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A Review of Activation Methods in Fly Ash and the Comparison in Context of Concrete Strength

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Abstract—*Increased energy usage in the current scenario demands the installation of larger and more power supply systems worldwide. This causes huge amount of fly ash generation every year which in turn sources its disposal problem. The associated ecological concern with fly ash necessitates its alternative engagement and increased utilization. A number of applications of fly ash have been investigated and adopted in various fields. Among all other applications, fly ash plays the vital role in construction industries producing stronger and more durable building materials. It has been widely adopted in concrete as cement replacement enhancing the fresh and hardening properties. With the excellent characteristics properties, fly ash is observed to be one of the best admixtures in concrete both economically and ecologically. The low hydration capacity of fly ash at initial period necessitated the introduction of activation techniques to enhance the fly ash activity towards improvement of initial concrete strength. Fly ash activation has been adopted through many activation techniques in the context of chemical, mechanical, thermal, mechano-chemical and physiochemical methods and many others. Though all the types of activation processes contribute in the increased initial compressive strength, the degree of improvement varies with its method. This article discusses the different techniques of activation adopted till now and their impacts on concrete strength and durability. The process description and the compressive strength of fly ash concrete at 7 and 28 days are presented for a comparative assessment.*

Keywords*: Fly ash, activation techniques, compressive strength*

1. INTRODUCTION

Fly ash is the effluent of coal fired power plant. The annual environmental load and huge land disposal problem due to massive generation of Fly Ash (FA) compels the researchers to think about the utilisation of FA. The inherent physical and chemical properties associated with fly ash have enabled its usage in various industries, among which construction is one major setup.

Researchers have proposed that FA is one of the best pozzolanic materials which, when used in concrete as partial replacement of cement, gives better workability, improved durability and long term strength due to presence of fine spherical shape particles (Poon et al, 2003, Ramezanianpour, 2014). Lam et al (1998) noticed that 15 to 25% FA replacement have beneficial effect on the tensile strength of concrete. They have also indicated that though the initial strength of FA concrete may be low, with age the strength is comparable with that of control concrete. Poon et al. (2000) recommended 45 % replacement of cement with class C FA for better compressive strength, heat of hydration and chloride diffusivity of concrete. Experimental results of Siddique (2004) indicated the continuous and significant increase in compressive, flexural, split tensile strength and abrasion resistance in high volume FA concrete with curing age of 90 days or more. It has also been noticed that the class F FA can be suitably used up to 50% level of cement replacement in concrete for reinforced concrete construction. Mittal et al (2005) reported that use of FA improves the workability, cohesiveness, pumping characteristics and surface finish of concrete. Sumer (2012) reported that, the addition of FA significantly increases the resistance to sulfate attack, regardless the type of FA. High volume FA envisaged more durability in Salt, acidic and sulphate prone environments (Balakrishnan and Awal (2014)).

From the brief review of the available literatures it is well established that incorporation of FA as partial replacement of cement in concrete is not only desirable from economic, ecological and energy conservation point of view but also from strength and durability perspectives. However, despite every outstanding characteristics of FA concrete, its low initial strength gaining property and high setting time has necessitated the use of new technologies called activation techniques to accelerate the FA hydration resulting early age strength gain. The following section deals with various activation techniques reported to accelerate the reactivity of FA in concrete. Subsequently a comparative assessment these methods considering of compressive strength of concrete as performance measuring index has been carried out.

2. ACTIVATION OF FLY ASH

Activation techniques are the method of enhancing activity of pozzolanic materials in order to improve the hydration capacity. A brief review of research articles citing different activation techniques and their sub-category is presented below.

2.1 Chemical activation

Several investigations have been carried out using chemical activators with a view to accelerate the hydration of FA blended cement and thereby improving the strength and durability properties of the concrete. Chemical activation are of various types depending upon the chemicals used.

a. Alkali activation

Alkali activation has been carried out using either alkaline reagents in hydroxide form or alkali salts.

Alkaline reagents: The alkaline reagents such as $Ca(OH)_2$, NaOH, KOH, etc. have been reported to be used as alkali acivator.

Fan et al (1999) activated the FA using $Ca(OH)_2$ and adding small amount of Na_2SiO_3. They noticed the acceleration of early cement hydration and hydrated crystallization promoting the setting and hardening of concrete. Palomo et al (1999) activated FA with highly alkaline mix of NaOH, KOH, water glass. They reported mechanical strengths in the 60 MPa range after curing at 85^0C for only 5 h. Mira et al (2002) demonstrated stimulation of pozzolanic reactions when lime is added to FA. Improved stability and durability of FA concrete were observed. Luo et al (2012) prepared an alkali activated slag FA cementitious material system using NaOH, Na_2CO_3 as alkali activator and slag respectively. They noticed the rheological characterization and mechanical properties to be sensitive to the change in alkali-activator temperature. The setting time was observed to be in conformance to the requirements of national standard when alkali activator temperature was 40°C. Ryu (2013) examined the effects of chemical changes of alkaline activators on the compressive strength and microstructure of mortar. A higher molarity of NaOH used as an alkaline activator not only produces higher compressive strength but also put considerable effect on the early strength giving a more compact structure.

Alkali salt: In this category of activation processes alkali salts such as Na_2CO_3, $Na_2O{\bullet}nSiO_2$, etc. are used as chemical activator. Rashad et al (2011) presented a comparative study of compressive strength of chemically activated FA using sodium silicate in three different concentrations of 20, 30 and 40 (wt.%) at different temperatures. The experimental results suggest that alkali activated FA is more resistive to elevated temperatures and also both the initial and residual strength depends upon the concentration of activators.

b. Sulphate activation:

Sulphate such as $CaSO_4$ and Na_2SO_4 are sulphate activators reported in literature. Lee et al (2003) studied the strength and micro structural properties of FA cement system using three types of chemical activator (Na_2SO_4, K_2SO_4, triethanoamine) and observed enhanced production of ettringite at the early ages resulting the reduction of the pore size ranging from 0.01 to 5 mm along with increased compressive strength. Criado et al (2010) studied the effect of sodium sulfate (Na_2SO_4) on the alkali activation of FA and observed declining trend in rate of activation.

c. Activation by High Molecular materials:

This include polymer activator. Liu Baozhu (2003) in Anhui University developed a new type of high molecular composite material (HB high-efficiency fly-ash) as activating admixture. Only about 1.5%–2.0% usage of such material has been observed to be activating pulverized fuel ash. As a result of usage of this polymer activator, the overall economic effectiveness of China National Petroleum Corporation and Liaoning Jinxi Oil Refinery has increased almost by 40%.

d. Composite activation:

Composite activators are prepared combining activators in complementing proportions. The effect of composite admixture is observed to be much better than that of the individual activators. Zhenglan et. al. (2007) in Anhui Architecture and Industry Institute found that composite activator prepared through mix of alkali, sulfate and water reduced component together enhances early and final strength of concrete using the pulverized fuel ash.

2.2 Mechanical activation

It is well known that the reactivity of FA depends on its particle size, glass content and glass composition (Jimenez and Palomo, 2003). The low reactivity of FA leads to slow setting and early strength development. The merit of using mechanical activation (sorting or grinding of FA) for improving the reactivity of FA concrete is well accepted (Hela and Orsakova, 2013) as it offers the possibility to alter the reactivity through physicochemical changes in bulk and surface without altering overall chemistry of the material (Kumar et al, 2005). Gordana et al (2007) demonstrated mechanical activation of a Portland cement and FA mixture and found the highest increase of compressive strength achieved in the early period of setting, which indicates an improvement in the early hydration of the mixture. Kumar et al (2007) suggested that the mechanical activation of FA can be used to develop improved blended cements with higher proportion of FA (50–60%) without degradation in cement properties. Aydin et al (2010) reported up to 60% replacement of mechanically activated FA gives higher compressive strength than control mortar. Experimental results of Sharma and Rani (2015) demonstrates slight increase in silica percentage, amorphous nature, specific surface area and surface roughness of FA after mechanical activation with the generation of sufficient activity on FA surface improving its durability. R Hela et. al. (2012) investigated the influence of mechanically activated fly ash on the properties of fresh and hardened concrete through grinding in ball mills and reported decreased consistency due to higher specific surface of fly ash and high values of strength of concrete in all phases of testing.

2.3 Mechano-chemical activation

In this method activation is done both by blending and grinding of the cementitious material to explore the increased Pozzolanic reactivity. This activation is able to break the glassy phase in the cement free systems. Senneca et al (2011) assessed the effectiveness of mechano-chemical activation of FA by ball milling in a lab scale apparatus They claimed noticeable modification of DTG profiles, indicative of enhanced reactivity. Sharma et al (2012) overviewed on chemical, structural, and morphological changes in FA properties with mechanical activation using high energy planetary ball mill and chemical activation by digesting with various mineral acids at 110ºC. The experimental results confirmed that the mechno-chemical activation can generate sufficient activity on FA surface rendering its potential application in heterogeneous catalysis. Sadique (2013) explored the pozzolanic reactivity of a calcium rich FA by mechano-chemical activation and the dispersion of the ground reactive blend particles by super plasticizer is found more effective than further alkali activation by NaOH. The progressive mortar compressive strength obtained is up 46 MPa after 28 days of curing.

2.4 Physio-chemical activation

Physio-chemical activation is the method in which the fly ash is activated both physically and chemically. This activation method can be subcategorised in to the following.

Low temperature calcination: In this activation method, the chemical components and mineral structure changes by adding some limestone and mineralized agent followed by calcination and quenching to obtain pulverized fuel ash of very high activity. The influence of calcinations on the microstructure, phase composition and strength development in geopolymer materials prepared using Class F FA is presented by Bakharev (2005). Min et al (2008) reported the improvement of early strength of the FA–lime binder due to addition of calcined phosphogypsum as it accelerates the pozzolanic reaction of FA. This is due to the formation of ettringite and gypsum. Further the curing conditions are also important for the strength development.

Thermal activation: In this activation process blending of the pulverized lime and pulverized fuel ash in a certain proportion, put in an autoclave for appropriate time and temperature followed by dehydration and cooling. This method deals with large investment and complicated techniques because the autoclave is needed. Saraswathy et al (2003) adopted this activation of fly ash blended cement to improve the corrosion resistance and strength of concrete. They reported that 20-30% of activated fly ash can replace cement successfully to get high strength and corrosion resistance.

Hydrothermal activation: The hydrothermal reactions of FA were investigated by Ma and Brown (1997). Calcium silicate hydrate, tricalcium aluminate hydrate and ettringite were observed during XRD analysis. It is confirmed that the variations in reactivity depended on the presence of added $Ca(OH)_2$ or $CaSO_4 \cdot 2H_2O$. Goni et al (2003) studied the effect of hydrothermal treatment on the pozzolanic reaction of FA and the corresponding compound formed. The results show that the alkalis play more important role than the fineness of the starting FA in their reactivity in hydrothermal condition. Sun and Wu (2009) determined the split tensile strength of hydrothermally activated FA. The results show splitting tensile strengths of about 1.0MPa by hydrothermal hot pressing alone. However, with a small amount of chemical activator (NaOH), the tensile strength can reach as high as 5.4MPa.

A summary of strength test results is offered in Table 1. The table represents the data as furnished by the researchers, scaled in some cases following linear interpolation.

TABLE 1: Compressive Strength variations in different activation methods

ACTIVATION TYPE	METHOD	INVESTIGATOR	CEMENTIOUS MATERIALS IN SPECIMEN	CS AFTER 7 DAYS	CS AFTER 28 DAYS	% increase in CS wrt NAFA at 28 days
Mechanical	Ground to have size class mean dia reduced by six times	Sulc et al. (2005)	OPC+FA	41	62	163.1
Mechanical	Ground for 60 minutes	Gordana et al. (2007)	OPC 70 + FA 30	36.6	52.6	335
Thermal	Curing at 400C	Shi and Dey	OPC 70 + FA 30	38	48	141
Chemical	NaOH, Sodium Silicate solution	Jimenez et al. (2003)	FA	55	59	Not Available
Chemical	Blending with NaOH (12%) and Sodium Silicate (50%)	Ryu et al (2013)	FA	34.3	44.8	Not available
Chemical	NaOH + 15% Sodium Silicate	Rashad (2011)	FA	42	50	Not available
Chemical	Sodium Sulphate	Criado (2010)	FA	35.46	37.16	54.17
Chemical	Sodium Sulphate	Criado (2010)	FA+ Blend of Sodium Oxide and Water glass)	57.66	67.57	120.9
Chemical	Sodium Sulphate and Lime	Qian et al. (2007)	OPC 70 + FA 30	28	40	173.9
Chemical	Calcined Photogypsum	Min et al. (2008)	65 FA+ 35 Lime	11.41	15.38	1200

ACTIVATION TYPE	METHOD	INVESTIGATOR	CEMENTIOUS MATERIALS IN SPECIMEN	CS AFTER 7 DAYS	CS AFTER 28 DAYS	% increase in CS wrt NAFA at 28 days
Chemical	Sodium Hydroxide	Mane et al. (2002)	30 FA + 70 OPC	25.44	52.02	101.7
Mechano-Chemical (Grinding + treating with alkaline/ salt)	NaOH	Arjunan et al. (2001)	OPC 80 + FA 20	*56*	99	162.3
	Ca(OH)2+NaOH			*38*	66	108.2
	Ca(OH)2+Na2SO4			*54*	90	147.5
	NaOH+Na2NO3			*43*	94	154.1
	Na2CO3+Ca(OH)2+Na2SO4			*57*	85	139.3
Mechanical	Ground	Saraswathy et al. (2007)	OPC 70 + FA 30	11.5	16.5	103
Thermal	900-10000C, 1 hr			13	18	112.5
Chemical	NaOH + CaO			15	22	137.5
Chemical	0.05% triethanolamine	Lee et al. (2003)	OPC 60 + FA 40	23	34	113.3
	K2 SO4			24	33.5.	111.6
	Na2SO4			21	31.5	105
Thermal and Chemical	Autoclave reactor	Mane et al. (2002)	40 FA		54.59	106.7

Italicized data are obtained through scaling from available data using linear interpolation; NAFA: Non-activated Fly ash, CS: Compressive Strength.

3. CONCLUSION AND RECOMMENDATION

It is noted from the concise review that a considerable amount of studies has already been towards enhancement of the reactivity of FA. However, in each of the aforementioned studies the primary objective is to improve the early age strength in concrete.

The strength data have a wide variation, since the quality of FA and the Cement are different in different research setup. In certain cases a very high % increase is noticed, however the initial strength noted is extremely low and thus cannot be considered as the best one. The comparison is expected to give future researchers a hint only, how these techniques can contribute to the strength changes. Chemical activation methods have come out as preferred methods by most of the researchers. Recent researches are also focusing on combinatorial activation techniques. Concretes made up of fly ash cannot be evaluated on compressive strength point of view only. There is lots of opportunities, where in durability related studies, micro-structural studies, mineralogical studies in perspective of concrete can be taken up. Moreover, it is observed that, most of techniques may not be suitable for mass production, since additional chemical and mechanical processing costs may unbalance the economic aspects related to cement replacement in concrete. The activation techniques thus may be seen as still an unexplored area in concrete research.

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