EFFECT OF CHEMICO-MINERALOGICAL CHARACTERISTICS OF INDIAN FLY ASHES ON THE PROPERTIES BLENDED CEMENT CONCRETE

Abstract:

The use of fly ash as supplementary cementitious material was originally motivated by its consistent pozzolanic activity and provide for sustainable development in the cement industry. India has been a pioneer in the manufacture of blended cements. Traditionally the blended cements in the country have been produced with calcined clay, mixes of calcined clay and fly ash, fly ash and granulated blast furnace slags. The improved understanding of the beneficial properties imparted by fly ash for both the plastic and hardened stages of blended cement concrete has led to a gradual growth in acceptance for the fly ash blended cements in India in the last decade. The enhanced durability of blended cement concretes has made it the preferred cement thereby replacing the earlier skepticism regarding its performance. The choice being governed by the judiciousness of quality control of the properties of the individual components at the manufacturing stage of the blended cement or at the Ready-Mix locations.

The paper discusses the effect of compositional characteristics of Indian fly ash such as the combustible content, particle shape & size distribution and its Chemico-mineralogical characteristics i.e its oxide composition, nature & contents of the crystallites and the compositional characteristics of the amorphous glassy phase of fly ash on its enhanced pozzolanic reactivity.

The paper also discusses in brief the available methods for assessing the pozzolanic reactivity of fly ash and further illustrates a rapid test (Alkali Reactivity Test) method developed at the author’s laboratory for assessing the pozzolanic activity of Indian fly ashes. The brief description of the test along with the Alkali Reactivity (R_A)

values for Class-F Indian fly ashes of different sources and of varying characteristics is also illustrated.

The authors conclude that a proper understanding of the interrelations of the fly ash & OPC characteristics is of immense importance for engineering the properties of the blended cements for durable Concrete.

1.0 INTRODUCTION

Based on the studies carried out on the fly ashes of different coal fired thermal plants in the country our observations have been that the Indian low lime Class-F (Grade–I) fly ashes are in no way inferior to the their European counter parts. To obtain the maximum benefits, what is desired is a complete understanding of the characteristics of each source and accordingly adopting the processing and quality control practices to achieve higher hydraulicity of the fly ash used in the Blended cements. In the Tropical climatic condition of the country, fly ash based Blended concrete is being presently equivocally accepted as an option for durable concrete structure.

Based on the experimentally observed results with Fly ash based blended cement & the reported data on the effect of use low lime class-F fly ash based cement / concrete on the properties of the concrete, the authors make an attempt at evolving an understanding of the influence of the properties and reaction mechanics of the fly ash component in determining the limiting / favoring conditions for improved properties related to the blended cement and concrete.

At the Research & Development division of The Associated Cement Cos. Ltd., considerable studies have been carried out on the fly ashes of different coal fired thermal plants and of differing compositions and mineralogy to understand the influence of fly ash characteristics on the properties of resultant cements and concrete. The studies indicate that the fly ash characteristics play a significant role in determining the performance of the cements and concrete. An understanding on this interrelationship thus would help in engineering the fly ash properties of the available fly ash so as to produce blended cement / concrete with improved performance.

It is of proven understanding that the characteristics of fly ash produced in the coal fired thermal plant is a function of nature of the coal, coal comminution system, boiler type & efficiency, fly ash collection ESP fields, loading at which the thermal plant operates etc., as a result from the same source the fly ash characteristics could vary substantially.

The paper discusses the effect of compositional characteristics of Indian fly ash such as the combustible content, particle shape & size distribution and its Chemico-mineralogical characteristics i.e its oxide composition, nature & contents of the crystallites and the compositional characteristics of the amorphous glassy phase of fly ash on its enhanced pozzolanic reactivity and also a rapid test (Alkali Reactivity Test) method developed at the author's laboratory for assessing the pozzolanic activity of fly ash. The paper further discusses the effect of fly ash characteristics on the early and later age strength developments of the blended cement and concrete.

2.0 Characteristics of Indian Fly ashes:

2.1 Chemical Composition

Generally, chemical composition of fly ash shows a wide diversity, however the major oxides present in Indian fly ash are SiO_2 (45-65%), Al_2O_3 (15 –35%), Fe_2O_3 (3-10%), CaO (0.3- 6 %). The Indian fly ashes are categorized, in line with the global trends, into two types Low lime Class F and High lime Class C. Fig. 1 shows the relative position of Indian Low lime class F fly ashes in a ternary plot with respect to SiO_2, Al_2O_3 and CaO. The ternary phase diagram of SiO_2-Al_2O_3-CaO indicates the relative positions of cementitious materials. As the lime in the fly ashes increases i.e. as the fly ash composition changes from low lime Class-F fly ash to lime containing Class - C fly ashes their relative position moves towards the center of the ternary diagram. At the authors laboratory ~ 55 Indian fly ashes were characterized for their chemical composition, the variation in SiO_2 & Al_2O_3 (expressed as SiO_2 / Al_2O_3 ratio) along with % CaO content is graphically shown in Fig.2

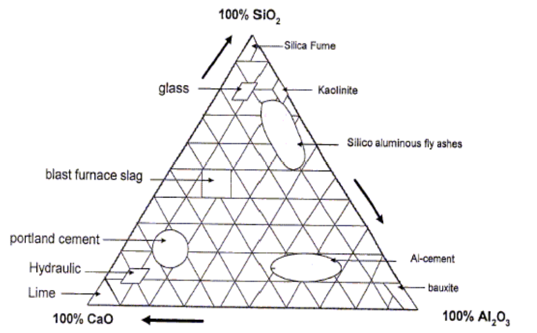

Fig.1: SiO_2-Al_2O_3-CaO system showing relative positions of Indian Fly ashes

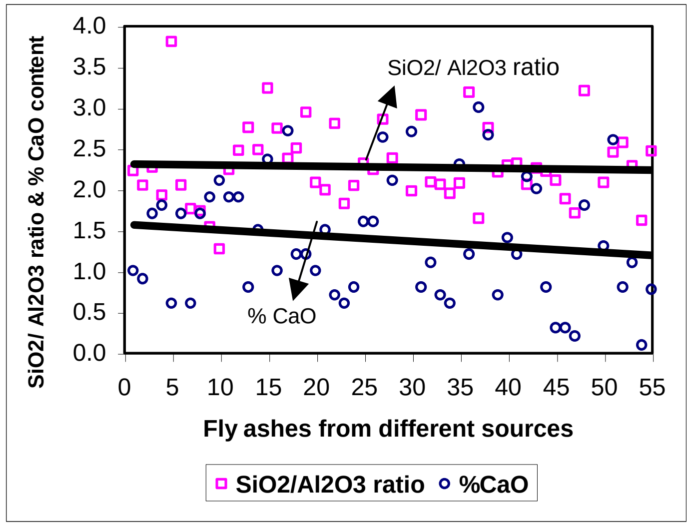

Fig.2: Variation in SiO_2, Al_2O_3 and CaO in Indian fly ashes

2.1.1 Combustibles in fly ash:

The effect of combustibles content, which can be mainly unburnt carbon is known to include discoloration, poor air entrainment behavior, more water requirement and low compressive strength. The simple test method for assessing combustible content or carbon content is by loss-on-ignition (LOI, a measure of carbon mass). The variation in % LOI of Indian fly ashes is shown in Fig. 3

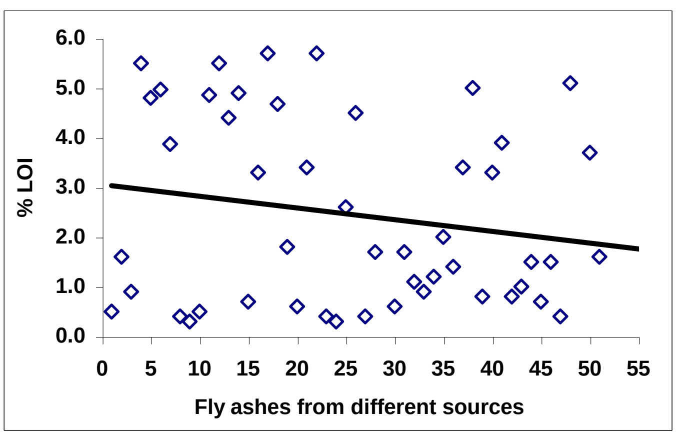

Fig. 3: Variation in LOI in Indian Fly ashes

The higher percentage of carbon or combustible contents in fly ash in undesirable as the porous surface area of carbon determines the capacity of the carbon to adsorb admixtures or other chemical admixtures [1,2]. This adsorption is undesirable, as it degrades the freeze-thaw resistance of the concrete because air bubble content is lowered. Three microscopically distinct carbon types have been reported to be present in high carbon fly ashes i)inertinite, which appears to have been entrained in the particles , prior to meting or combustion ii)isotopic and iii) anisotropic carbon which appear to have passed through the melting stage. It has been observed that the capacity of the carbon in fly ash to adsorb the chemical admixtures is a function of the type of carbon present in the fly ash [(3)] and thus it is not always related to the LOI or carbon content of the fly ash.

Another indirect method [(4)] for measuring the adsorptive behavior of fly ash is the performance of air entraning admixture and fly ash by applying thermal analysis and petrographic examination. The results indicated that fly ash containing higher portion of isotropic coke has a greater adsorption capacity than fly ash containing high portion of anisotropic coke

Presence of high carbon in fly ash thus would tend to use more water [(5)] thus affect the compressive strength characteristics of the resultant blended cements of fly ash based blended concrete. It is not recommended to use a high-carbon (> 5 percent) content fly ashas per Indian standards, but if used, the dosages of air-entraining agent and other chemical admixtures need to be optimised with the use high carbon fly ash. At the authors laboratory, the effect of combustible content in fly ash on physical properties of the resultant PPC (Portland Pozzolana Cement) (Indian standards) was studied .The Fig.4 depicts the PPC prepared with a 20% high carbon fly ash (with 12.6% combustible content) and with 1% combustible content.

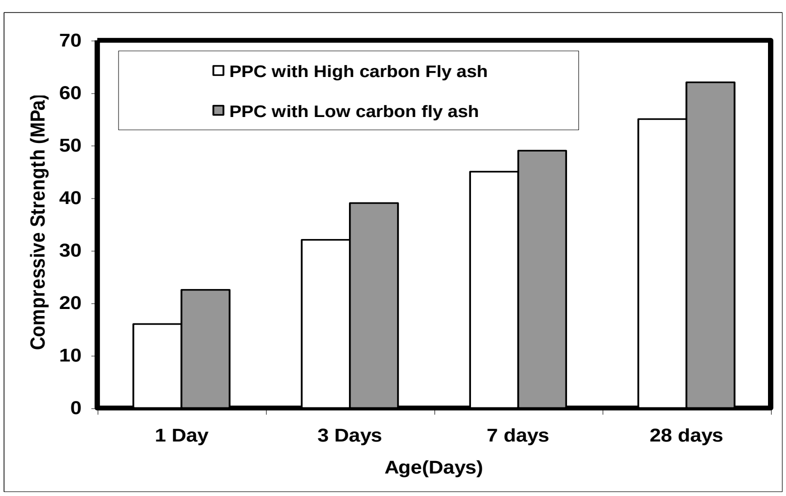

Fig.4 : Effect of high carbon content in fly ash on compressive strength

2.2 Pozzzolanic Reactivity of Fly ash

The methods of testing the pozzolanic activity of pozzolans or supplementary cementitious materials (SCMs) have been studied by many researchers for more than 100 years. However since the different pozzolans are so myriad and of different quality it has been difficult to establish appropriate test methods for assessing the pozzolanic activity. Lea [6] summarized and discussed the various methods of testing the pozzolanic activity at the International symposium on the chemistry of Cement in 1938, while Malquori [7] also discussed the test methods at the Fourth International symposium on chemistry of cement in 1960, which serve as an useful guides for the research on methods of testing the pozzolanic activity of pozzolans.

Moran and Gilliland [8] divided the test methods into three categories of tests

- Tests on the Pozzolans alone
- Tests on Pozzolans –lime mixtures
- Tests on Pozzolan-Portland cement blends

In the light of present knowledge of test methods and techniques, which are a combination of various methods such a classification is difficult, however the relatively frequently used methods could be categorized in the form shown in Table – 1.

Table - 1: Classification of methods of testing pozzolanic activity, which are frequently used

Classification		Example of Test Method
Method testing of strength by accelerated curing of pozzolan-lime or pozzolan-cement mortar		ASTM C 593-66T ASTM C 402-65T
Method adding Pozzolan to lime solution	Quantitative measurement of lime	Chapelle's Method Moran & Gilliland's method
	Quantitative measurement of lime and alkali	AFNOR P 15 – 462 Italian standards ISO recommendation No 1156
Method treating pozzolan or pozzolan-lime mixture with acid or alkali	Quantitative determination of SiO_2	Florentin's method
	Quantitative measurement of SiO_2+ Al_2O_3	ASTM C 379 – 56T Poliet & Chausson's method Austrian standards Charisius method
	Quantitative measurement of SiO_2+ Fe_2O_3	Steopoe's method
	Quantitative measurement of SiO_2+ Al_2O_3+ Fe_2O_3	Sestini & Santarelli's modification of Baire's method Feret- Florentin method
	Quantitative measurement of insoluble residue	Guillaume's method AFNOR P – 15 - 301
Method measuring development of dissolution heat of pozzolan		Jambors method
Measurement of electrical conductivity of Lime –pozzolan solutions		Lea's method

Some of the test methods are observed to be suitable and correlate well with the strength results of lime – mortars, However they do not correlate to the behavior of Cement – pozzolans blends. In the widely used standards of various countries, the chemical methods of testing pozzolanic activity are more suited for judging activity of the pozzolanic materials and some of which are time consuming and do not provide a yard - stick to correlate to the strength of standard –cured mortar or concrete. For e.g. the pozzolanic activity test in the different standards provides the degree to which a pozzolan (fly ash) contributes to strength development of the resultant blended cement mortar but does not provide a measure of the pozzolanic reaction of the used pozzolans. The compressive strengths of a blended cement mortar / concrete is partly due to the pozzolanic activity and partly due to the packing effect of the added pozzolans.

The estimation of the amount of free calcium hydroxide remaining in the hydrated pozzolana-cement has also been used as an indication of the pozzolanic reactivity of the pozzolans. A number of methods [9] as shown in the Table – 2 have been used to determine the free calcium hydroxide content in the hydrated lime-pozzolana or pozzolana – cement mortar or concrete.

Table – 2: Methods used for estimation of free calcium hydroxide

Method	Originally described by	Applied to Pozzolanic Mixes by
Sugar	--	Sestini & Santarelli
Glycerol	Emley, Lerch & Bogue	Katherein; Rodt
Ethylene Glycol	Schlapfer & Bokowski	Forsen; Rodt
Phenol	Konarzewski & Lukaszewicz	Sestini and Santarelli; Witterkindt
Lime solution	Bake well & Bessey; Forsen	Forsen; Lea
Calorimetric	Bessey	Lea; Vittori

In such methods presence of the unhydrated cement compounds prevents use of aqueous solutions, as further hydration would occur during extraction. In pozzolana – cement mixes most of these methods do not give results having any precise accuracy however the methods have been observed to be consistent in themselves and are valuable for comparison of the hydration characteristics.

The pozzolanic activity of the pozzolana has also been reportedly monitored through estimation of the un-reacted pozzolana content of the hydrated blended cement pastes at different ages of hydration. Different chemical extraction methods have been studied by researchers for different pozzolans(10,11) and the studies have revealed suitability of specific methods for a given type of pozzolana. As per IS 3812 –2003, the pozzolanic reactivity of fly ash can be assessed by lime reactivity (4.5 MPa min.) and cement replacement test (80% min.). The interrelationship of LR, CR values and blains spefcific surface area for Indian fly ashes is graphically shown in Fig. 5

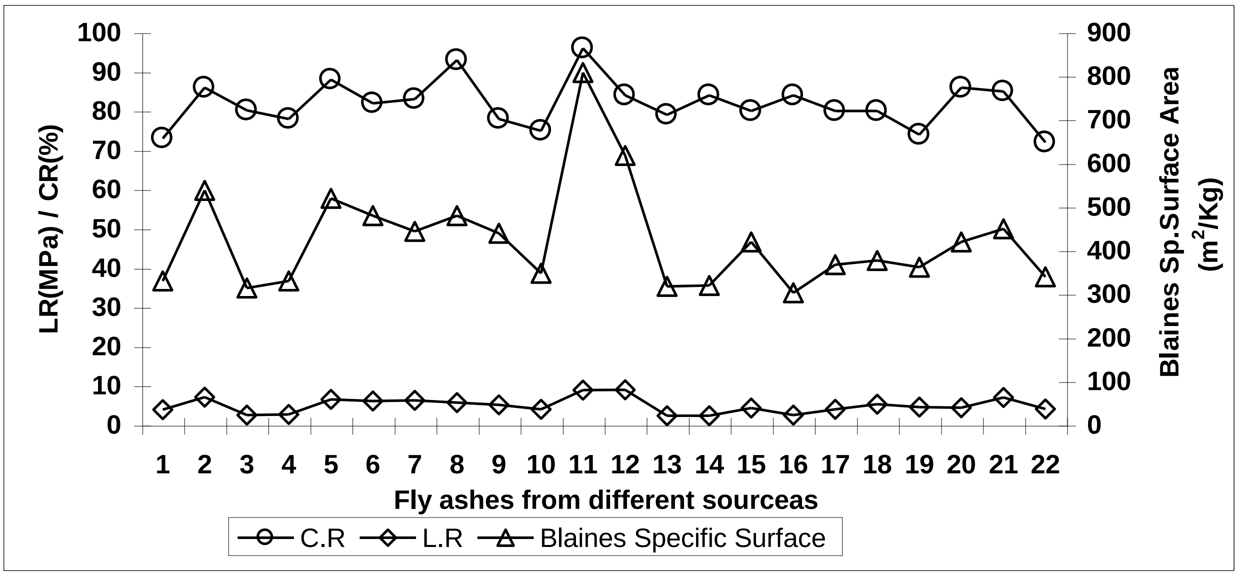

Fig.5: Interrelationship between Lime Reactivity and Cement Replacement Test for Indian Fly ashes

As it can be observed from Fig. 5 that, a very poor correlation exists between the L.R & C.R. values, although the L.R. values at times are lower the C.R. values of fly ash are well above the BIS requirements, indicating that the lime reactivity of fly ash is affected by the mode of disposal. However, on regrinding the fly ash to higher fineness, the L.R values are observed to increase. Besides the LR and CR values are also governed by the mineralogy, morphology and fineness of fly ash. This necessitates evolving a method for assessing the pozzolanicity of fly ash.

2.2.1 Alkali Reactivity Test for Fly ash

At the authors laboratory, a simple method for assessing the pozzolanic reactivity of fly ash was developed [12] based on the principle of reaction of hydroxyl ions with the amorphous / glassy phase of fly ash. The R_A represents the alkali-reactivity value for the fly ash under Test (R_A, measured in mili mloes / lit.). The quantity of fly ash and molarity of alkali hydroxide solution used for the test was optimized at the authors' laboratory and the data is represented graphically in Fig. 6

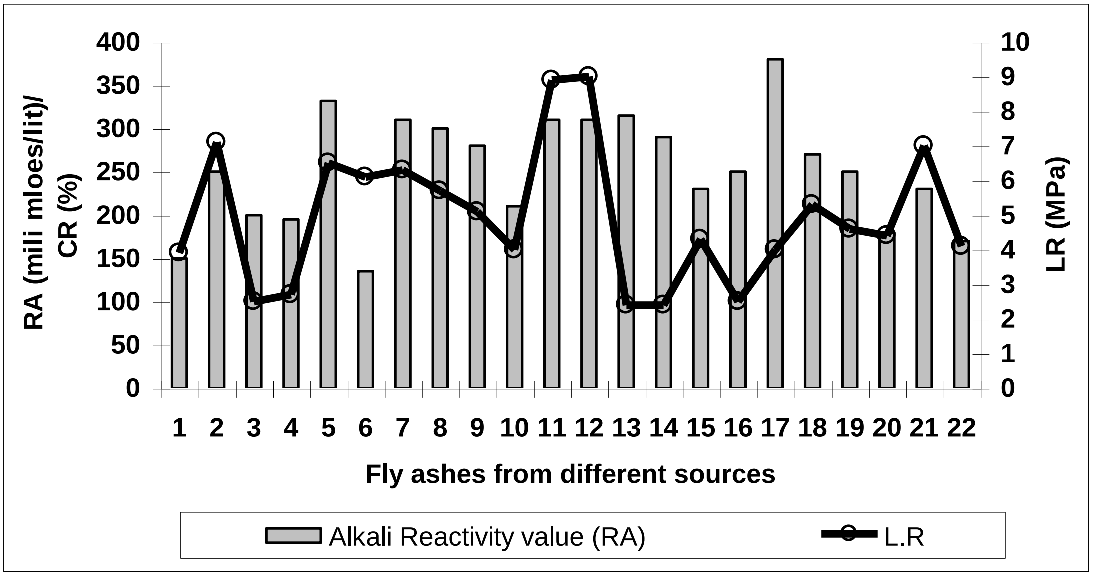

Fig.6: Alkali Reactivity Value and LR

Interrelationship between of fly ash characteristics

In an attempt to evolve the interrelationship between the fly ash characteristics, viz. Alkali reactivity value **(R_A)**, Lime Reactivity **(L.R)** , Cement Replacement Test Value **(C.R)** and the Blaine's specific surface, statistical analysis of the available data was carried out. The statistical analysis indicates a strong relationship between the fly ash characteristics as indicated by the correlation coefficient of 0.88. The correlation could be represented by an empirical equation given below :

Blaine's Specific Surface(m^2/Kg)= - 3136 - 0.236 R_A+44.84 L.R+63.76C.R.

Fig.7 represents the characteristics of a set of fly ash samples along with the predicted values of fineness calculated from the empirical equation to achieve LR and CR as per BIS requirements

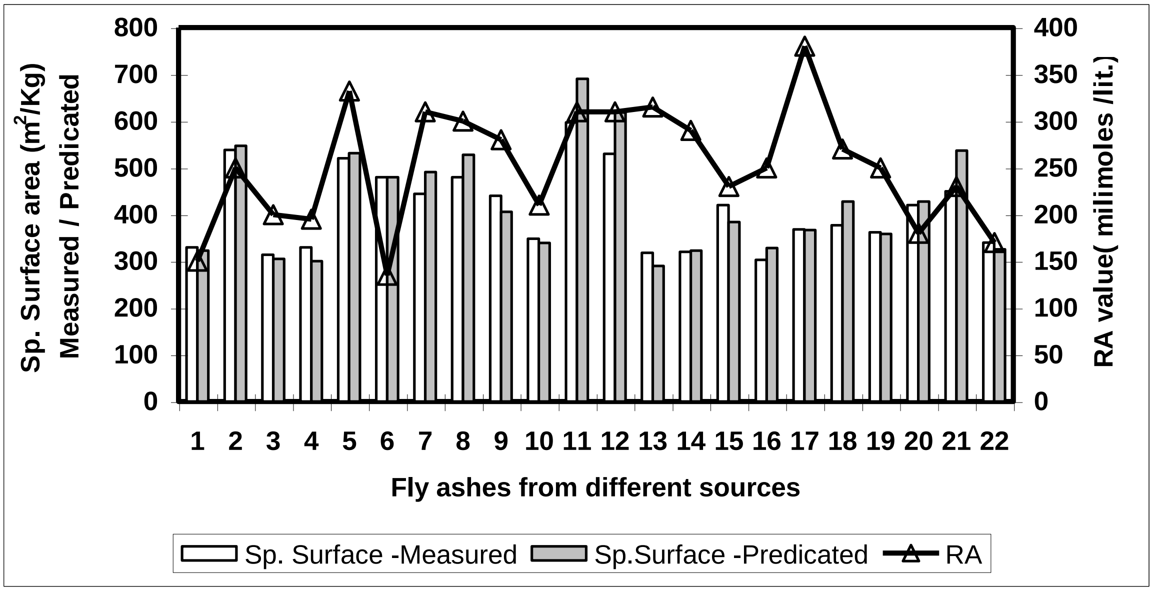

Fig.7: Correlation between Alkali Reactivity Value and Sp. Surface area (measured and Predicated)

2.3 Mineralogy of Indian fly ashes:

In India, the mineralogical characteristics of fly ash produced, has been observed to vary. The mineralogy of fly ash has 15 -30% Mullite, 15-45 % Quartz, 1-5% Magnetite, 1-5% Hematite and around 25 - 35% of amorphous glassy alumino - silicate phase [13]. In the high lime Class - C fly ashes besides the presence of crystalline hydraulic active phases, the amorphous glassy phase is calcium rich and more reactive than the alumino silicatic amorphous phase of Low Lime Class - F fly ashes, which is comparatively latently hydraulic [14,15]. The amorphous phase in fly ash is thus the reactive part in fly ash responsible for the secondary hydration and the consumption of free calcium hydroxide during the pozzolanic reactions. The crystalline phases of fly ashes such as Mullite, quartz, hematite, magnetite are non hydraulic while crystalline calcium aluminate phases present in some of the Class -C fly ashes are cementitious in nature. Thus the chemico-mineralogical composition of the fly ash determines the reactivity of the fly ashes, it also has a bearing on the concrete properties such as durability.

2.3.1Effect of Mineralogical composition of the fly ash:

It would be immensely important to understand that the characteristics of fly ashes are assemblages of particles produced by combustion and melting of individual small particles of ground coal. Each particle is heated and undergoes changes independently of other particles, while passing through the burning zone of the power plant boiler. Its composition reflects that of the inorganic portion of the particular coal fragment, with whatever changes have occurred due to selective vaporization of components and perhaps subsequent surface deposition. In any of these events, the composition of each particle is necessarily different from its neighboring particles and overall chemical analysis is only an average description of the assemblage. Another feature of fly ash is that individual fly ash particle vary in content of crystalline component like quartz, Mullite, Iron oxide, calcium bearing compounds (in Class C fly ashes) and amorphous or glassy phases.

As discussed above a considerable distinction exists between low lime class F fly ash from bituminous coal and high lime class C fly ash produced from lignitic or sub-bituminous coal. Depending on composition of the clay mineral constituents, the boiler temperatures, coal fineness used in the boiler type as well as the efficiency of the heat recuperation systems the fly ashes would show a difference in the glassy amorphous phase contents and the nature and extent of minerals present. Which would determine the pozzolanic potential of the fly ash and its resultant effect on the performance characteristics of the cements /concrete.

Another way of looking at the pozzolanic reactivity is by the reaction of the fly ash in hydrated cement pastes. The Fig. 8 depicts comparative pozzolanic reactivity of two compositionally similar low lime class –F fly ashes differing in Mineralogy and amorphous contents. The method used has been evolved at the authors laboratory for comparing the reactivity of fly ashes [16] The XRD showing the difference in mineralogy is shown in Fig.9.

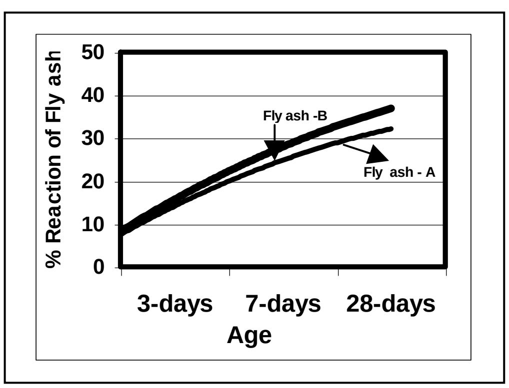

Fig.8 :Comparative Pozzolanic reactivity of Class F Fly ashes differing in the amorphous content

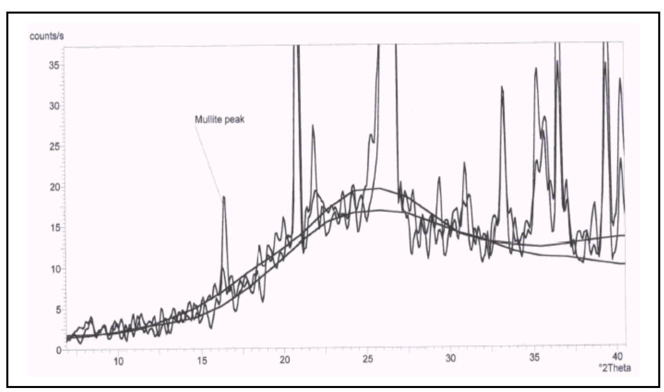

Fig.9:XRD fly ashes of differing mineralogy

2.3.2 Effect of amorphous phase composition of the fly ash :

At the authors laboratory attempts were made to characterized tha amorphous phase of fly ashes from different sources concentrating on %SiO_2/Al_2O_3 ratio and comparing it with the original fly ash. Fig.10 shows the relative composition of amorphous phase (i.e %SiO_2/Al_2O_3 ratio in amorphous phase in comparison to the original fly ash) of some of the class F Indian fly ash.

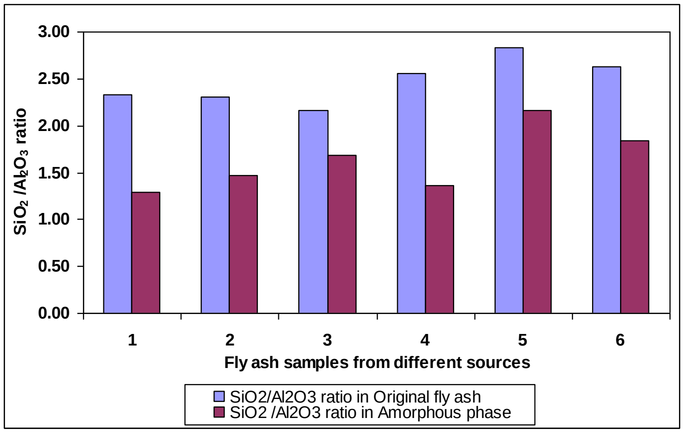

Fig. 10: Comparison of % SiO_2/Al_2O_3 ratio in original fly ash and in amorphous phase

It has been observed that as the composition of the amorphous phase changes that is as the alumino-silicate amorphous glassy phase becomes calcium rich or as the %SiO_2/Al_2O_3 ratio of the amorphous phase changes there is distinct shift observed in the peak maxima of the amorphous hump observed in XRD, i.e there is a shift in the maxima of the amorphous hump towards that of the hump maxima of the granulated blast furnace slag. This could be related to the changes in the composition of the amorphous glassy phase. The Fig. 11 depicts the amorphous hump maxima of different fly ashes of different CaO content, the figure also shows the nature of the amorphous hump of Class F fly ash, Class C fly ash and GGBS for comparison.

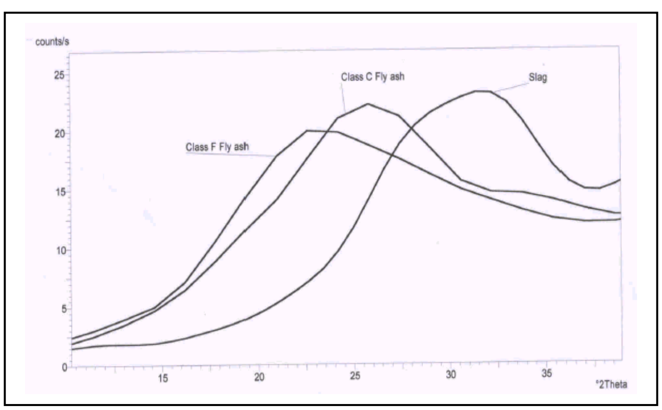

Fig.11: XRD of fly ashes with different S/A showing difference in amorphous humps

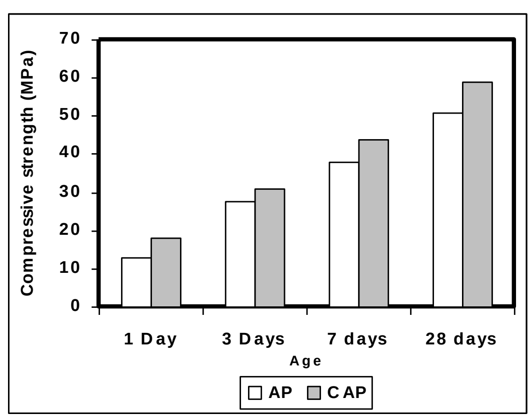

Fig.12: Compressive Strength of PPC with Fly ash AP & CAP

As already indicated that the lime rich amorphous alumino silicate has higher pozzolanic reactivity. Fig.12 depicts the compressive strength characteristics of PPC made with different amorphous phase composition.

2.4 Morphology of fly ashes:

The fly ash samples were coated with a thin layer (250-300 A^0) of conducting material (Gold) using sputter coater and examined in samples JEOL-KEVEX (Model No. JSM 5406). Scanning Electron Microscope was used at 12 KV accelerating voltage. The morphological characterisation of fly ash under SEM revealed that fly ash consisted mainly of spherical particles of 2-8 micron size, mostly as cenospheres and some angular particles of quartz and mullite having size in the range of 1-6 micron as shown in Fig. 13 & 14

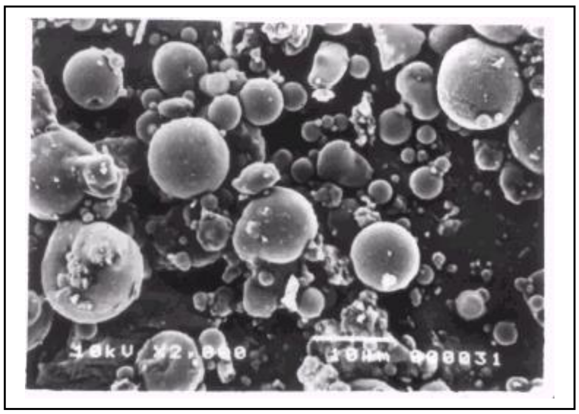

Fig.13: SEM photomicrograph of fly ash showing the spherical particles

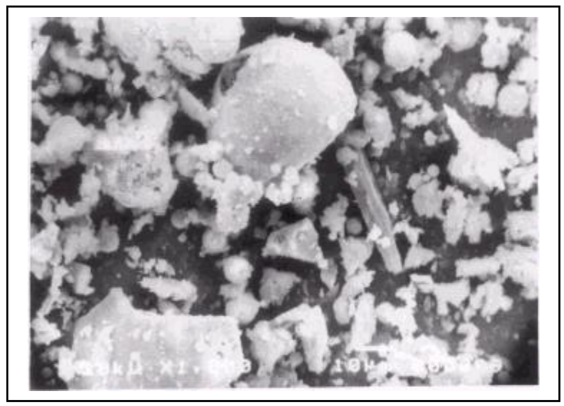

Fig.14 : SEM photomicrograph of fly ash showing the angular particles

2.5 Particle size Distribution of fly ash :

Fly ash as a finer product contributes the performance of the resultant cement and concrete as a pozzolanic additive, as an inert/reactive filler and as an inorganic particulate plasticiser because the sphericity of and smaller size of its particles. As discussed earlier the fineness of the fly ash available from a given source varies considerably depending on the ESP field from which it is collected as well as other operational parameters of the thermal plant. On an average the fineness in terms of residues on 45 microns ranges from 12 to 50 %.

The studies carried out on effect of different size fractions[17] of fly ash on the mortar properties (30 % fly ash) indicated that the each size fraction of the fly ash tends to behave differently and have different influence on the pozzolanic properties

Studies carried out at the authors laboratory [18] have helped evolve an understanding of the influence of the particle characteristics and reaction mechanics of the fly ash component and it could be stated with a high degree of confidence that by optimization of the comminution system, an engineered particle size distribution of fly ash can be achieved in the size fractions of the resultant cement, which enhances the pozzolanic activity fly ash and helps improve the performance of the resultant blended cement. Fig. 15 & 16 illustrates mortar and concrete properties of the Normal PPC and Engineered PPC .

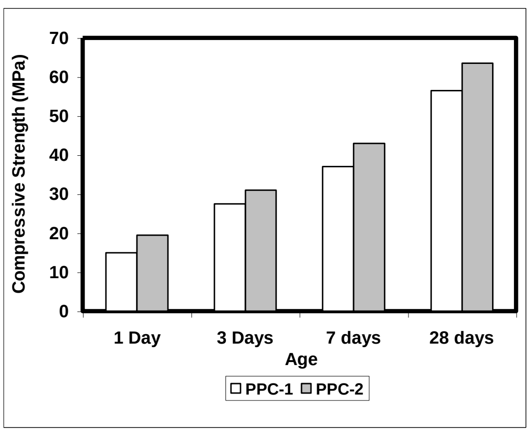

Fig. 15 : Mortar Properties of Normal PPC and Engineered PPC with 25% fly ash

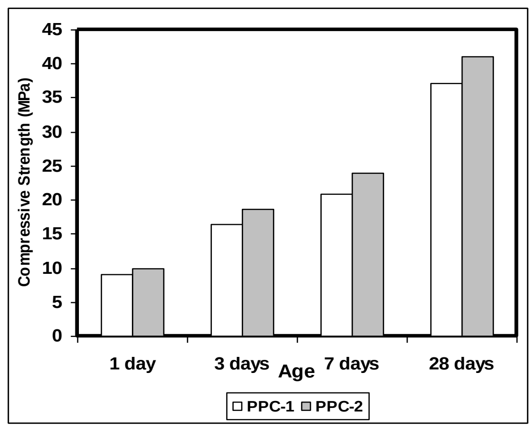

Fig. 16 : Concrete Properties of Normal PPC and with Engineered PPC with 25% fly ash

CONCLUSIONS:

The aspects discussed in the paper illustrates that the low Lime Class-F fly ash available in the country are compositionally most suited for use in Blended cement /blended concrete. A proper understanding of the influences of the fly ash characteristics and with use of proper methods for reducing variability, improving its particle characteristics in the resultant cement can help to engineer the properties of the resultant blended cement concrete.

The empirical equation developed could be useful for assessing the suitability of fly ash for use in Portland Pozzolana Cement. Predicting the fineness of fly ash at which the

L.R and C.R. would be above the BIS requirements. (After determining the R_A value for a given fly ash the Blaine's specific surface required, for achieving LR of 4.5 MPa and CR of 80%, can be calculated). The alkali reactivity test and fineness can thus be used as a rapid test to assess the fly ash quality, its suitability in PPC.

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