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Author post-print (accepted) deposited in CURVE September 2016

Original citation & hyperlink:

Sadique, M. and Coakley, E. (2016) The influence of physico-chemical properties of fly ash and CKD on strength generation of high-volume fly ash concrete. *Advances in Cement Research*, volume (in press)

<http://dx.doi.org/10.1680/jadcr.15.00103>

DOI 10.1680/jadcr.15.00103

ISSN 0951-7197

ESSN 1751-7605

Publisher: Thomas Telford (ICE Publishing)

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The influence of physico-chemical properties of fly ash and CKD on strength generation of high volume fly ash concrete

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Abstract

This paper presents a laboratory study on the use of Cement Kiln Dust (CKD) as an activator of fly ash when used in high volumes within concrete. Two separate batches of fly ash and CKD were tested to assess the effect of material variability on binder properties and compressive strength gain. Ternary blends of fly ash (55% - 65%), CEM 1 (30%) and CKD (5% - 15%) and quaternary blends that included moderate amounts (15% - 18.5%) of Ground Granulated Blast-Furnace Slag (GGBS) were prepared. Physico-chemical properties of individual binder materials were compared and concrete compressive strength was measured at 2 days, 7 days and 28 days. Ternary blends of 60% fly ash, 30% cement and 10% CKD resulted in moderate early age and 28 day strength and addition of GGBS enhanced strength significantly due to increased ettringite formation. Particle fineness, water demand and LOI content of fly ash and CaO and SO₃ content of CKD were found to be the main physico-chemical factors that influence compressive strength gain.

Key words: Ash utilisation; cement kiln dust; GGBS; chemical properties; compressive strength.

1. Introduction

Reduction in Portland cement consumption is a key aim of the construction industry in reducing CO₂ emissions associated with construction. Increasing the use of waste by-products as cement replacements was the major sector plan objective set out by UK Mineral Product Association (Mineral Products Association, 2011). Although High Volume Fly Ash (HVFA) concrete benefits from increased long term strength and durability relative to Portland cement mixes, its early age strength is generally lower. The pozzolanic reaction occurs relatively slowly which increases concrete setting

times and reduces the rate of early strength gain and these effects have been found to be more severe for higher levels of cement replacement (Kayali and Sharfuddin Ahmed, 2013, Bouzoubaâ and Lachemi, 2001) The aim of this investigation is to use Cement Kiln Dust (CKD) to activate the cement replacement materials used to increase early age strengths of concretes containing high volumes of fly ash.

Many investigations on HVFA concrete have aimed to ensure sufficient compressive strength is achieved by using a low water to cementitious materials ratio with the required workability achieved by the use of superplasticisers. Naik and Singh (Naik and Singh, 1991) used a melamine based superplasticiser to achieve water to cementitious materials ratio between 0.29 – 0.33 for fly ash proportions between 40% – 70%. Compressive strength results for the mix containing 70% fly ash were only 2.3 MPa for 1 day testing but this increased substantially to 56.6 MPa after 28 days. For lower replacement levels, higher early age strengths were observed but 28 day strengths were similar for all replacement levels. Atis (Atiş, 2003) investigated HVFA concrete mixes using a carboxylic type superplasticiser with similar mix proportions to Naik and Singh (1991) and found 1 day strength of only 1.8 MPa but moderate 28 day strength of 33.2 MPa for 70% fly ash. Bouzoubaa and Lachemi (2001) and Yazici (Yazıcı, 2008) investigated production of self-compacting concrete containing high volumes of fly ash which takes advantage of improved fluidity and increased resistance to segregation of HVFA concrete mixes. However, low early strength was also apparent in these investigations, particularly for higher fly ash contents.

Physical and chemical properties of the ash have a significant effect on the strength gain of HVFA concrete. Bouzoubaa and Fournier (Bouzoubaâ and Fournier, 2003) tested concrete made with ash from two different sources and found that concretes made with ash of higher CaO content and with finer particles generally achieved higher strengths. Bouzoubaa et al (Bouzoubaâ et al., 2001) carried out a range of testing on HVFA concretes where the cement and ash were pre-blended and ground and equivalent mixes where the cement and unground ash were added separately at the mixer. They found that concretes made with the pre-blended binder had shorter setting times, greater mechanical properties and improved durability characteristics and they attributed the improved performance to increased ash fineness and improved homogeneity of the blend. Jiang and Malhotra (Jiang and Malhotra, 2000) tested concretes with 55% fly ash from 8 different ash sources and observed higher strengths for concretes made with ashes of higher CaO content. They also commented that higher equivalent alkali content and finer ash particles tended to increase strength.

Several investigations on HVFA concretes have examined the effect of inclusion of GGBS within the binder. Zhu et al (Zhu et al., 2012) tested high binder content mortars with 30% cement, 40% – 70% fly ash and 0% – 30% GGBS. They found that compressive strength increased at all ages with increasing GGBS content within the blend relative to the strength of the binary blend of 70% fly ash and 30% cement. However, the main increase in strength was observed for just 10% inclusion of GGBS, particularly at early age (e.g. increases in 3 day strength relative to the binary blend were 27%, 34% and 44% for 10%, 20% and 30% of GGBS respectively). Compressive strength testing by Li and Zhao (Li and Zhao, 2003) found that a concrete made with 60% cement and 40% fly ash had 1 day strength 46% less than for the control 100% cement concrete mix. However, measured 1 day strength of an equivalent mix with 60% cement, 25% fly ash and 15% GGBS was only 13% less than the control. The enhanced early age strength when GGBS is included was attributed to a number of factors; GGBS reacts with hydrated lime from cement forming a secondary calcium silicate compound, which (along with ettringite and hydrated lime) precipitate around fly ash particles and increase the hydration rate of ash. Also, when GGBS hydrates, it provides OH^- ions and alkalis to the pore fluid which break down the glassy phase of fly ash.

Cement Kiln Dust (CKD) is a by-product of the cement manufacture industry that is of similar composition to cement but typically contains higher proportions of alkalis and sulphates. Its high alkalinity makes it suitable for activation of fly ash and GGBS with a view to enhancing early age reactivity. However, Kunal et al (Kunal et al., 2012) highlight that CKD is highly variable and its properties are influenced by properties of raw materials, fuel used in cement manufacture and kiln type so this should be considered when using in such applications.

Although a reasonable amount of literature is available on CKD – GGBS systems, literature on use of CKD to activate fly ash is quite scarce. Babaian et al (Babaian. et al., 2003) investigated the reactivity of CKD – fly ash systems but the main emphasis of this investigation was the effect of grinding so no meaningful conclusions can be drawn in relation to unground binder constituents. Wang et al (2004) investigated the effect of curing temperature and NaOH addition on the behaviour of CKD – fly ash systems. They found compressive strengths as low as 2.1 MPa after 28 days for binders of 50% CKD and 50% fly ash without using elevated curing temperatures or NaOH addition although the strength increased to 10.1 MPa after 56 days. Maslehuddin et al (Maslehuddin et al., 2010) investigated concrete mixes including cement, CKD and other cement replacement materials. For their concrete mix with 70% cement, 20% fly ash and 10% CKD, they measured 3 day compressive strength of 30.3 MPa (compared with 38.3 MPa for an equivalent mix with 100% cement). They also tested a mix made with 80% GGBS, 15% CKD and only 5% cement

and found a moderate 3 day strength of 23.0 MPa. Chaunsali and Peethamparan (Chaunsali and Peethamparan, 2013) examined pastes with 70% CKD (from two different sources) and 30% of either fly ash or GGBS cured at an elevated temperature of 75 °C. For both fly ash and GGBS mixes, they observed higher compressive strengths at all ages for mixes with the CKD that had the higher free lime and sulphate contents. They noted that the higher free lime content led to greater C-A-S-H gel forming during the pozzolanic reaction and that the higher sulphate content and alumina content increased ettringite formation. They also noted that the CKD that led to greater strengths had a smaller particle size throughout the particle size distribution range.

The aim of this investigation is to evaluate the use of CKD in HVFA concretes with particular emphasis on early age strength. Note that assessment of the effect of increased alkalis, sulphates and chlorides due to inclusion of CKD on the durability of such concrete mixes is beyond the scope of the current study. The effect of physical and chemical properties of both the fly ash and CKD from two different sources is assessed and the effect of use of moderate amounts of GGBS within the blend is evaluated.

2. Experimental work

2.1. Binder materials

All binders within this investigation were made up of CEM I 52.5 (referred to as CEM throughout this report), fly ash, CKD and in some mixes, GGBS. Two batches of fly ash from different sources and two batches of CKD from the same source were collected for use in this study to establish the effect of binder material variation on resulting concrete properties. Figure 1 shows Particle Size Distribution (PSD) plots for all raw binder materials determined using a Malvern Mastersizer 2000 with an MU sampler. Median particle size from PSD results, Blaine fineness and density in accordance with BS EN 196-6 and where relevant, standard consistence in accordance with BS EN 196-3 and activity index as defined by BS EN 450 were established and are presented in Table 1. Scanning Electron Microscopy (SEM) images of both batches of fly ash and CKD taken by a JOEL 6060LV Scanning Electron Microscope are shown in Figures 2 and 3 respectively so that the particle morphology can be compared between batches. The chemical composition of the raw binder materials was determined using X-Ray Fluorescence (XRF) carried out using a PAN analytical Axios Advanced XRF spectrometer and the resulting oxide proportions are given in Table 2. The mineralogical composition of the binder materials was determined by X-Ray Diffraction (XRD) using a Bruker D8 Advance with DaVinci and results are shown in Figure 4.

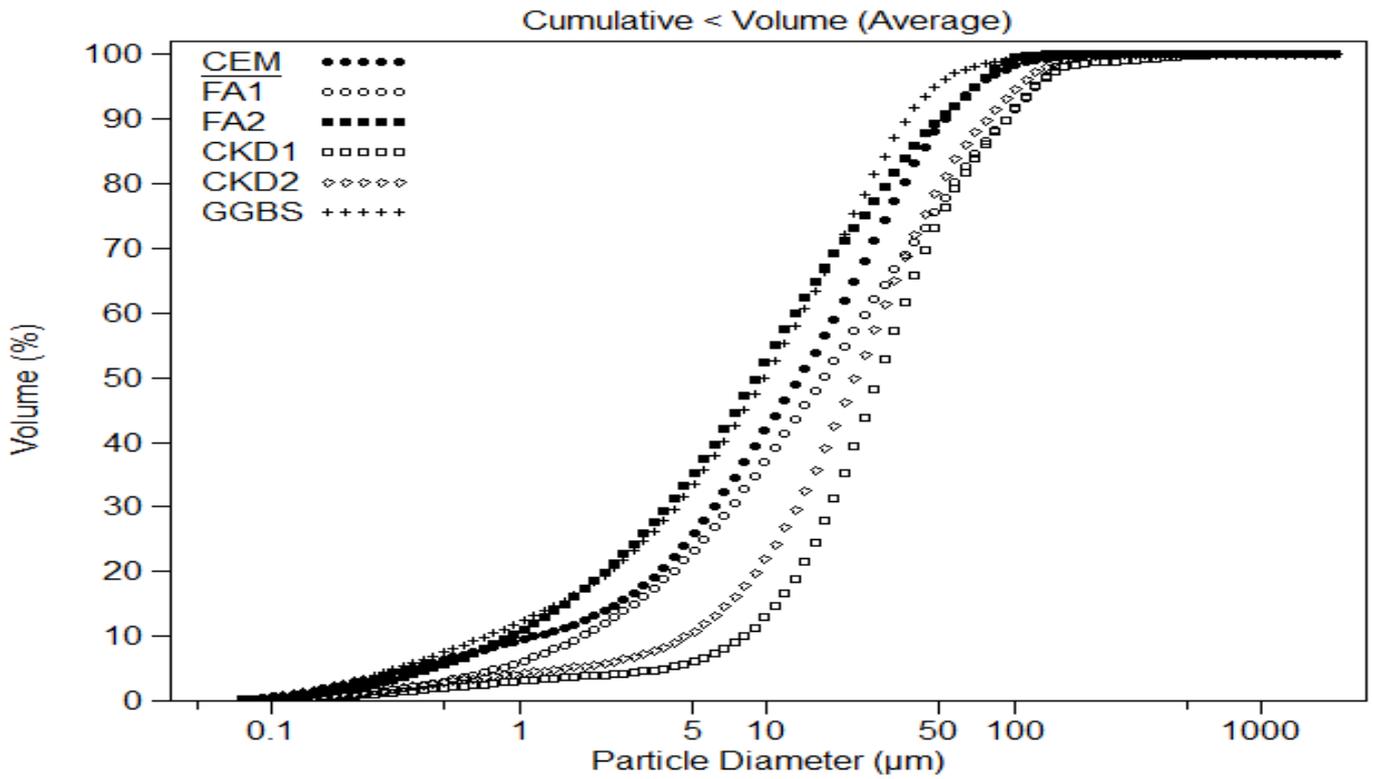
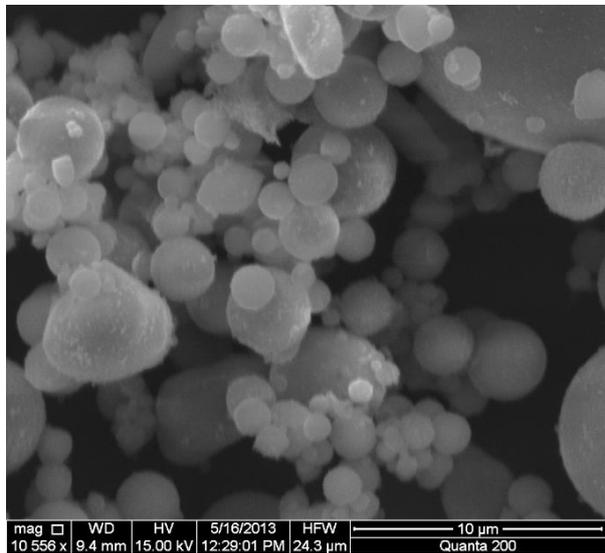
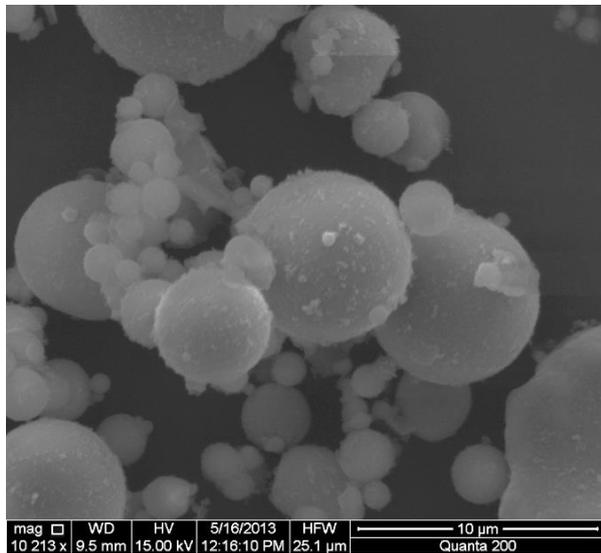
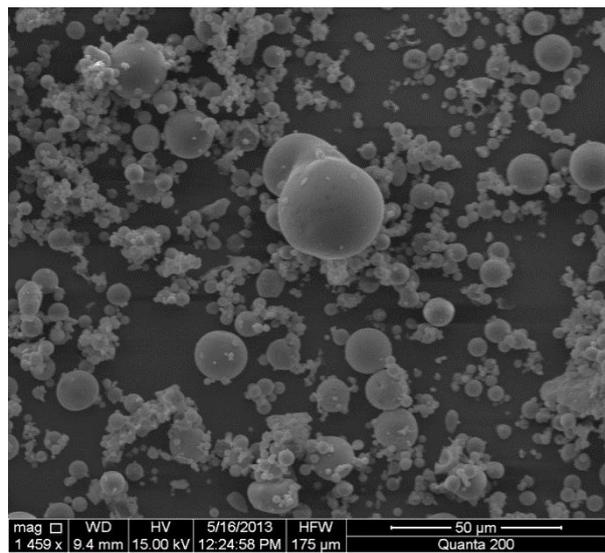
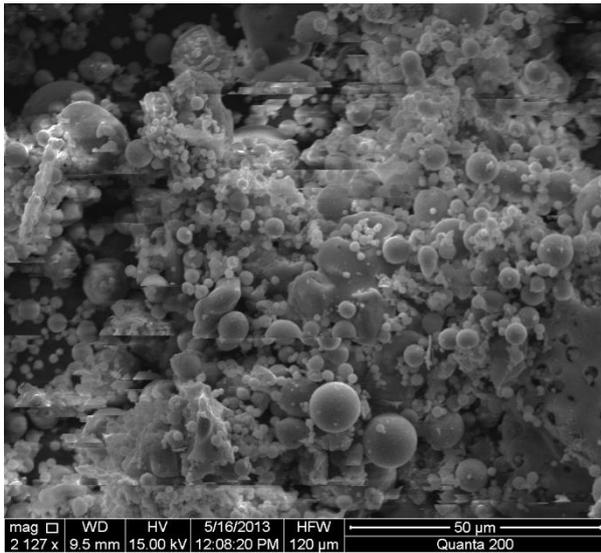


Figure 1: Particle size distribution plots of raw binder materials

Table 1: Physical properties of raw binder materials

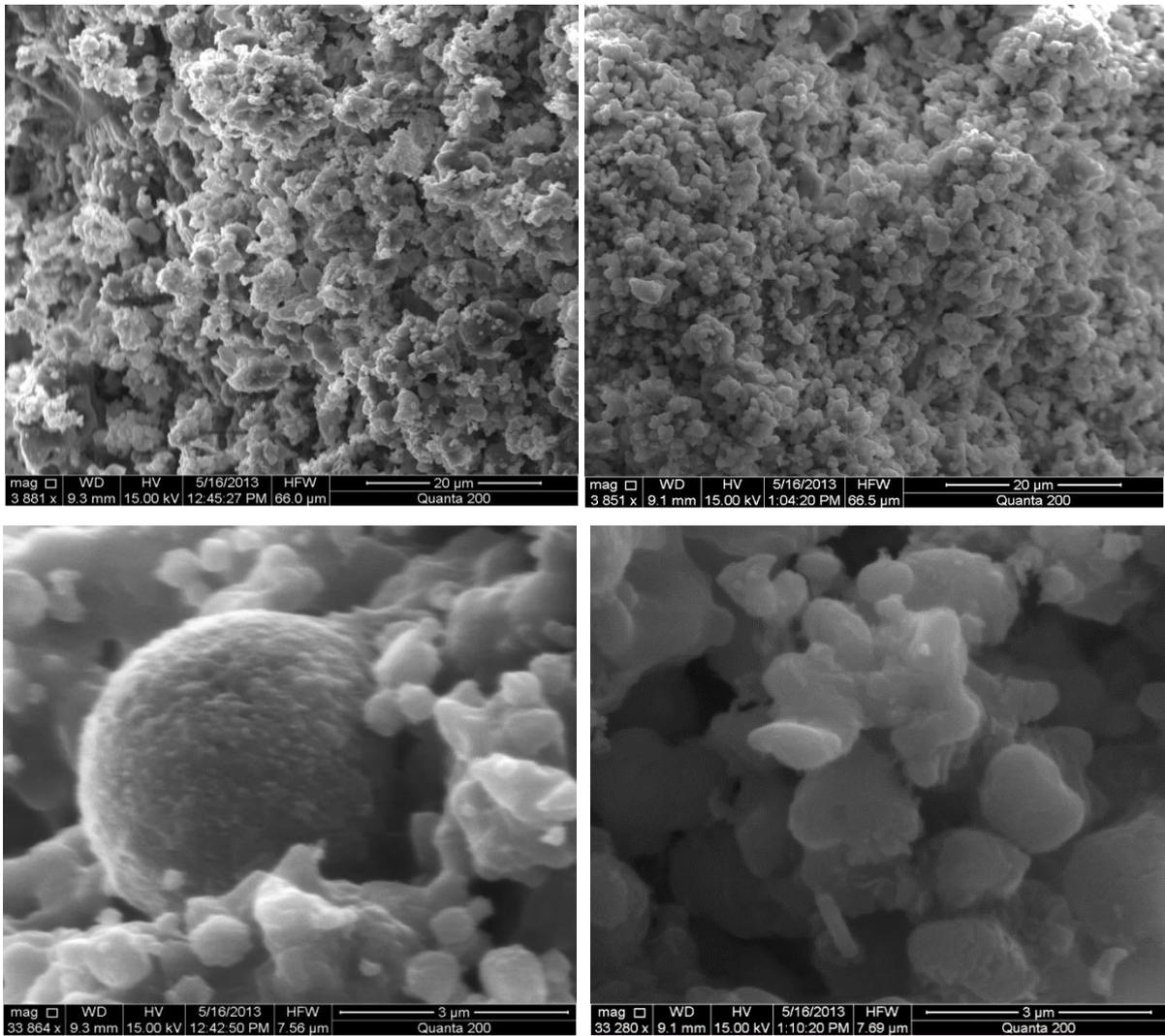
	CEM	FA 1	FA 2	CKD 1	CKD 2	GGBS
D ₅₀ (μm)	13.5	17.1	9.0	28.4	22.8	9.8
SSA (Blaine) (cm ² /g)	3493	4505	4092	1992	2489	4567
Density (g/cm ³)	3.205	2.096	2.220	2.871	2.734	2.750
Standard Consistency	29%	34%	25%	64%	49%	-
Activity Index	-	95%	106%	-	-	-



(a)

(b)

Figure 2: SEM images of (a) FA 1 and (b) FA 2 at different magnifications



(a)

(b)

Figure 3: SEM images of (a) CKD 1 and (b) CKD 2 at different magnifications

Table 2: Oxide proportions (%) of raw binder materials

	CEM	FA 1	FA 2	CKD 1	CKD 2	GGBS
SiO ₂	19.60	52.10	51.20	14.00	15.40	54.70
Al ₂ O ₃	4.71	19.60	24.34	3.92	3.80	40.87
Fe ₂ O ₃	3.25	7.10	10.17	2.27	2.55	0.80
CaO	64.00	4.40	2.79	56.80	54.10	0.02
MgO	1.17	2.00	1.46	0.94	0.97	0.24
Na ₂ O	0.27	1.06	1.28	0.44	0.56	0.20
K ₂ O	0.73	1.93	2.57	4.94	4.90	1.95
SO ₃	2.94	0.54	0.26	4.96	3.84	0.00
TiO ₂	0.26	0.87	1.01	0.22	0.23	0.02
LOI	3.22	9.50	4.30	10.20	13.20	0.94

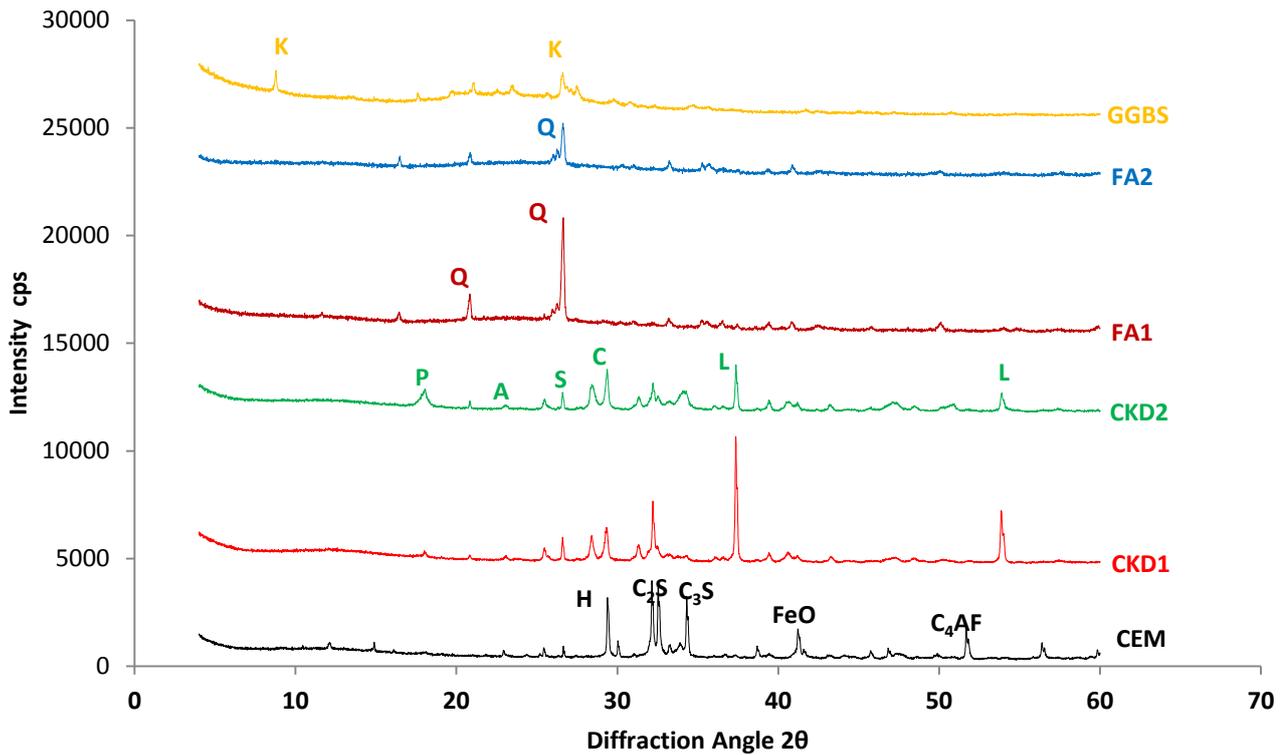


Figure 4: Powder XRD of raw binder materials (Q- quartz, C- calcite, L- lime, S- sylvite, H- hatrurite, P- portlandite, A- anhydrite)

2.2. Mix proportioning

A total of 15 concrete mixes were cast using various combinations of the binder materials as detailed in Table 3. Apart from the control mix (100C) that contained cement only within the binder, all other mixes contained 30% cement and the mix code describes the remaining binder constituents. The code for the other constituents should be interpreted as follows: A1 / A2 represents fly ash 1 / 2, KD1 / KD2 represents cement kiln dust 1 / 2 and S represents ground granulated blast-furnace slag. The number before each constituent represents the percentage of that constituent relative to the total binder content.

Apart from binder material proportioning, all other constituents were kept constant throughout the test programme. The total binder content was maintained at 580 kg/m³ and the water : binder ratio was 0.25 throughout. Several researchers have used similarly high binder contents and low water : binder ratios when developing HVFA concrete (Poon et al., 2000, Yazıcı, 2008). Fosroc Auracast 200 polycarboxylate based superplasticiser was added at a dosage of 1 lit / 100 kg binder

to all mixes. River gravel with a notional maximum size of 10 mm was used as the coarse aggregate and sharp sand with a notional maximum size of 5 mm was used as the fine aggregate. 60% of the coarse aggregate and 40% of the fine aggregate was used throughout and the particle size distribution plot of this aggregate proportioning is shown in Figure 5. A number of trial mixes were initially prepared to finalise the water content, superplasticiser content and aggregate proportioning to achieve a target slump of between 120 mm – 150 mm for the control concrete mix.

Table 3: Binder constituents of concrete mixes

Mix code	CEM kg/m³	FA 1 (kg/m³)	FA 2 (kg/m³)	CKD 1 (kg/m³)	CKD 2 (kg/m³)	GGBS (kg/m³)
100C	580	-	-	-	-	-
70A1	174	406	-	-	-	-
65A1-5KD1	174	377	-	29	-	-
60A1-10KD1	174	348	-	58	-	-
55A1-15KD1	174	319	-	87	-	-
60A1-10KD2	174	348	-	-	58	-
55A1-15KD2	174	319	-	-	87	-
55A1-15S	174	319	-	-	-	87
50A1-5KD1-15S	174	290	-	29	-	87
45A1-10KD1-15S	174	261	-	58	-	87
45A1-6.5KD1-18.5S	174	261	-	37.7	-	107.3
70A2	174	-	406	-	-	-
60A2-10KD2	174	-	348	-	58	-
55A2-15KD2	174	-	319	-	87	-
45A2-6.5KD1-18.5S	174	-	261	37.7	-	107.3

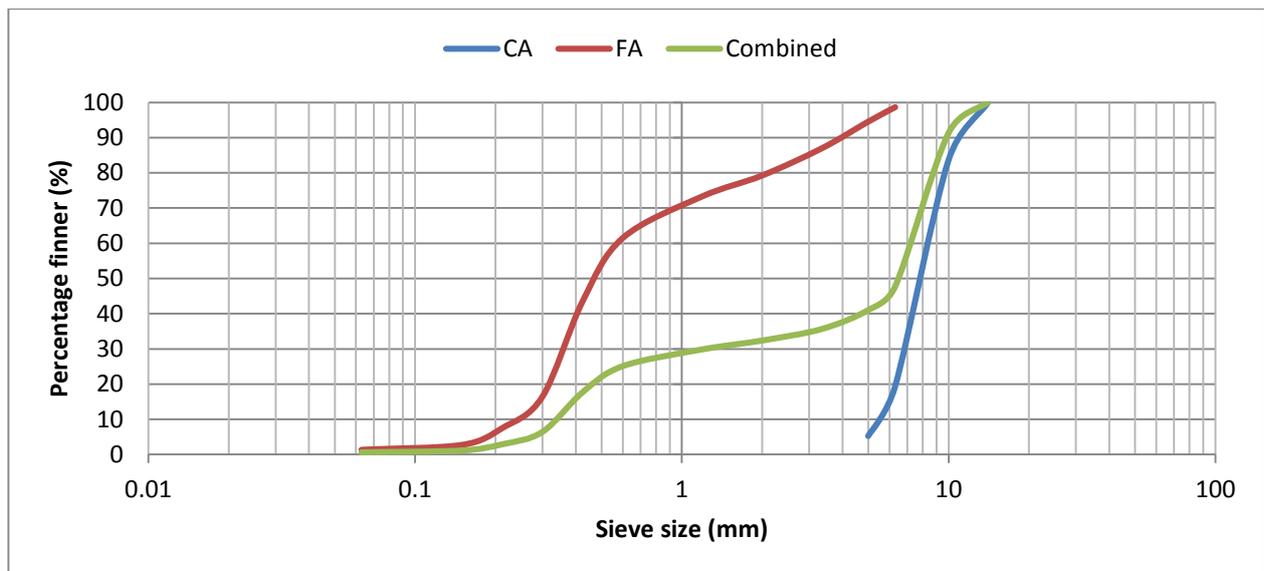


Figure 5: Particle size distribution of coarse aggregate (CA), fine aggregate (FA) and 60% coarse and 40% fine aggregate (combined)

2.3. Experimental procedure

All mixes were prepared in a pan concrete mixer and the dry constituents were mixed first. The superplasticiser was diluted with some of the mixing water as per the manufacturer’s specifications and added to the mixer, followed by the remaining water. Once mixed, nine 100 mm cubes were cast in accordance with BS EN 12390-2:2009 using a vibrating table compaction mode. Samples within moulds were covered with polythene to reduce moisture loss and hardened samples were demoulded after 24 hours. Cube samples were then cured under water at 20°C until required for compressive strength testing in accordance with BS EN 12390-3:2009 after 2 days, 7 days and 28 days after casting. All compressive strength results presented are the average of three samples.

3. Results and Discussion

3.1. Effect of CKD content

Figures 6 and 7 show graphs of compressive strengths for mixes that contain varying amounts of CKD with fly ash 1 and 2 respectively. Compressive strengths of mixes containing 100% cement are also included in Figures 6 and 7 for reference. However, it is acknowledged that a cement content

of 580 kg/m³ produces a high strength concrete whereas the aim of the HVFA concrete mixes is to achieve the compressive strength of a structural concrete grade with moderate early age strength.

For fly ash 1, contents of 0%, 5%, 10%, and 15% CKD 1 were tested and both 2 day and 7 day strength generally increased with CKD content (although there was little difference in the early age strength of mixes with 5% and 10% CKD). The highest 28 day strength for the same mixes was achieved by the mix containing 10% CKD. For fly ash 2, contents of 0%, 10%, and 15% CKD 2 were tested and the highest strength at all ages was achieved by the mix containing 10% CKD with the most significant difference in strength between mixes at 28 days. Replacing 10% of the fly ash within the binder with CKD caused an increase in 28 day strength of 28.2% and 20.8% for fly ash 1 and 2 respectively (relative to the strength of the mixes containing 70% fly ash and 30% cement). Results here show that inclusion of more than 10% CKD begins to have a negative effect on compressive strength, particularly at 28 days.

Table 2 shows that both batches of CKD have appreciably higher alkali contents than the cement used (Na₂O equivalents of 3.69 for CKD 1 and 3.78 for CKD 2 compared to 0.75 for cement). Increased alkalinity of the blend due to incorporation of CKD accelerates the dissolution of the glassy phase of fly ash. Also, high sulphate contents of the CKD's increase stable ettringite formation when used with fly ash (Wang et al., 2004). These effects have contributed to observed increases in strength.

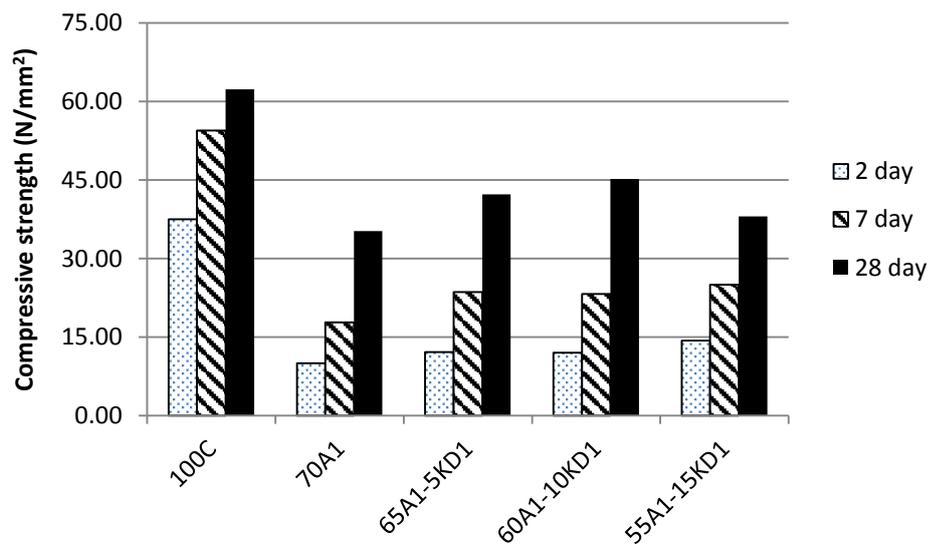


Figure 6: Compressive strengths of fly ash 1 mixes with varying amounts of CKD 1

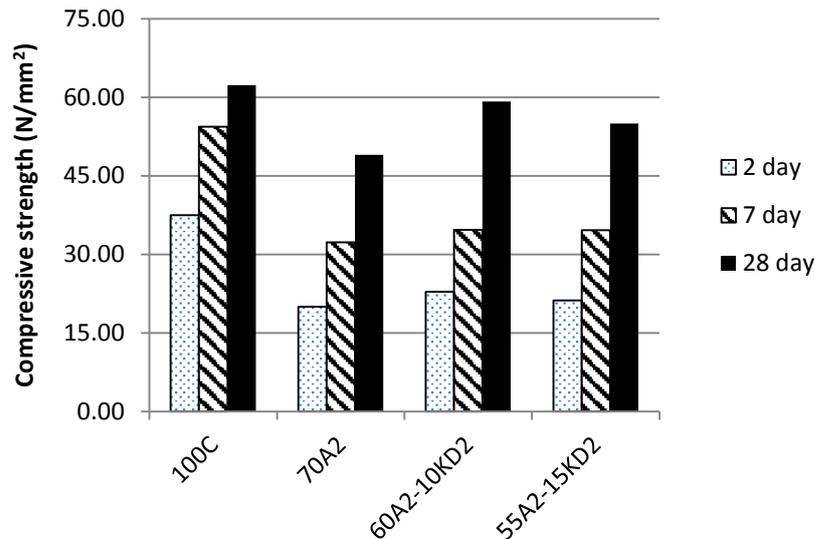


Figure 7: Compressive strengths of fly ash 2 mixes with varying amounts of CKD 2

3.2. Effect of inclusion of GGBS

Figure 8 shows compressive strengths of fly ash 1 mixes that include varying amounts of GGBS and in some cases, CKD 1. Firstly, it is noted that replacing 15% of the fly ash content with GGBS (without inclusion of CKD) caused increases in strength at all ages. This correlates with the findings of Zhu et al (Zhu et al., 2012) and Li and Zhao (Li and Zhao, 2003) that replacing moderate amounts of fly ash with GGBS within the binder enhances strength gain. For quaternary blends that include CKD, increases in compressive strength at all ages were observed. 2 day strengths of quaternary blends were impressive (all in excess of 16 N/mm²) and 28 day strengths were on a par with that for the 100% cement mix.

Inclusion of CKD increases the reactivity of the GGBS as well as the fly ash within the blend. Chaunsali and Peethamparan (Chaunsali and Peethamparan, 2013) investigated the hydration process of CKD – fly ash binders and found that crystalline hydration products after 1 day were ettringite, calcium hydroxide and gypsum. The same phases were also observed after 1 day in an earlier study on CKD – GGBS binders (Chaunsali and Peethamparan, 2011). In these studies, ettringite formation was identified as contributing significantly to strength development, particularly at early age.

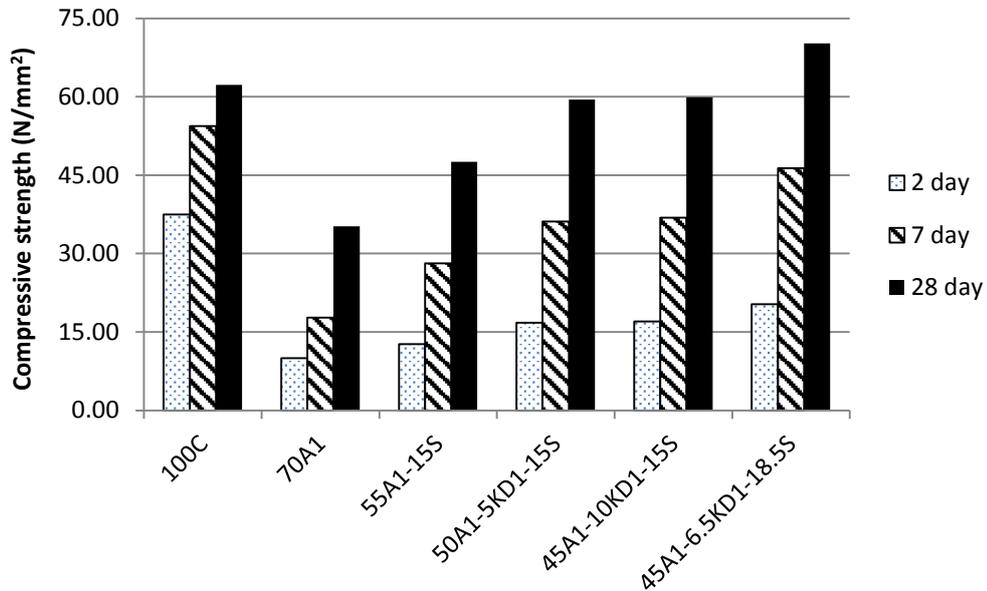


Figure 8: Compressive strengths of fly ash 1 mixes containing GGBS

3.3. Effect of fly ash variability

When examining the particle size distribution plots of fly ash 1 and 2 from Figure 1, it is apparent that fly ash 2 is composed of finer particles throughout the particle range. However, it is noted that the measured Blaine fineness from Table 1 contradicts this observation. The water demand of fly ash 1 is higher based on standard consistence measurements from Table 1 and Felekoglu et al (Felekoglu et al., 2009) reported that particle size, shape, surface morphology and porosity all significantly affect ash water demand. It is apparent from SEM images in Figure 2 that fly ash 2 is composed of more fine spherical particles than fly ash 1 which supports observations from the particle size distribution plots. Also, the presence of large flaky shaped unburned organic matter was apparent in fly ash 1. It is also noted that the activity index for fly ash 2 was higher than for fly ash 1.

Table 2 shows that fly ash 1 had a significantly higher LOI content (greater than the maximum limit of 9% in BS EN 450-1:2008) than fly ash 2. Figure 9 shows images of both raw fly ashes and the darker colour of fly ash 1 can be attributed to the presence of organic matter (Cockrell, 1972). The higher unburned carbon content of fly ash 1 is likely to have contributed to its observed higher water demand (Rajamma et al., 2009). Higher concentrations of CaO and MgO and lower concentrations of Al₂O₃ and Fe₂O₃ were identified in fly ash 1 (relative to fly ash 2) but the concentration of SiO₂ was similar in both ashes. Major phases identified in fly ash samples were

quartz (SiO_2) and mullite ($2\text{Al}_2\text{O}_3\text{SiO}_2$) with minor phases of gypsum and magnetite. The reduced crystal peaks of fly ash 2 (relative to fly ash 1) are also notable from Figure 4. The presence of increased pozzolanically active amorphous phases in fly ash 2 was thought to be dominant in this reduction in crystal phases (Jones et al., 2006, Sadique et al., 2012).

Figure 10 shows compressive strength results for pairs of concrete mixes from the experimental programme where the only difference between a mix pair is the fly ash used. Comparison of strength results within a mix pair can highlight the interaction of both fly ashes with cement only, with cement and CKD 2 and with a combination of cement, CKD 1 and GGBS. Generally, fly ash 2 mixes were stronger than the corresponding fly ash 1 mixes at all ages (with the 28 day strength of mix 45A1-6.5KD1-18.5S being the exception). The higher proportion of amorphous phases relative to crystalline phases in fly ash 2 is thought to be the main contributory factor to observed higher compressive strengths for fly ash 2 mixes. However, the finer particle size and lower water demand (which would enable better concrete compaction for a fixed water content) would have also contributed to the superior performance of fly ash 2 mixes.

Use of CKD 2 with fly ash 1 appeared to have a negative effect on strength (relative to the 70% fly ash 1 and 30% cement mix). However, blending CKD 2 with fly ash 2 caused an appreciable increase in compressive strength at all ages. Figure 11 shows the difference in appearance of concrete cubes cast with both ashes blended with 10% CKD 2. Crystalline efflorescence is apparent on the surface of the 60A1-10KD2 cube which may have adversely affected compressive strength. Upon contact with water, ionic species from alkali sulphates (K^+ , Na^+ and SO_4^{2-}) within CKD dissolve in the liquid phase due to high solubility and form hydrates. A high ratio of SO_4^{2-} to available $\text{Al}(\text{OH})_4$ from the aluminate phase favours the formation of ettringite (Aft) (Taylor, 1997). The lower ionic bond between aluminate phase and alkali sulphates was thought to be responsible for dissolution of alkali salts and the observed efflorescence on the cube surfaces. The higher visual porosity and surface fragmentation caused by the higher water demand of fly ash 1 would have also significantly reduced compressive strength.



Figure 9: Difference of colour between fly ash 1 and fly ash 2

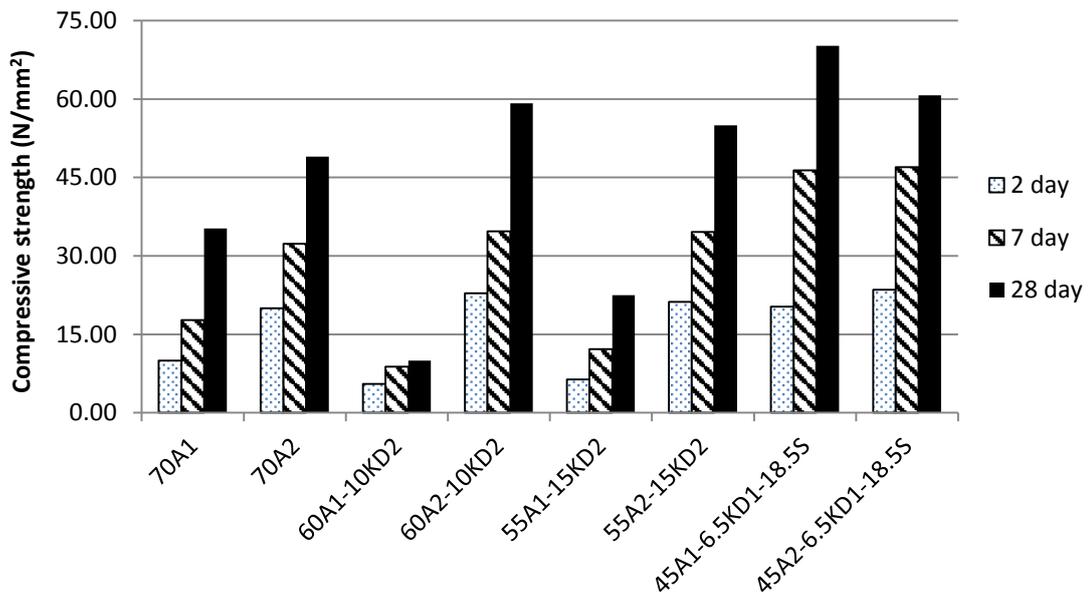


Figure 10: Compressive strengths of selected fly ash 1 mixes and corresponding fly ash 2 mixes

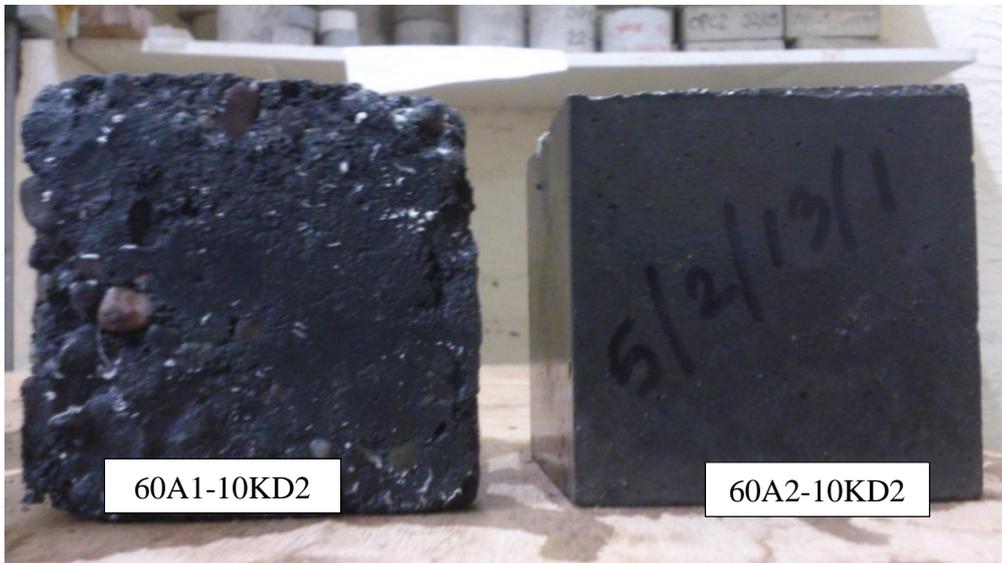


Figure 11: Difference of appearance of fly ash 1 and fly ash 2 cubes activated with CKD 2

3.4. Effect of CKD variability

Particle size distribution plots in Figure 1 show that CKD 2 is finer than CKD 1 throughout the particle size range. This is supported by the Blaine fineness results from Table 1 and also SEM images from Figure 3. Table 1 also shows that CKD 1 had a significantly higher water demand than CKD 2.

When comparing the oxide concentrations for CKD 1 and CKD 2, the main differences are the higher CaO and SO₃ concentrations and lower LOI content of CKD 1, Table 2. However, the reduced crystal peaks of CKD 2 relative to CKD 1 would suggest that CKD 2 is likely to be more reactive, Figure 4. In both CKD's, major phases of lime (CaO), portlandite (Ca(OH)₂), quartz (SiO₂), anhydrite (CaSO₄) and sylvite (KCl) are present.

Figure 12 shows compressive strength results for fly ash 1 concrete mixes activated with CKD 1 and corresponding mixes activated with CKD 2. The low strengths of mixes that combine fly ash 1 with CKD 2 are discussed in section 3.3. Significantly higher strengths were observed when fly ash 1 was blended with CKD 1. Chaunsali and Peethamparan (2013) identified higher free lime content and higher sulphate content of CKD improves strength development when blended with fly ash or GGBS. They found that more ettringite formed when the CKD with higher sulphate and alumina contents was used and that higher free lime contents of CKD increased the development of C-A-S-H gel. In this investigation, the higher sulphate content of CKD 1 would have contributed to observed

higher compressive strengths. However, the physical properties of CKD 1 (coarser particle size and higher water demand) contradict the expected influence on compressive strength. As CKD is a highly variable material (Kunal et al., 2012), further research on the effect of a broader range of physical and chemical properties of CKD on the strength development and long-term performance of HVFA concrete relating to any possible adverse effects of high amount of alkalis and sulphates is required. The further investigation of hydrates using SEM, XRD and TGA will also provide definitive understanding of future performance of HVFA concrete.

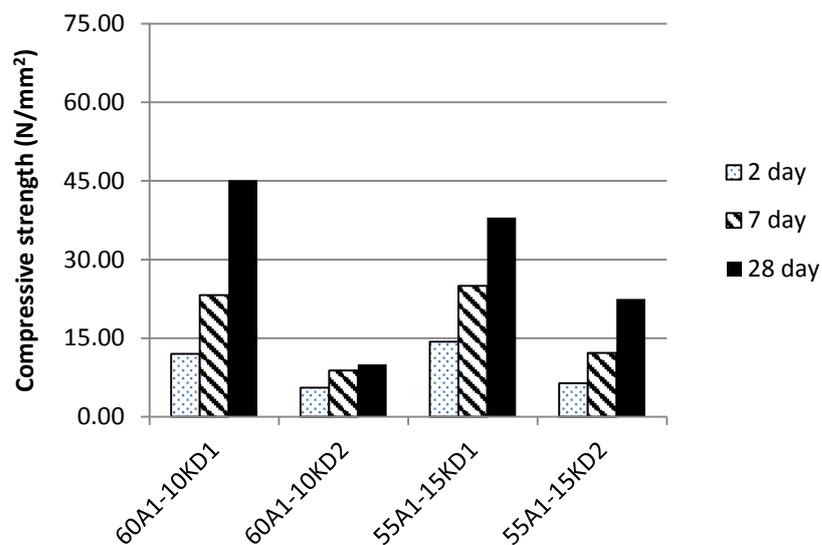


Figure 12: Compressive strengths of selected fly ash 1 mixes with corresponding amounts of CKD 1 and CKD 2

4. Conclusion

From the results of the current investigation, the following conclusions can be drawn:

1. When considering ternary blends of cement, fly ash and CKD, 10% of CKD blended with 60% fly ash and 30% cement appeared to be a favourable binder proportioning in developing both early age and 28 day compressive strength.
2. Notably high strengths were observed at all ages for quaternary blends that include 15% - 18.5% GGBS and enhanced strength gains are primarily attributed to ettringite formation.

3. Of the two ashes tested, the finer ash of lower water demand led to greater concrete strengths. The lower LOI content and higher proportion of amorphous phases of this ash also contributed to increased strengths.
4. Of the two CKD's tested, the CKD of higher CaO and SO₃ content resulted in increased activation of fly ash. However, as CKD is a highly variable material, further investigation of a broader range of physical and chemical properties of CKD's in association with XRD and TGA analysis of hydrates at different ages is suggested. Analysing any possible adverse effects of high amount of alkalis and sulphates on long-term performance of this HFVA concrete also suggested.

5. Acknowledgment

This work was supported by the Engineering and Physical Sciences Research Council (EPSRC) under Grant [EP/J016055/1]. The authors gratefully acknowledge the Engineering and Physical Sciences Research Council for the funding provided.

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