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Evaluation of activated high volume fly ash systems using Na₂SO₄, lime and quicklime in mortars with high loss on ignition fly ashes

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ABSTRACT

In general, concretes made with blended Portland cement containing high volumes of fly ash provide an alternative to conventional Portland cement concrete to enable carbon footprint reduction. This study evaluates the chemical activation of four fly ashes in blends with Portland cement, by assessing their effects on hydration and compressive strength. In this study, a sieving process is used to regulate the fly ash composition, which has an effect in the chemistry and reaction of the mix. The results show the importance of the amorphous content of the fly ash with respect to achieving a high compressive strength. The effect of sodium sulfate, added as an activator, is significant in terms of compressive strength at early age for two of the fly ashes studied; in this case, the parameter used to correlate with the compressive strength evolution is the amount of portlandite consumed through pozzolanic reactions. However, sodium sulfate does not have the same effect on fly ashes with a high amount of Fe₂O₃, in which portlandite consumption is much lower.

Keywords: High volume fly ash, sodium sulfate, lime, quicklime, loss on ignition

1. INTRODUCTION

 CO_2 emissions have become the main environmental concern world-wide in recent years; a report from the PBL Netherlands Environmental Assessment Agency concluded that 35.7 billion tonnes of CO_2 were produced in 2014 **[1, 2]**. This number is prompting various industries to focus on reducing CO_2 emissions through implementation of new technologies or innovations. The impact of cement production is approximately 8% of total global CO_2 emissions **[2]**. There are many approaches to reduce this emissions footprint, one of the most remarkable of which is the use of supplementary cementitious materials (SCMs) in the concrete mix to replace a significant fraction of the Portland cement, resulting in mixes which range from high volume fly ash or slag concretes, to alkaliactivated materials or geopolymers **[3, 4]**; it is important to consider that in some cases there is an increase in costs depending on transportation, fly ash source and activator, and that these factors can also contribute to the CO₂ footprint **[4]**. Currently the most commonly used fly ash percentage in Portland cement blends is about 20% to 30%, and researchers have extensively studied mixes with fly ash content higher than 30% with the view of extending this range upwards **[5-8]**. Although there is environmental pressure to increase the percentage of fly ash in concrete mixes, it is important to study technical concerns such as strength development and durability when seeking to increase the cementitious material replacement to levels as high as 60% to 80% **[9]**; some of the main issues to study are related to early strength, setting time, resistance to chloride ingress and carbonation, and extra costs resulting for example from high curing temperature or high superplasticizer dosages in ready-mixed concrete production **[10]**.

When a high volume fly ash concrete is designed, it is necessary to consider a reduction in the water to cementitious material ratio; as this is reduced, superplasticizer is increased to maintain the same slump [11, 12]. Although such a concrete is considered a 'green' alternative, it is not an optimal one in many instances due to the increased consumption of a high-cost superplasticizer. Moreover, setting time and early strength are affected, and at some point become critical issues to be controlled. Researchers have also studied the possibility of using binders based on 100% fly ash with the use of activators other than Portland cement (PC) [13-16]; some of these studies identified the need to cure at elevated temperatures to reach target strengths in cases where the fly ash reactivity was not high [17]. Intermediate mixes, whose fly ash percentages are between 50% and 100%, have been studied [18] considering setting time, rheology, and early strength evolution.

The addition of calcium sulfate anhydrite (CaSO₄) in conjunction with 55% fly ash as a cement replacement was studied using different curing methods **[19]**; curing at 65 °C for 6 h before normal curing allowed the strength to increase by 70% compared to a control mix after 3 days. Large amounts of ettringite were found at early age. Gypsum was also used in this study; although gypsum was more effective than anhydrite in yielding later age strength, anhydrite increased early age strengths significantly. No technical reason for this performance was given, and some further research was recommended. The necessity of researching the durability of these concretes was also emphasized. Activation of Portland-fly ash and lime-fly ash cements was accelerated using Na₂SO₄, resulting in increased compressive strength at early ages **[20]**. Na₂SO₄ had a better effect when using a higher fly ash percentage, and a grinding process applied to the fly ash was shown to increase the strength significantly.

A mix with 30% Portland cement, 70% fly ash and NaOH with sodium silicate could achieve similar strengths compared to a control mix with 70% Portland cement and 30% fly ash [21]. This effect does not occur when using NaOH alone as an activator. Although sodium silicate allows the target strength to be achieved, this activator does increase the slump loss [22].

Sodium silicate is in general one of the most effective activators for fly ash. This activator is widely used in different applications such as adhesives, well cements, acid resistant concrete and shotcretes **[23]**, and has a plasticizing as well as accelerating effect. CaCl₂ is also an effective activator; it reduces the solubility of Ca(OH)₂, which accelerates the dissolution of fly ash particles, thus affecting the pozzolanic reaction and is more effective at later ages **[24]**, but chloride addition is unsuitable for applications involving steel reinforcing. K₂SO₄, Na₂SO₄, and triethanolamine were also shown to improve early strength development **[25]**. The same study demonstrated a decrease of calcium hydroxide and an increase of ettringite content when using 40% fly ash replacement in cement. The latter effect helped reduce the pore size. The strengths were in most cases similar to the sample without activator; K₂SO₄ had the best effect, increasing the strength significantly.

On the other hand, unburned carbon present in the fly ash can affect concrete consistency. An increment in the loss on ignition (LOI) of the fly ash increases the water requirement for a given consistency [26]. The same carbon cellular particles which affect the water content also affect the air content, making it necessary to increase the air entraining admixture significantly to achieve the desired air content [27]. Although these non compliant fly ashes could have an effect on concrete water or admixture demand, it is evident they have a positive effect as a cementitious material and can be used in different concrete applications [28, 29]; for instance, they have been evaluated to be used in pervious concretes [29].

Considering the current state of the art, the main objective of this study was related to understanding the reactivity of different high-loss on ignition fly ashes at a 50% PC replacement level in the presence of Na₂SO₄, lime and quicklime as activators. The binder phases, hydration reactions and the effect of mix design and curing duration on the compressive strength of samples cured at 23 ℃ were evaluated for different fly ash compositions.

2. MATERIALS AND METHODS

2.1. Materials

The fly ashes named as TP, FB and TG are high-LOI fly ashes, whereas TA is a reference low-LOI North American fly ash. Table 1 presents the chemical compositions of these fly ashes. TA has the highest silica content among the fly ashes. All of the fly ashes reach the ASTM C 618 Class F requirement for the sum of major oxides (SiO₂, Al₂O₃ and Fe₂O₃) **[30]**, where TA has the highest value and FB the lowest. However, it is not possible to directly classify them as Class F fly ashes, because all exceed the basic maximum 6% LOI requirement for that classification, although all do fall within the 12% absolute maximum which is allowed subject to meeting performance criteria. The FB fly ash has 12% LOI, whereas TP and TG are between 8% and 10%. TA is the only one that receives beneficiation treatment after production in the thermoelectric plant, and so has the lowest LOI percentage of 1.53%. The sodium sulfate activator has a purity of 95%.

The mineralogical composition of each of the fly ashes, and of the Portland cement (Type III according to ASTM C150 **[31]**) is presented in Table 2 and Table 3 respectively. Figure 1 presents XRD data for all fly ashes studied. The highest amorphous content is displayed by TA. All of the fly ashes contain quartz and mullite as the main crystalline phases; TA also contains a notable content of crystalline iron oxides, while FB contains a minor phase identified mineralogically as hatrurite, but which is likely to be tricalcium silicate (C_3S) introduced via a small amount of contamination by Portland cement in processing or transport.

2.2. Sample preparation

Table 4 includes the mix proportions for mortars. Although most of the procedure presented in ASTM C 109 **[32]** was followed for mortar preparation, some special considerations must be mentioned:

- The activator was added along with the mix water; it was mixed until it was completely dissolved using a Hobart mixer (Model N50). Sodium sulfate and lime had good solubility, whereas quicklime in contact with water increased the temperature and formed white solid forms. This effect increased with increasing dosages. Portland cement and fly ash were added to the mix of water and activator; it was mixed for 30 s at the lowest speed setting (level 1, 140 min⁻¹). After that, sand was added and mixed for 30 s. It was then left for 1.5 min and mixed again for 1.5 min at a speed setting of 2 (285 min⁻¹).
- Samples were cast into steel 50 mm cube moulds, which had been coated with an organic mould release agent, and then vibrated to remove excess entrapped air.
- Samples were cured in the moulds at 100% humidity in a curing room at a temperature of 23 °C.

2.3. Analytical characterization

The chemical composition was determined using a PANalytical Axios sequential wavelength dispersive X-ray fluorescence (XRF). The mineralogy was evaluated by X-ray diffraction (XRD) using a PANalytical X'PERT-PRO MPD in Bragg-Brentano configuration with an X'celerator detector. The Rietveld method was used to quantify the crystalline phases via the X'Pert HighScore Plus software package; a 10 wt % spike of rutile was used as an internal standard for the amorphous content quantification.

Samples of 60 mg were used for the thermogravimetry procedure and The Instrument used was a Thermogravimetric Analyzer TGA 2950. Tests were performed with a controlled nitrogen atmosphere with a 40 mL/min flow. The temperature was increased from ambient to 950 °C at a rate of 10 °C/min. These results were analysed using TA Universal Analysis software, including determination of the numerical derivative of the mass loss curve (i.e. differential thermogravimetry). Isothermal calorimetry was performed using an I-Cal8000 from Calmetrix. Heat flow was measured for 24 hours to visualise the effect of the fly ash and its interaction with lime, quicklime and sodium sulfate.

3. RESULTS AND DISCUSSION

3.1. Initial characterization

The composition of fly ash changed upon increasing its fineness after a sieving process (<74 μ m and <45 μ m); the amorphous material and LOI contents were different for each fineness. Table 5 summarizes the effect on the main parameters. By sieving the material, not only did the particle size decrease but also the LOI content was reduced. OS (original size) TA has the lowest LOI and the highest SiO₂ amongst all the fly ashes; this is due to the fact of being a commercial fly ash and the treatment (i.e LOI reduction) it receives from the fly ash supplier. In contrast, OS FB includes the highest LOI and lowest SiO₂ content, due to the combustion system used by the energy producer and the fact of not being improved or beneficiated by any pre-treatment.

The strength achieved by each mortar formulation varied depending on the fly ash amorphous content: Figure 2 shows how the compressive strength of mortar samples with 20% fly ash was improved when the amorphous content of the ash increased, across the full set of ashes analysed. Three points are shown in Figure 2 for each ash, representing the original as-received material, and the ash sieved to pass 74 μ m and to pass <45 μ m. For TP and TG, when the average particle size was reduced by sieving, the LOI content also decreased, but the compressive strength decreased because the amorphous content was lower. No changes in the water/cement ratio were needed between samples with sieved and unsieved ashes, as the water demand was not changed by the sieving process; in this case, the effect of the LOI was not evident in terms of water demand. However, it appears that depending on the amorphous content of a fly ash, mechanical treatment may not be necessary to achieve reasonable strength, even for these ashes which are relatively coarse and display high LOI values. The results of similar analysis based on reactive SiO₂ content, on reactive Al₂O₃ content, on fineness, and on LOI, did not show correlations as direct as the relationship based on the total amorphous content [33], and therefore it is hypothesise that this is the dominant factor controlling fly ash reactivity in these systems.

3.2. Compressive strength

Table 6 presents the mix designations and the scope of the variables investigated. As seen in Figure 3a, the compressive strength of the sample with 50% TP using Na_2SO_4 was improved by approximately 40% compared to the control without activator (TP/OS/50), as the activator has evidently enhanced the reaction process during the first 3 to 7 days.

When quicklime and lime were added to the mix, there was little effect on the compressive strength. The optimal dosage was found to be 3% for both quicklime and lime. Comparing quicklime and lime, the former had a higher compressive strength after 3 days, even passing that of the control sample (TP/OS/50); after 7 and 28 days, the lime mix had a higher compressive strength, and by 56 days, the quicklime mix improved its strength as seen in Figure 3a. Antiohos *et al.* **[34]** used quicklime as an activator with fly ashes of both Class C and Class F, and observed that not only did this appear to help to create physical CH connections between fly ash particles, but also the solubility of SiO₂ was improved; this increased the compressive strength with Class C fly ash, but the effect was negative when using Class F fly ash **[34]**. Considering the compressive strength evolution using lime and quicklime, Shi **[35]** found that there was an optimal portlandite level; when it was passed, the Ca(OH)₂ that was not involved in any reaction weakened the matrix. Shi also found that the initial heat released from the reaction between quicklime and water helped to accelerate the pozzolanic reaction, and that the Ca(OH)₂ resulting from this hydration of quicklime could be more soluble than manufactured lime **[35]**.

Figure 3b shows how sodium sulfate increased the compressive strength as the activator dosage increased. These results were compared against those of the FB/OS/50 control sample, and the highest strength with activator almost doubled the performance of the un-activated control; FB has a high amorphous content and could affect the activation process. Sodium sulfate dosage had the same effect on both TP and FB; there was not a significant variation in the strength at 1% and 1.5% addition levels, but a significant improvement was observed upon increasing the dosage to 3.5%.

Although quicklime increased the compressive strength at a dosage of 3% when used with FB, it did not have the same effect as sodium sulfate: Figure 3b presents a tendency in which a significant increase in strength was only observed after 28 days, which seems to contradict the observations of Shi **[35]** regarding an early-age accelerating effect of quicklime. Lime mixes had low strengths compared with the control. Although quicklime and lime mixes had a delayed effect compared to sodium sulfate mix, the strengths seemed to be improved at later ages, which suggests an enhancement of the long-term pozzolanic reaction rather than any significant activation effect. This is consistent with the well known early-age stability of portlandite in fly ash-blended cement systems, as the clinker reacts to yield a pore solution environment which rapidly becomes saturated with respect to portlandite before the fly ash gradually starts to react and consume it via the pozzolanic reaction, and so the addition of extra calcium hydroxide has little effect on early age strength development.

For most of the activators used in conjunction with TG, the highest dosage was the most effective. Mixes with sodium sulfate and TG had a high variability between results at different ages as presented in Figure 3c), although there was a tendency to obtain a consistent strength evolution with a dosage of 3%. Mixes with quicklime (TG/OS/50/Q/5) and lime (TG/OS/50/L/5) had the best performance with a dosage of 5%; although the compressive strength in some cases could not surpass that of the control samples, the evolution from 7 to 28 days was significant.

Sodium sulfate improved the compressive strength compared with the control mix with TA as presented in Figure 3d. The mix with 1% sodium sulfate had acceptable behavior at different ages, and the sodium sulfate activator seemed to induce this fly ash to react at early ages. The other two activators investigated, lime and quicklime, generally did not achieved the strength of the control mix using the different fly ashes studied.

Overall, it is clear that Na_2SO_4 had a positive effect on the activation process. This was also evident by the work of Qian *et al.* **[20]**, who evaluated the effectiveness of this activator with HVFA mixes and found that Na_2SO_4 reacted directly with the $Ca(OH)_2$, increasing the alkalinity and accelerating fly ash dissolution; SO_4 increased the formation of ettringite, affecting the density of the mortar matrix positively **[20]**. Donatello et al. **[36]** also showed a significant accelerating effect from addition of Na_2SO_4 as an activator in binders containing 80% fly ash and 20% Portland cement, although at their very low clinker content the addition of Na_2SO_4 also suppressed ettringite formation, which was not observed in this study (see section 3.5 below), and this indicates differences in the chemical mechanisms induced by ettringite growth depending on the relative availability of aluminate from C_3A at early age.

3.3. Isothermal calorimetry

Figure 4 presents the observed heat evolution from each of the formulations studied during the earliest period of reaction, with a focus on the pre-induction, induction and acceleration periods. FB had the highest first peak (mixing peak) with quicklime. Generally, delays in the reactions leading to the acceleration period increase with increasing fly ash content, which is due to the dilution of PC. Wei et al **[37]** also mentioned a possible delay due to the reaction of the Ca from the solution with the aluminum on the

fly ash particle surfaces. According to Figure 4, when quicklime and lime were included, the setting time decreased; the acceleration peak was reached in less time. Sodium sulfate increased the peak of the heat flow by 0.4 mW/g but there was a delay in the final setting time of approximately 2 h compared with the mix FB/OS/50; because this activator introduced sodium and sulfate into the mix, a reaction with fly ash aluminates was expected similar to the way this aluminum content could react with calcium from the solution **[37]**. This again differs from the results of Donatello et al. **[36]** who found a strong acceleration in early setting through the addition of Na₂SO₄ to a cement containing 80% fly ash. It seems that because the 50% Portland cement content here is sufficient to give a relatively normal setting behavior with the fly ash acting as a filler at early age, the Na₂SO₄ gives a slight retardation of the onset of the acceleration period but a higher maximum rate of heat release (around 15-18 hours), and this higher rate of reaction once the acceleration period does commence is responsible for the increased strength of these cements from 3 days onwards.

3.4. Thermogravimetry

The calcium hydroxide content of each paste was determined using thermogravimetry, and the results are summarised in Figure 5. The Ca(OH)₂ content began to decreased before 7 days of curing when using TP with sodium sulfate (compare TP/OS/50/A/1/3 and TP/OS/50/A/1/7), whereas with quicklime and the control mix with 20% fly ash (TP/OS/20/0/0/7), this occurred after 7 days. The fact that portlandite consumption started earlier using sodium sulfate, shows its influence as an activator for fly ash, and this is consistent with the longer-term strength development data; the fly ash glass phases are broken down with this activator due to a more highly alkaline environment, accelerating the consumption of Ca(OH)₂ [25]. Owing to nucleation and the seeding effect [22], the amount of Ca(OH)₂ per mass of cement for mixes including 50% fly ash was higher than the mix with 100% cement, as only limited pozzolanic reaction had taken place within the 28 day timeframe of the TGA experiments.

3.5. X-ray Diffraction

From the XRD evaluation (Figure 6), the amorphous and crystalline phase content of each of the hydrated binders was quantified (Table 7). It was found that the content of tobermorite was the highest in the sample with 100% cement at 3 days. Portlandite content was the highest for the mix with 100% cement followed by the mix with lime. Portlandite was almost halved for mixes with activators during the first 3 days. Sodium sulfate had an effect on ettringite formation; as shown in Figure 6 and Table 7, most mixes with sodium sulfate had the highest ettringite content, which is in contrast to the results of Donatello et al. **[36]** at higher fly ash content as noted above. Unreactive crystalline phases such as quartz and mullite, and the amorphous content appeared in high proportions for mixes including fly ash. The quartz and mullite content remained proportional for mixes including 50% fly ash compared with the original ash, providing an internal cross-check on the quality of the quantification results and demonstrating again that these phases are not consumed as the glassy component of the fly ash reacts. The amorphous content is composed of the combination of C-S-H and the residual fly ash amorphous fraction.

3.6. Scanning electron microscopy

From SEM images, including those shown in Figure 7, it can be deduced that sodium sulfate promoted ettringite formation in these cements. Figure 7 shows ettringite formation over fly ash particle surfaces of TP at ages of 7 and 28 days, when 50% fly ash was used with sodium sulfate. The presence of portlandite and C-S-H is also evident in these SEM images.

3.7. The importance of Fe₂O₃

As noted above, mixes with TP and FB activated by sodium sulfate produced more ettringite than the others; this helped to increase initial compressive strengths. Correspondingly, the amount of portlandite consumption was higher for these mixes at an early age. However, for mixes with TG and TA this did not occur. The main apparent differences between these groups of fly ashes is the high Fe_2O_3 content of TG and TA, and so it may be deduced that this had a negative effect on the activation process. The mechanism by which this may have happened remains unclear, and the role of Fe supplied by fly ash in determining its reaction in alkali-activated binders is far from fully understood, as the Fe in glassy fly ash phases is likely to reduce the rate of their dissolution according to standard glass chemistry arguments. However, once it does dissolve, it may also play an active role in forming reaction products in the hydrated cement, as is the case for Fe supplied by C_4AF in the cement. Thus, it is important that the effect of Fe_2O_3 content should be studied in more detail to understand its influences, both negative and positive, in hybrid cementitious systems activated by sodium sulfate, as this remains an area with many open questions.

4. CONCLUSIONS

According to the results of this study, the amorphous content of fly ash was a key factor controlling compressive strength development for mixes with low fly ash content (20%) and without any activator. By increasing fly ash fineness, its composition was also changed due to the heterogeneity of the ash and unequal distribution of phases between size fractions; the measured amorphous and LOI contents varied depending on the fineness following sieve separation. The compressive strength of mortar samples was improved when the amorphous content increased; in some cases, however, when the particle size and the LOI content decreased, the compressive strength decreased, which probably occurred because the amorphous content of the fine fraction of some of the ashes studied was low.

The effect of sodium sulfate on the reaction of hybrid cement mixes containing TP and FB was significant at initial ages. The increased amounts of ettringite and the accelerated portlandite consumption were reflected in the compressive strength evolution. However, sodium sulfate did not have the same effect on fly ashes TG and TA, and the amounts of ettringite formation and portlandite consumption were not as significant as those for the first two fly ashes. The main difference between these fly ashes was the higher amount of Fe_2O_3 in ashes TG and TA compared to the others investigated.

Portlandite consumption was one of the main indicators of fly ash reaction; portlandite consumption commenced after 3 days when using sodium sulfate with 50% of either TP and FB blended with Portland cement. For most of the cases studied, portlandite production in mixes including lime and quicklime continued to increase after 7 days. In the case of ettringite, its formation helped improve the compressive strength in the first days for mixes with TP and FB; for these mixes, the formation of C-S-H increased, becoming measurable by XRD as part of the amorphous content at later ages. Mixes with sodium sulfate had the highest ettringite and amorphous contents. Samples with lime and quicklime produced ettringite in low proportions, and the amorphous content did not increase significantly with time.

The reference fly ash (low loss on ignition) did not perform better than the high-LOI ashes as expected. The effect of the Fe_2O_3 content is highly relevant to the activation process, but remains poorly understood; this appears to reduce the speed of dissolution of the reactive components of fly ash.

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Figure captions

Fig. 1. X-ray diffractograms of the fly ashes studied

Fig. 2. Influence of the amorphous content of the fly ash on the 28-day compressive strength of cements formulated with 20% replacement of Portland cement by each fly ash

- Fig. 3. Compressive strength evolution: a) TP, b) FB, c) TG, d) TG
- Fig. 4. Results of isothermal calorimetry testing
- Fig. 5. Ca(OH)₂ content of each mix as determined using TGA
- Fig. 6. X-ray diffractograms of hardened binders after 3 days of curing
- Fig. 7. SEM images of selected binders: a) TP/OS/50/A/1/7, b) TP/OS/50/A/1/28

Table captions

Table 1. Chemical and physical properties of precursor materials tested

Table 2. Mineralogical composition of fly ashes as determined by quantitative X-ray diffraction

Table 3. Mineralogical composition of cement as determined by quantitative X-ray diffraction

Table 4. Mix proportions

Table 5. Fly ash composition before and after sieving process

Table 6. Mix ID encoding (order, description and code per variable)

Table 7. XRD results











Fig. 5







Chemical and Physical properties	TP	FВ	TG	TA	
SiO ₂ (%)	56.67	43.83	55.14	58.58	
Al ₂ O ₃ (%)	20.65	28.11	17.63	19.96	
Fe ₂ O ₃ (%)	4.92	4.39	9.77	10.21	
(SO ₂)+(Al ₂ O ₃)+ (Fe ₂ O ₃) (%)	82.24	76.33	82.54	88.75	
SO ₃ (%)	0.06	0.09	0.09 0.11		
Na₂O (%)	0.07	0.89 0.56		0.76	
CaO (%)	3.27	5.99	3.64	3.17	
K₂O (%)	1.59	1.28	1.78	2.29	
MgO (%)	0.62	1.74	1.38	1.50	
LOI (%)	10.74	12.00	8.74	1.53	
Density (g/ cm ³)	2.09	2.11	2.26	2.32	
Retained # 325 (%)	38.20	29.90	30.50	18.70	

Composition (%)	TP	FB	TG	TA
Quartz	18	8	22.8	14.8
Mullite	15.1	20.6	8.8	6
Hematite	0.7	0.5	1.2	1.2
Magnetite			1.2	
Coesite			0.4	
Calcite	1.2			
Lime				0.6
Magnesioferrite				1.3
Amorphous material	64.5	69.3	65.6	76

Mineralogical Composition of Cement (%)								
C₃S C₂S C₄AF C₃A (cubic)				C₃A (orthorhombic)	Quartz			
52.1	30.5	10.2	3.3	0.8	0.3			

Hy ash [%]	0%	20%	50%	50%
Cement [g]	500	400	250	250
Fly ash [g]		100	250	250
Sand [g]	1375	1375	1375	1375
Water [ml]	242	242	242	242
				+ Activator

	Seve - Treatment	Main Parameters						
Hy ash		LOI	Fe ₂ O ₃	CaO	SO ₂	Amorphous		
	As received	10.7	4.9	3.3	56.7	64.5		
TP	<74 µ m	8.7	5.9	0.6	59.5	67.3		
	< 45 µ m	5.1	5.3	1.4	62.3	59.6		
FB	As received	12.0	4.4	6.0	43.8	69.3		
	<74 µ m		3.8	3.2	45.0	60.2		
	< 45 µ m	5.8	4.8	6.9	45.4	63.6		
TG	As received	8.7	9.8	3.6	55.1	65.6		
	<74 µ m	1.5	11.2	2.6	63.1	56.1		
	< 45 µ m	1.9	10.5	4.4	56.9	65.5		
ТА	As received	1.5	10.2	3.2	58.6	76.0		
	<74 µ m	1.3	10.7	3.0	57.9	75.5		
	<45µm	1.5	10.4	2.8	56.6	78.1		

Mix ID (1/ 2/ 3/ 4/ 5/ 6)						
Letters and numbers order	Description					
1	Cementitious material name					
2	Cementitious material size					
3	Cementitious material percentage					
4	Activator					
5	Dosage (%)					
6	Age – Days					
1 - Cementit	ious material Name					
Œ	Cement					
TP, FB, TG, TA Fly ash						
2 - Size						
10 D50 - Cement						
OS	Original Sze - Fly ash					
3 - Cementitiou	us material percentage					
0	0%					
20	20%					
50	50%					
100 100%						
4 - Activators						
A	Sodium sulfate					
Q	Quicklime					
L	Lime					

Mix ID	Quartz low	Tobermorite 9 ^a	Mullite	Portlandite	Ettringite	C₂S+C₃S	C₄AF	C₃A	Amorphous content
CE/10/100/0/0/3	1.5	15.9		14.7	4.6	17.3	3.0	1.0	42.0
FB/OS/50/A/1/3	2.5	4.8	8.2	3.7	4.3	4.5	0.8	0.3	71.0
FB/OS/50/L/1/3	3.4	9.5	8.1	7.1	4.4	8.3	0.7	0.1	58.6
TA/OS/100/A/1/3	17.5		6.9		0.9				74.8
TA/OS/50/L/1/3	2.8	10.5	1.1	10.6	0.7	11.0	0.7		62.6
TG/OS/50/L/1/3	7.6	6.7	4.2	7.7	1.8	8.4	0.5	0.3	62.9
TP/OS/50/A/1/3	7.3	7.4	7.8	6.0	5.6	4.3	0.6	0.2	60.8
TP/OS/50/L/1/3	7.6	4.3	8.4	6.9	3.1	5.6	0.8	0.4	62.7