This, and the agglomerates and products forming during wet storage, may lead to the differences obtained for the fly ashes between reactivity tests. Given the effects noted in the data above, and that in most cases the fly ashes did not meet the Standard requirements, processing, which has been shown to give benefits to dry

(Kiattikomol et al., 2001) and wet stored (Cheerarot and Jaturapitakkul, 2004; McCarthy et al., 2018) fly ash previously, is likely to be necessary for its use at a practical level.

Concrete cube strength

The cube strength results for dry/wet laboratory stored (180 d) fly ash concretes are shown in Figure 8 (left) against median particle size. These indicate less effect of wet storage on concrete strength at 7 d, with differences between 1.5 and 6.0 MPa (greater for DFA3). This corresponds to the period of limited fly ash reactivity (and less contribution to concrete property development by the material), under the curing conditions used (water at 20°C), which generally initiates at 7 to 14 d (Fraay et al., 1989; Papadakis, 1999). At later test ages, effects increased between dry and 180 d wet storage, with differences at 90 d of between 2.5 and 15.0 MPa (again most noticeable for DFA3, see also changing gradient).

As indicated in Figure 8 (right), for selected stockpile fly ash concretes, the differences in results at a given age were in the range of 3.5 to 5.0 MPa and this remained essentially unchanged over the 7 to 90 d test range. A comparison of laboratory wet stored fly ash concretes (in Figure 8 (left), i.e., not including the dry materials) gave similar ranges of between 2.0 and 4.0 MPa for the same test ages. It is evident that there are some differences in reactivity influences, as noted previously (McCarthy et al., 2017), depending on the test media (slurry, mortar or concrete) and developing microstructure (affecting lime access) the material is used in.

Processed Wet Stored Fly Ash

Laboratory grinding

As suggested above, fly ash from wet storage areas is likely to require processing before use. To investigate the influence of this on reactivity, two of the stockpile fly ashes (SFA1 and SFA2) were subjected to grinding in a ball mill. Each vessel of the mill contained 125 g of fly ash and used 6 hardened steel balls, with the process mainly carried out for 20 min, at average speed. A trial examining different regimes indicated that this material quantity and grinding time should enable breakdown of agglomerates.

The stockpile fly ash results are given on a OH^-/CaO chart in Figure 9 (left). As noted above, SFA1 and SFA2 gave values above the lime saturation curve at 8 and 15 d. Following grinding, which reduced median particle sizes by half to two-thirds, with fineness (45 µm sieve retention) less than 10% in both cases, values above the lime saturation curve were still obtained at 8 d, with levels below this only achieved by 15 d. Although there were reductions in particle size, it appears that there were not enough exposed reactive surfaces, and agglomerates/products present still inhibited lime access, with the test only being passed with lime migration over longer periods.

Activity index data are also shown for stockpile processed fly ash (SFA1, SFA2, SFA3 and SFA4) against median particle size in Figure 9 (right). Grinding of the fly ashes enabled the materials to achieve values above 75% and 85% at 28 and 90 d and thereby to pass the test, albeit only marginally at the earlier age, with more noticeable differences as this increased. These data, therefore, give general agreement with lime consumption in terms of the period of greater reactions in fly ash.

Cube strength results for concretes containing SFA1, SFA2 and SFA5 and a lagoon fly ash (LFA5, from a related study) are given in Table 5, together with (fly ash) median particle size and sub 10 µm contents (i.e., reactive component; Gambhir, 2013; Moghal, 2017). The materials were ball milled directly (SFA1, SFA2) or sieved at 600 µm (SFA5, LFA5), prior to carrying this out. The fly ash conditions following ball milling for periods up to 30 min (SFA1, by SEM) are shown in Figure 10, which indicate gradual reductions in agglomerates, as mentioned above, with the fracture of larger particles also appearing to occur. This is similar to effects noted previously with dry fly ash following 30 min grinding, where almost all components were less than 30 µm in size (Payá et al., 1995).

There were relatively minor differences in concrete cube strengths with grinding up to 30 min for SFA1, despite fly ash median particle size reductions from 31 µm to 12 to 15 µm. Slightly more noticeable effects were found with SFA2 (with 20 minutes grinding), between 1.5 and 5.0 MPa over the test age range, corresponding to greater differences in initial/final median particle sizes, suggesting material influences on the outcome. Benefits to concrete strength with processed wet stored fly ash were found when the sub 10 µm content increased above about 50%, similar to McCarthy et al. (2018), (median particle size < 10 µm) for SFA5 and LFA5 following 60 min ball milling, which were more noticeable at later test ages.

Pilot-scale Techniques

In order to examine fly ash reactivity following processing using industry techniques and increased volumes, a pilot-scale trial was carried out. This used stockpile fly ash (SFA8), treated by various means, including flash drying/de-agglomeration (pin mill; PFA2), and then air classifying (particle separation - testing the fine fraction; PFA3) or micronizing (particle size reduction; PFA4). Although LOI was within Standard limits (BSI, 2012), triboelectrostatic carbon separation was carried out at pilot-scale (Bittner et al., 2014), for some processed fractions (low carbon (LC)), see Table 2.

Physical characteristics and LOI, and results from lime consumption, activity index and concrete cube strength (w/c ratio 0.53, 30% fly ash in cement, water content 184 l/m^3, SP dose 0.3 to 0.5% for 130 ± 20 mm slump) tests are shown in Table 6. While median particle sizes were similar between SFA8 and PFA2 (the changes for 45 µm sieve retention, seem to reflect the different test methods), this reduced for PFA3 and PFA4. Carbon removal gave further reductions in median particle size for PFA2, although increases were noted for PFA4. The reason for the latter is unclear but may relate to the high fineness of PFA4, since this can affect fly ash/carbon separation (Wang et al., 2021).

As indicated in Table 6, there was relatively low lime consumption at 8 d, with all values less than 15%, although they generally followed fly ash fineness. At 15 d, with the exception of PFA4, all processed materials had values greater than 30%, suggesting reactive material at this test age. Similar effects were noted with activity index, which gave gradual increases with reducing fly ash median particle size. These data are also shown against sub 10 µm content in Figure 11 (left). In this case, values around the 28 d, 75% limit, were noted, with material receiving greater processing (air-classifying, micronizing, carbon separation), passing. The same fly ashes passed at the later age (90 d, 85%), with the gradient of the trend line steeper in this case. Carbon removal gave increases in reactivity with coarser fly ash, with less effect at greater fineness.

The concrete results are shown in Figure 11 (right) against sub 10 µm content and indicate minor differences in cube strength between stockpile, flash dried/de-agglomerated (PFA2) and air classified (PFA3) materials, with small increases for micronizing (PFA4) and greater effects at the later test age. Related work suggests that benefits noted with processing on water requirement (which follow fineness), could also be taken advantage of, to further extend these (McCarthy et al., 2019b). The results, therefore, give general agreement with those noted for the tests in the laboratory.

ESTIMATING FLY ASH REACTIVITY

Although wet storage affects fly ash properties and behaviour, with reductions in reactivity noted, the research has shown that the material has potential as an active component in concrete. While fly ash could be recovered from stockpile storage and directly used, it is likely to require processing in order to meet Standard requirements. In the current study, drying was used to recover handling properties, pre-treatment by coarse screening (in some cases) and grinding in a ball mill in the laboratory were adopted. This was also shown to be possible by these and other means at pilot-scale, with reactivity results depending on the methods used. Differences in energy requirements (costs) of processing are likely between these and in practice would need to be balanced against the properties achievable.

Given the sensitivity of fly ash reactivity to the effects of wet storage and processing, attempts were made to explore if a measure of fineness could provide a means of estimating the property. This focussed on the sub 10 μm content of the material, since this has been shown to relate to various properties of recovered fly ash concrete previously (McCarthy et al., 2018). The fly ashes considered included those, (i) in dry form, from the laboratory study, (ii) stored in the laboratory at 10% moisture, reported in the paper, (iii) held for different periods at 15 and 20% moisture in the laboratory, (iv) from the eight stockpiles, (v) from the stockpiles and following ball milling (4 No) and (vi) produced during the pilot-scale trial. The results in Figure 12, show that fly ashes with sub 10 μm contents greater than about 30% achieve the 75 and 85% activity index requirements in BS EN 450-1 (BSI, 2012). This value corresponds to the PC used in the study but suggests potential for providing a rapid estimate of fly ash reactivity, irrespective of the storage conditions/processing method used.

CONCLUSIONS

Laboratory wet storage gave gradual reductions in fly ash reactivity, noted in both lime consumption and activity index tests from early storage up to 730 d. This could be related to coarsening of fly ash, corresponding to agglomerate and product formation (by SEM) and increases in SSA (by N_2 adsorption) during storage, with greater effects for finer fly ash. Wet stored fly ashes tended to give better properties, for both reactivity tests, with shorter holding periods and at later ages (15 d and 90 d).

Stockpile fly ashes gave similar reactivity behaviour to those wet stored in the laboratory with respect to meeting Standard requirements. Given the changing effects occurring with time, the results suggest lime accessibility to reactive fly ash particle surfaces may affect the process. The random type behaviour noted for the stockpile fly ashes appears to relate to the influences of initial dry fineness and wet storage period on reactivity, observed in the laboratory tests.

Tests on concrete indicate small differences in cube strength for dry and 180 d laboratory wet stored material at early ages, corresponding to the period of least fly ash reactivity. Differences were material-dependent and increased with test age. For both wet stored laboratory and stockpile fly ashes, small differences in cube strength occurred between materials in comparisons made against their median particle size at different test ages.

Laboratory grinding of stockpile fly ash enabled material to pass the reactivity tests. For lime consumption this was at 15 d, while for activity index there were passes at 28 d (marginal) and 90 d. This suggests exposure of sufficient reactive particle surfaces is an important factor affecting reactivity. Grinding effects on concrete strength were influenced by median particle size before/after processing and the extent to which this reduced (and sub 10 µm content increased). Noticeable effects on strength mainly occurred at later test ages.

Similar effects to those in the laboratory were found for pilot-scale processing, with increasing reactivity noted for particle separation/size reduction techniques. Carbon removal gave increased reactivity with coarser fly ash, with less effect at greater fineness. To meet activity index requirements, fly ash was required to have sub 10 µm contents of 30% or more, irrespective of the storage conditions/processing used. There is the need to examine this for PC from different sources. Some increases in concrete strength were noted, particularly at later ages, when fly ash sub 10 µm contents exceeded levels of about 50%.

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List of Tables

Table 1 Experimental programme for wet storage effects on fly ash reactivity (main properties tested)

Table 2 Experimental programme for processing effects on wet stored fly ash reactivity (main properties tested)

Table 3 Physical and chemical characteristics of dry fly ashes

Table 4 Physical and chemical characteristics of stockpile fly ashes

Table 5 Wet stored (stockpile/lagoon) and processed fly ash concrete cube strength development and physical characteristics

Table 6 Characteristics of stockpile fly ash before and following pilot-scale processing

Table 1 Experimental programme for wet storage effects on fly ash reactivity (main properties tested)

Wet storage	**Fly Ash**[1]	**Storage Periods**	**Property**
Laboratory	DFA1[2], DFA3, DFA5	Dry, 1 d, 7 d, 30 d, 90 d, 180 d, 730 d	Particle size distribution Specific surface area Lime consumption Activity index (Tested at selected storage periods) Scanning electron microscopy (DFA1, DFA3; Dry, 730 d wet stored) Concrete cube strength (Dry, 180 d wet stored)
Site Stockpile	SFA1, SFA2, SFA3, SFA4, SFA5, SFA6, SFA7, SFA8	Unknown	Particle size distribution Lime consumption Activity index Concrete cube strength (SFA1, SFA2, SFA3, SFA5, SFA6, SFA7)

Parenthesis indicates tests were only made on fly ashes/at storage periods given, otherwise all variables considered

[1] DFAX – Initially dry fly ash; SFAX – Stockpile fly ash

[2] Fly ashes moistened with water at 10% by dry mass, sealed and stored at 20°C

Table 2 Experimental programme for processing effects on wet stored fly ash reactivity (main properties tested)

Processing	**Fly Ash**[1]	**Processing**	**Property**
Laboratory	SFA1, SFA2 SFA3, SFA4	As received; As received, Grinding[2], 20 Min	Particle size distribution Activity index Lime consumption (SFA1, SFA2) Scanning electron microscopy (SFA1[3])
	SFA1,	As received; As received, Grinding, 10 Min, 20 Min, 30 Min;	Concrete cube strength
	SFA2,	As received; As received, Grinding, 20 Min;	
	SFA5,	< 600 µm[4]; < 600 µm, Grinding, 60 Min;	
	LFA5	< 600 µm; < 600 µm, Grinding, 60 Min	
Pilot-scale	SFA8, PFA2,	As received; SFA8, Flash dried/ De-agglomerated;	Particle size distribution Lime consumption Activity index Concrete cube strength (SFA8, PFA2, PFA3, PFA4)
	PFA2 LC,	PFA2, Carbon removal;	
	PFA3,	PFA2, Air classified;	
	PFA4,	PFA2, Micronized;	
	PFA4 LC,	PFA4, Carbon removal	

Parenthesis indicates tests were only made on fly ashes/for processing given, otherwise all materials/processes considered

[1] SFAX – Stockpile fly ash; LFA5 – Lagoon fly ash; PFAX – Pilot-scale processed fly ash

[2] Grinding in a ball mill for the periods indicated

[3] SFA 1 also tested after 10 and 30 minutes grinding

[4] < 600 µm – fly ash passed through 600 µm sieve before testing/ball milling

Table 3 Physical and chemical characteristics of dry fly ashes

CHARACTERISTIC	**DRY FLY ASH**		
	DFA1	DFA3	DFA5
Physical Properties/Loss on Ignition			
Fineness, 45 µm sieve retention, %	33.9	5.7	18.4
Loss-on-ignition	8.3	5.6	13.6
Oxide composition, %			
CaO	4.5	3.1	2.2
SiO_2	47.9	50.1	41.3
Al_2O_3	20.3	22.4	23.4
Fe_2O_3	7.4	7.6	6.7
MgO	-	1.6	0.9
TiO_2	0.9	0.9	0.9
P_2O_5	0.6	0.4	0.9
K_2O	2.2	2.5	2.3
Na_2O	1.5	1.7	0.7
SO_3	1.8	1.2	2.0
Mineral composition, %			
Quartz	5.0	8.8	1.6
Hematite	0.6	2.1	2.8
Magnetite	0.2	0.3	0.0
Mullite	5.7	5.7	6.4
Glass / Others[1]	80.2	77.7	75.6

[1] not including LOI

Table 4 Physical and chemical characteristics of stockpile fly ashes

CHARACTERISTIC	**STOCKPILE FLY ASH**							
	SFA1	SFA2	SFA3	SFA4	SFA5	SFA6	SFA7	SFA8
Physical Properties/Loss on Ignition								
Moisture Content[1], %	12.7	21.1	17.7	15.7	6.0	19.8	17.6	15.7
Fineness, 45 µm sieve retention, %	53.8	47.9	49.6	41.1	42.0	63.2	41.9	62.3
Loss-on-ignition	9.7	8.9	10.6	15.9	6.1	3.5	6.9	6.3
Oxide composition, %								
CaO	4.4	2.3	3.3	2.1	2.1	4.3	2.5	2.8
SiO_2	44.3	43.7	47.4	41.2	51.2	49.6	48.2	46.5
Al_2O_3	21.8	22.9	19.5	22.7	22.7	25.2	23.6	24.5
Fe_2O_3	9.0	9.4	8.0	7.4	5.8	6.2	8.8	8.4
MgO	1.6	1.5	1.6	1.0	1.4	1.3	1.5	1.6
TiO_2	1.1	1.0	0.8	0.9	1.0	1.2	0.9	1.0
P_2O_5	0.6	0.4	0.7	0.8	0.2	0.9	0.3	0.4
K_2O	2.0	2.4	2.0	2.3	2.8	1.7	2.8	2.5
Na_2O	0.8	0.7	0.9	1.0	1.0	1.1	0.8	0.9
SO_3	1.6	1.3	2.3	1.4	1.7	1.6	0.8	1.8
Mineral composition, %								
Quartz	4.3	7.5	8.9	1.1	11.7	11.4	8.0	4.3
Hematite	1.7	1.7	1.1	0.5	1.4	1.9	1.5	1.4
Magnetite	0.2	0.0	0.3	0.1	0.2	0.2	0.1	0.1
Mullite	7.0	10.0	6.4	3.2	10.4	17.0	10.0	9.3
Glass / Others[2]	77.2	71.9	72.7	79.2	70.2	66.0	73.5	78.6

[1]As-received, [2]not including LOI

Table 5 Wet stored (stockpile/lagoon) and processed fly ash concrete cube strength development and physical characteristics

FLY ASH/ PROCESS	**CUBE STRENGTH, MPa**					**MEDIAN PARTICLE SIZE, µm**	**SUB 10 µm PARTICLES, % by volume**
	3 d	**7 d**	**28 d**	**56 d**	**90 d**		
SFA1	19.5	28.5	38.0	44.5	48.0	31	21
SFA1 BM10	20.5	26.5	39.0	44.0	44.0	15	36
SFA1 BM20	20.0	23.5	37.0	43.5	48.5	14	35
SFA1 BM30	17.0	25.5	38.5	44.0	48.0	12	40
SFA2	19.0	24.5	37.5	42.5	45.5	44	20
SFA2 BM20	20.5	27.5	39.5	47.5	47.0	15	35
SFA5 < 600 µm	17.5	24.0	34.5	42.5	44.5	26	21
SFA5 < 600 µm, BM60	18.0	25.0	37.0	43.0	47.0	8	57
LFA5 < 600 µm	19.0	25.0	36.5	43.5	45.5	21	28
LFA5 < 600 µm, BM60	18.0	24.5	37.0	46.5	51.0	8	57

BMXX – fly ash ball milled for XX min; < 600 µm – fly ash passed through 600 µm sieve before testing/ball milling

Table 6 Characteristics of stockpile fly ash before and following pilot-scale processing

CHARACTERISTIC	SFA8	**PROCESSED FLY ASH FRACTION**				
		PFA2	PFA2 LC	PFA3	PFA4	PFA4 LC
Physical and Loss-on-Ignition						
Fineness, % retained on a 45 µm sieve	62.3	30.6	30.8	1.5	0.6	4.4
Median particle size (d_{50}), µm	33.0	33.2	9.2	13.8	7.1	24.9
Loss-on-Ignition, %	6.3	6.1	3.1	6.3	6.6	3.3
Reactivity						
CaO Consumed, % – 8 d	1.7	2.1	1.0	7.2	9.4	14.1
– 15 d	5.0	30.8	47.6	50.7	21.6	-
Activity Index, % – 28 d	70	70	75	75	80	80
– 90 d	80	83	93	88	103	104
Concrete[1] Cube Strength, MPa – 28 d	32.5	31.5	-	31.0	34.5	-
– 90 d	40.0	39.0	-	41.0	43.0	-

SFA8 – Stockpile feed material; PFA2 – Flash dried/de-agglomerated;

PFA2 LC – Flash dried/de-agglomerated/carbon removal; PFA3 – Flash dried/de-agglomerated/air classified;

PFA4 – Flash dried/de-agglomerated/micronized; PFA4 LC – Flash dried/de-agglomerated/micronized/carbon removal

[1] W/C of concrete = 0.53

List of Figures

Figure 1. CaO consumed at 8 d (left) and 15 d (right) of dry and laboratory wet stored (10% moisture, 20°C) fly ash after various storage periods

Figure 2. Comparison of OH^- and CaO concentrations at 8 d (left) and 15 d (right) of dry and laboratory wet stored (10% moisture, 20°C, various storage periods up to 730 d) fly ash (as per BS EN 196-5 (2011a))

Figure 3. Relationships between median particle size and 8 d (left) and 15 d (right) CaO consumed of dry and laboratory wet stored (10% moisture, 20°C, various storage periods up to 730 d) fly ash (solid line gives trend for dry materials; points move left to right with increasing storage period)

Figure 4. Physical characteristics and reactivity of dry and wet stored (10% moisture, 20°C) fly ash after various storage periods

Figure 5. Scanning electron microscopy images of dry DFA1 (left) and DFA3 (right) (top) and after 730 d storage period (bottom)

Figure 6. Comparison of OH^- and CaO concentrations at 8 d (left) and 15 d (right) of stockpile fly ash (as per BS EN 196-5 (2011a))

Figure 7. Relationships between median particle size and CaO consumed (left) and activity index (right) of stockpile fly ash

Figure 8. Comparisons of median particle size and concrete cube strength of dry, laboratory wet stored (10% moisture, 20°C, 180 d) (left) and stockpile fly ash (right)

Figure 9. Effect of grinding on OH^- and CaO concentrations (left) and median particle size and activity index (right) of stockpile fly ash

Figure 10. Scanning electron microscopy images of stockpile fly ash: SFA1 (top left) and after ball milling for 10 min (top right), 20 min (bottom left) and 30 min (bottom right)

Figure 11. Relationship between sub 10 µm particle content and activity index (left) and concrete cube strength (right) of stockpile fly ash before and following pilot-scale processing

Figure 12. Relationship between sub 10 µm particle content and activity index of various fly ashes (dry, laboratory stored and stockpile; and laboratory and pilot-scale processed)

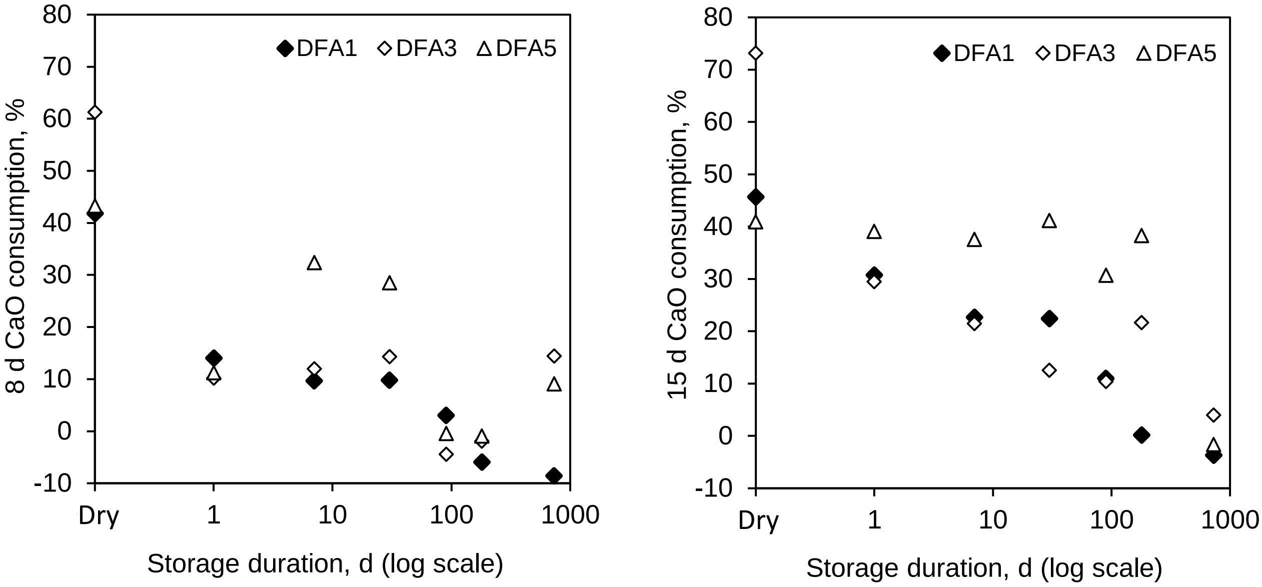

Figure 1. CaO consumed at 8 d (left) and 15 d (right) of dry and laboratory wet stored (10% moisture, 20°C) fly ash after various storage periods

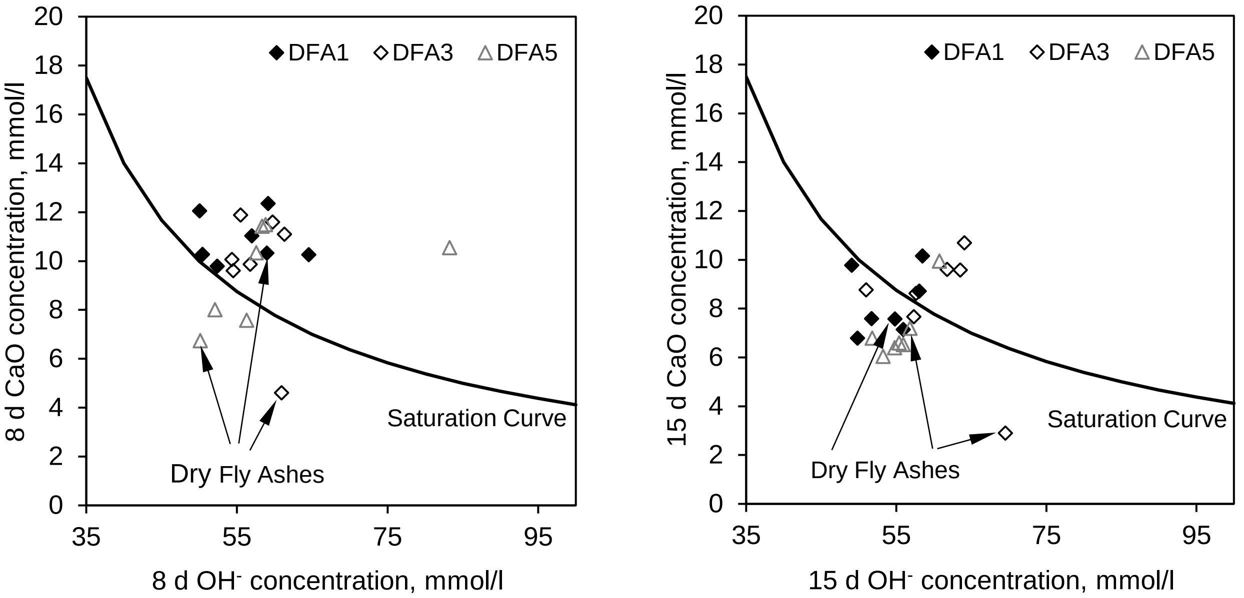

Figure 2. Comparison of OH^- and CaO concentrations at 8 d (left) and 15 d (right) of dry and laboratory wet stored (10% moisture, 20°C, various storage periods up to 730 d) fly ash (as per BS EN 196-5 (2011a))

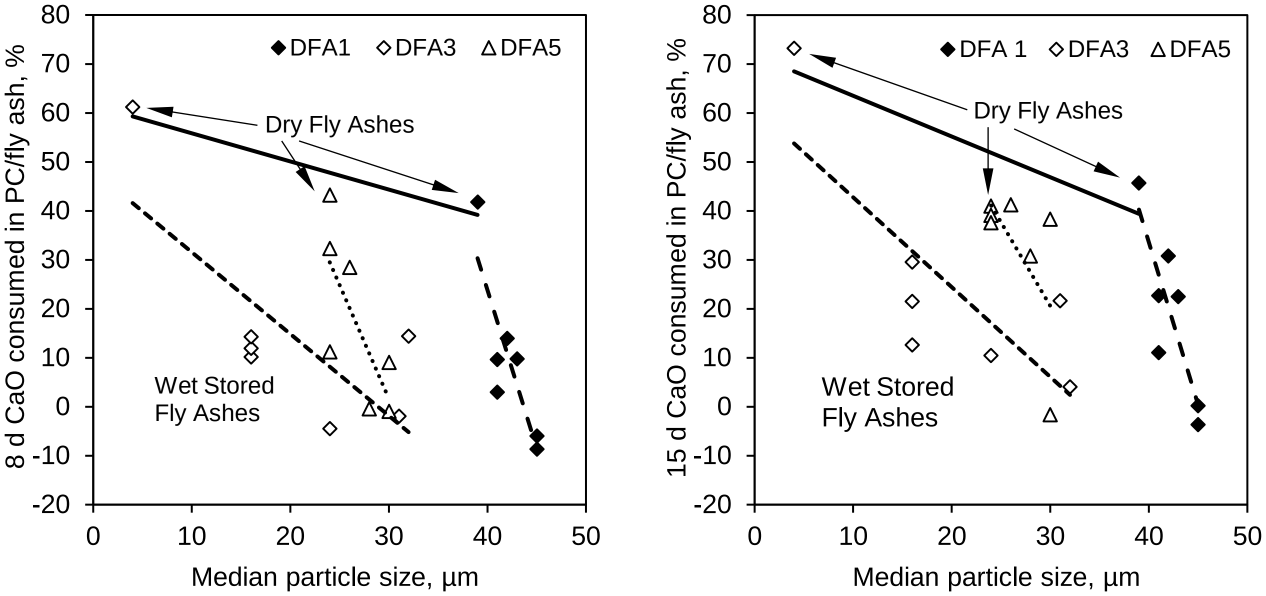

Figure 3. Relationships between median particle size and 8 d (left) and 15 d (right) CaO consumed of dry and laboratory wet stored (10% moisture, 20°C, various storage periods up to 730 d) fly ash (solid line gives trend for dry materials; points move left to right with increasing storage period)

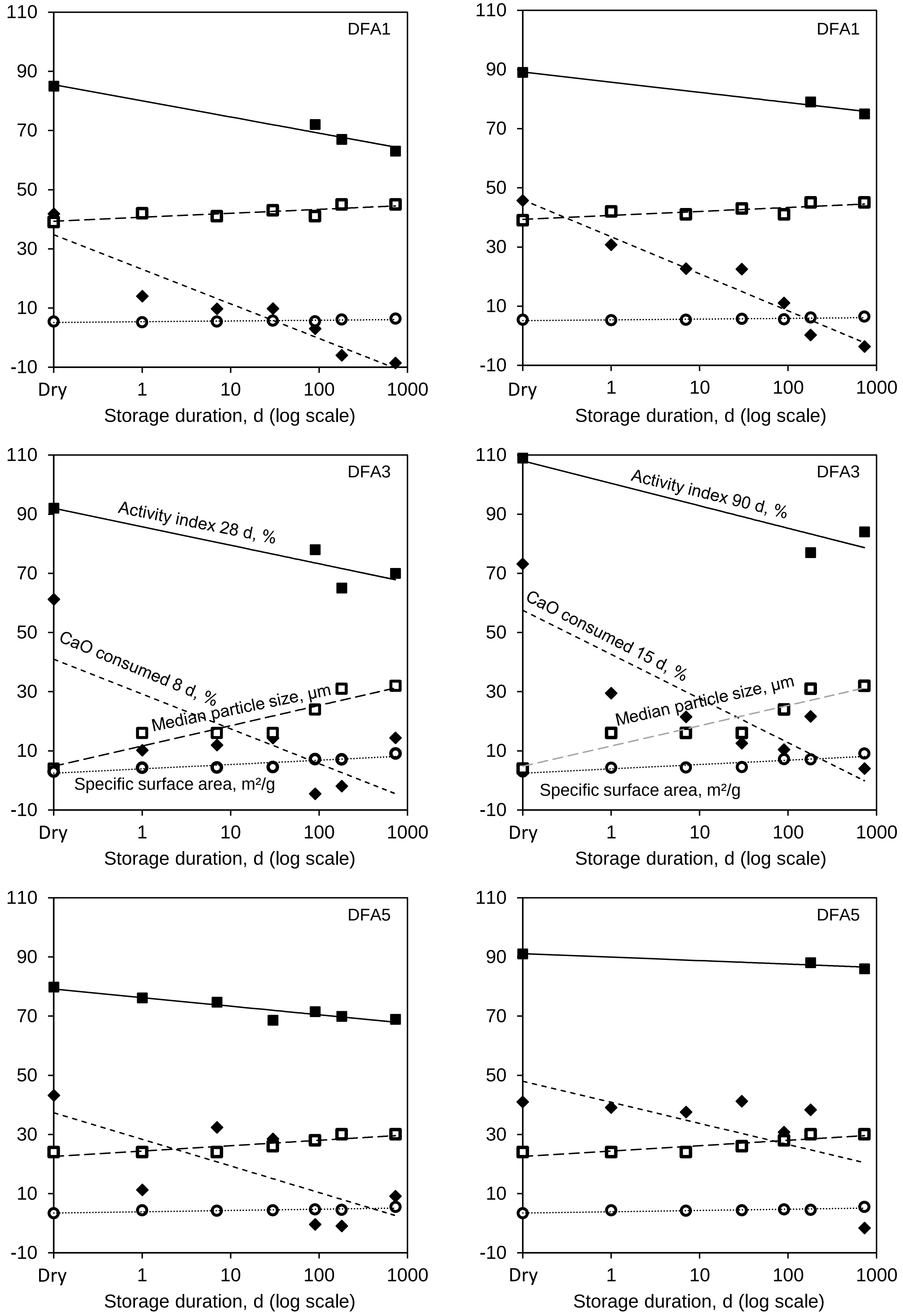

Figure 4. Physical characteristics and reactivity of dry and wet stored (10% moisture, 20°C) fly ash after various storage periods (labels shown for DFA3 apply to all fly ashes)

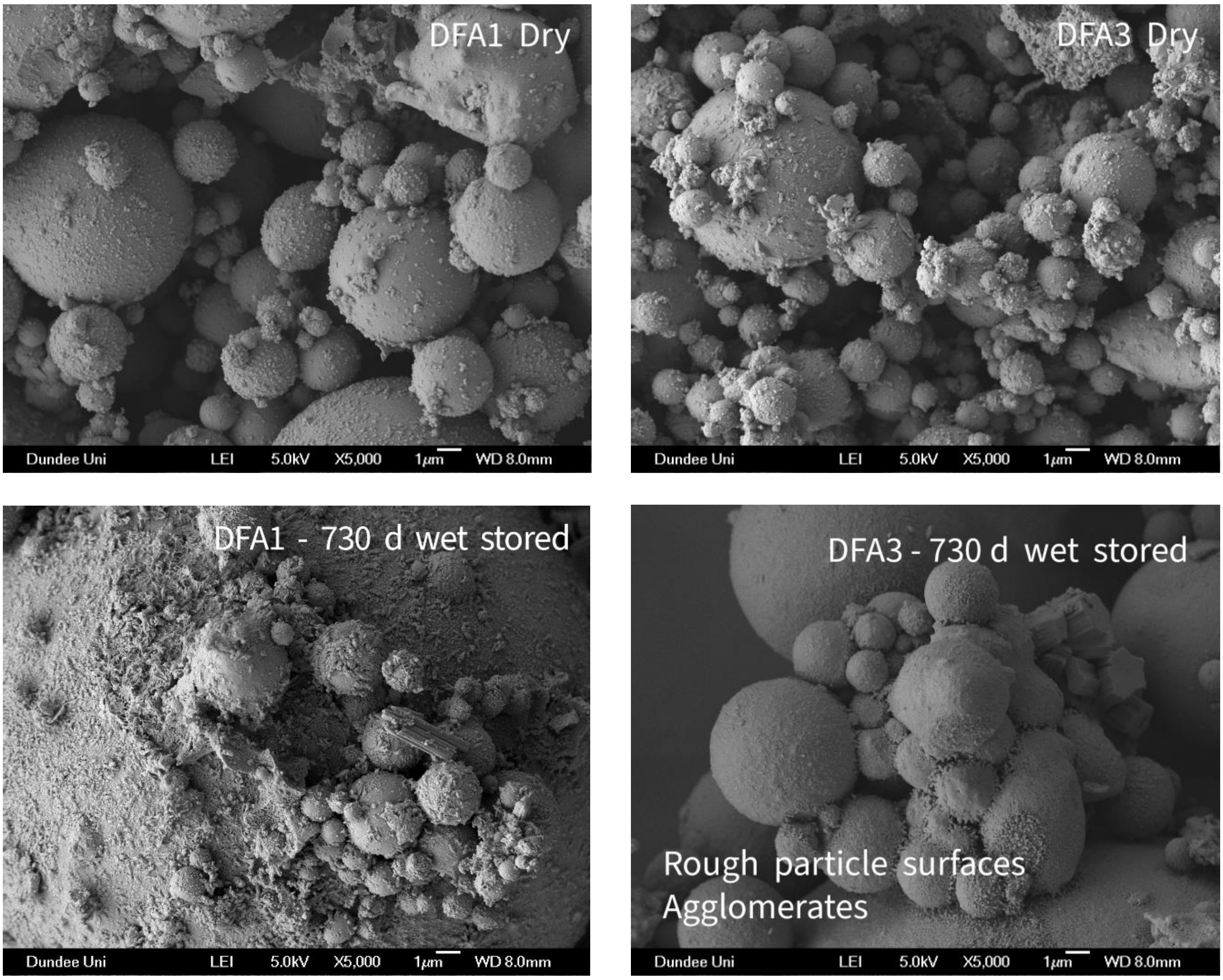

Figure 5. Scanning electron microscopy images of dry DFA1 (left) and DFA3 (right) (top) and after 730 d storage period (bottom)

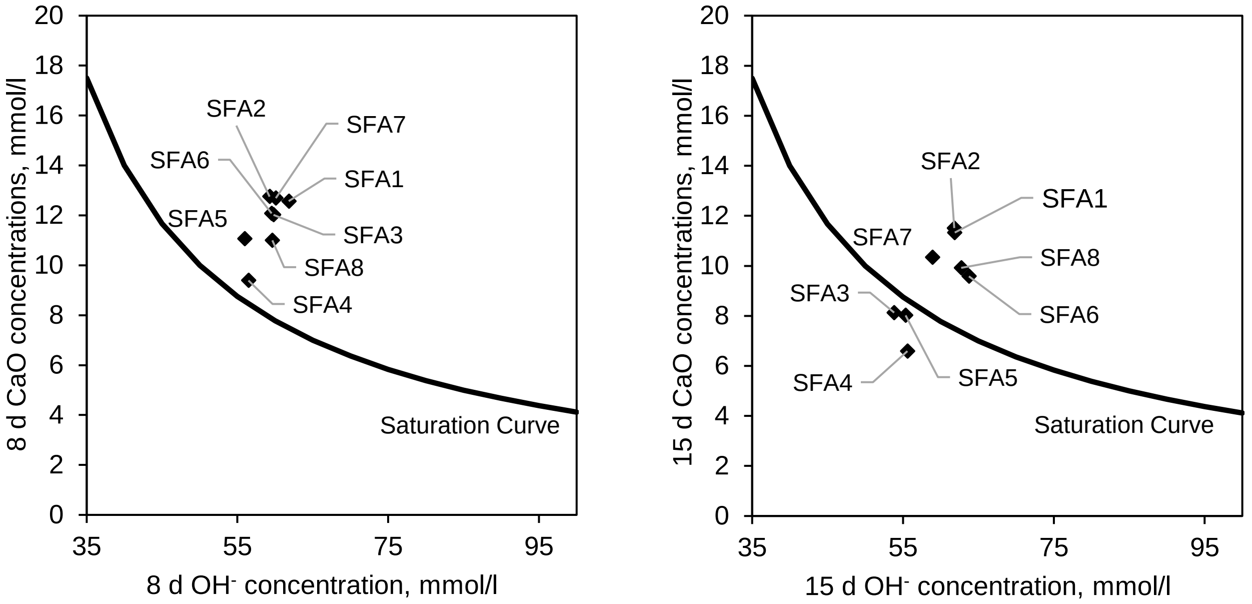

Figure 6. Comparison of OH^- and CaO concentrations at 8 d (left) and 15 d (right) of stockpile fly ash (as per BS EN 196-5 (2011a))

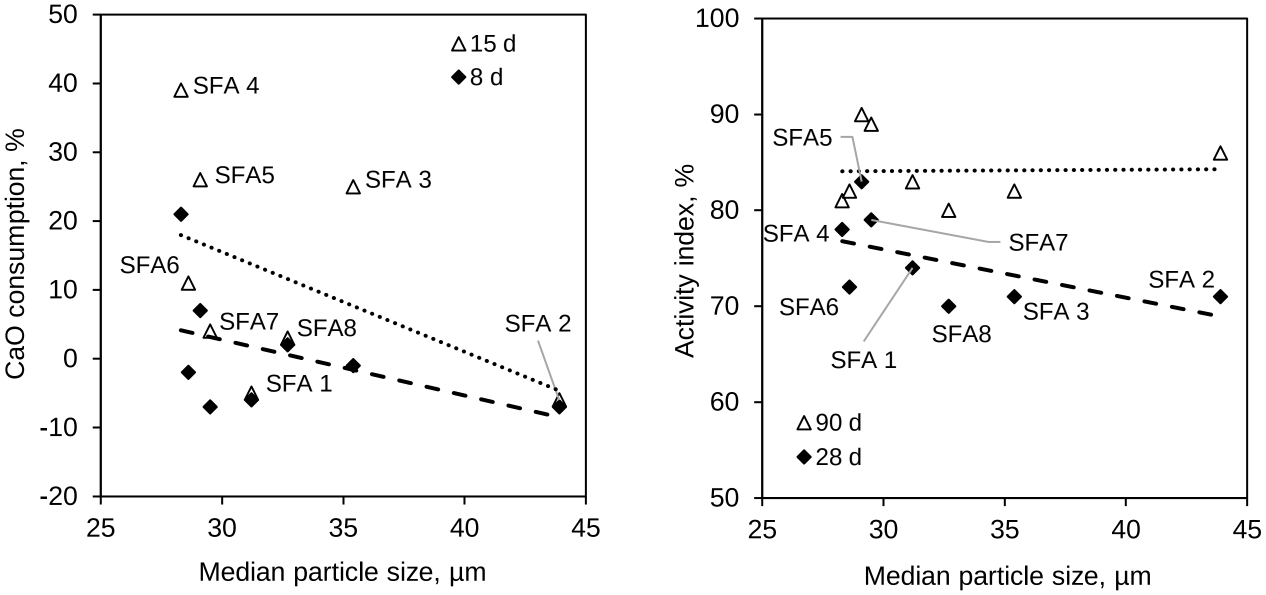

Figure 7. Relationships between median particle size and CaO consumed (left) and activity index (right) of stockpile fly ash

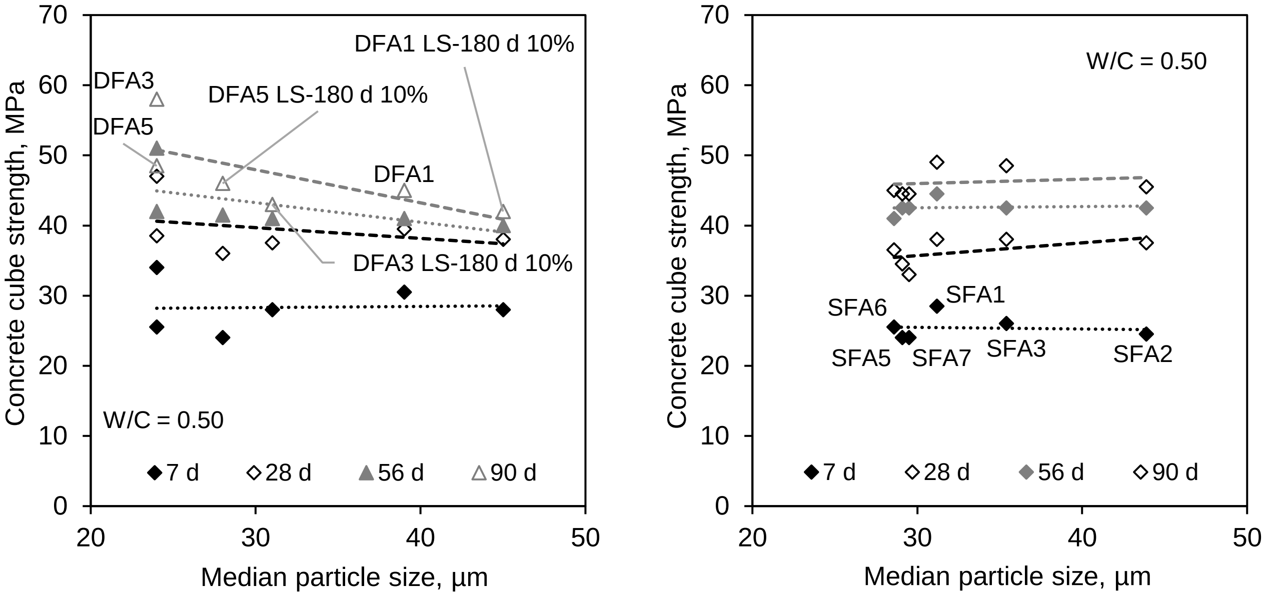

Figure 8. Comparisons of median particle size and concrete cube strength of dry, laboratory wet stored (10% moisture, 20°C, 180 d) (left) and stockpile fly ash (right)

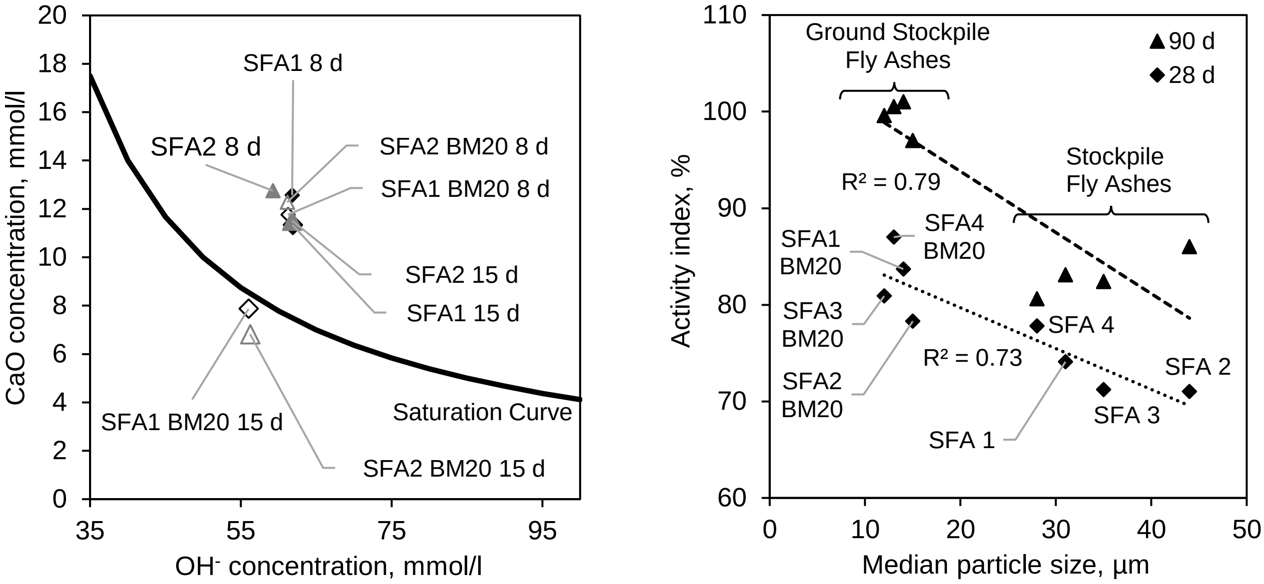

Figure 9. Effect of grinding on OH^- and CaO concentrations (left) and median particle size and activity index (right) of stockpile fly ash

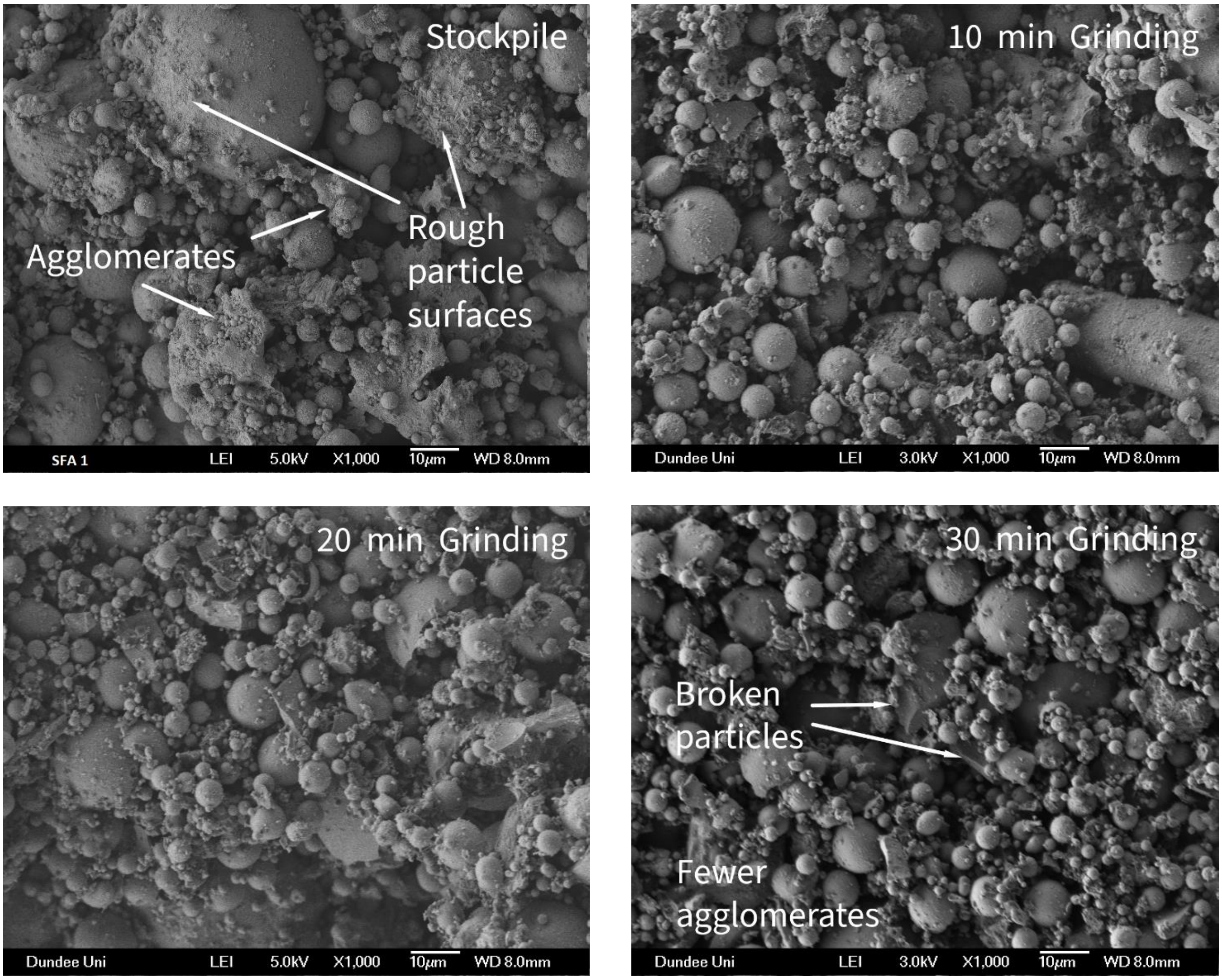

Figure 10. Scanning electron microscopy images of stockpile fly ash: SFA1 (top left) and after ball milling for 10 min (top right), 20 min (bottom left) and 30 min (bottom right)

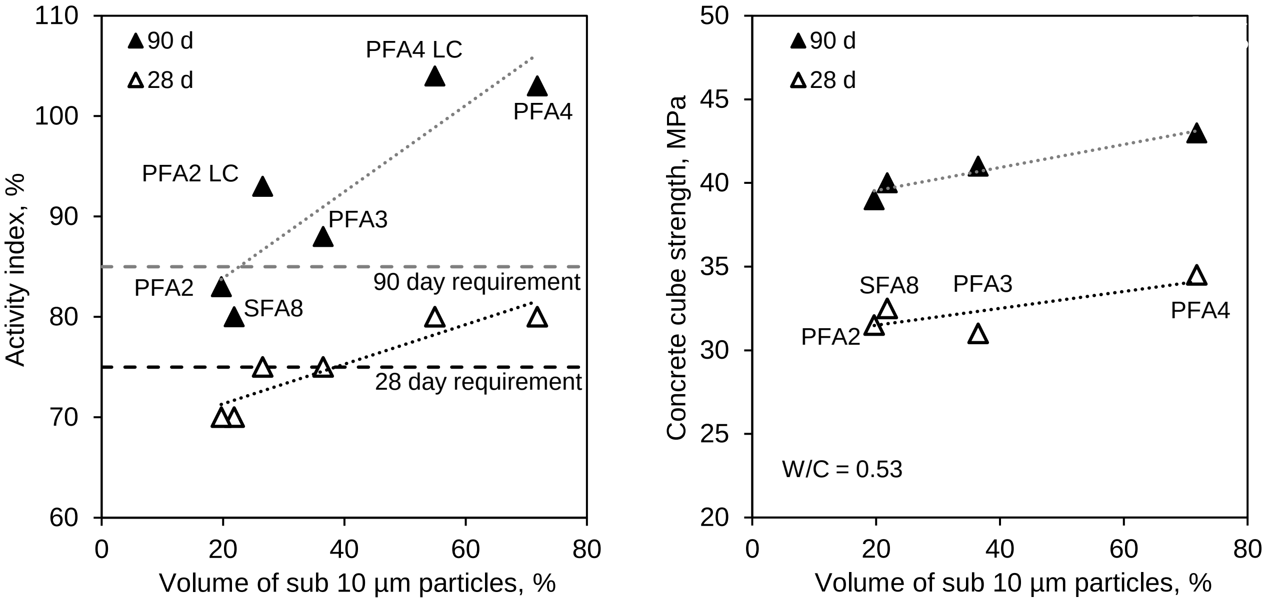

Figure 11. Relationship between sub 10 µm particle content and activity index (left) and concrete cube strength (right) of stockpile fly ash before and following pilot-scale processing

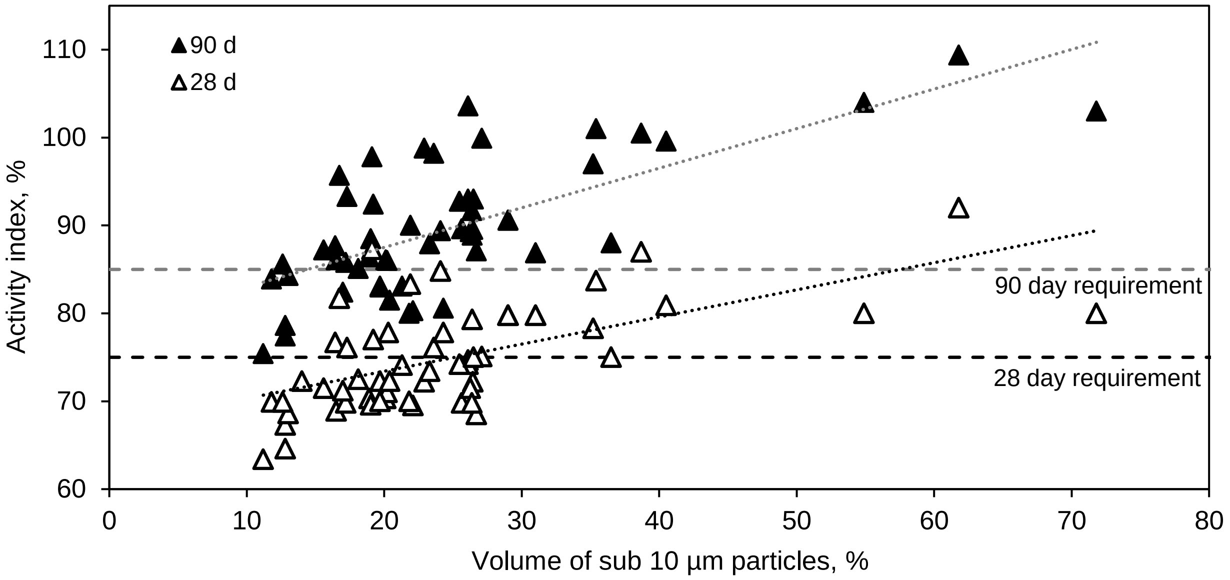

Figure 12. Relationship between sub 10 µm particle content and activity index of various fly ashes (dry, laboratory stored and stockpile; and laboratory and pilot-scale processed)