

## **Indian Fly Ashes, Their Characteristics, and Potential for Mechano-Chemical Activation for Enhanced Usability**

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### **ABSTRACT**

In India, the generation of electricity is overwhelmingly dependent on combustion of high-ash coal, the present availability of fly ash has already exceeded 130 million tonnes and its generation is likely to touch 170 million tonnes by 2011-12. Although the present utilization of fly ash is close to 50% of the quantity generated, the directive is to effectively utilize the entire quantity that would be generated by the year 2011-12. This objective can only be met, if one looks for newer avenues of bulk utilization. In order to achieve this target several technological endeavours are being made to enhance the quality and reactivity of fly ashes through mechano-chemical activation. This paper addresses the issues pertaining to the intrinsic quality parameters of the Indian fly ashes, potential of chemical activation, feasibility of adopting the newer milling systems such as vibration and attrition milling, etc. Finally, the need for converting the fly ash grains to submicrocrystalline particles and, thereby, significantly altering their properties and behaviour has been touched upon.

### **INTRODUCTION**

The thermal power plants in India are primarily dependent on the combustion of high-ash bituminous coal in pulverized fuel fired systems. Pulverised lignite fired boilers and pressurized fluidized-bed combustion systems are in operation to a limited extent. Hence, the low-lime fly ash (similar to Class F of ASTM C 618) is the prime variety generated in India, although significantly smaller volumes of high-lime fly ash (comparable to ASTM Class C) and PFBC fly ash are available in the country.

In step with the progressively increasing capacity of coal-fired thermal power plants, the quantity of fly ash has been increasing in leaps and bounds as can be seen from Table 1. The table also shows that the rate of generation of fly ash far exceeds the incremental growth rate of its utilization.

In the next three or four years the target of 100 per cent utilization of fly ash likely to be generated is by itself a daunting task. If one considers the expected generation of fly ash over the next two decades, the volume projected is gigantic and its utilization programme will have to be far more challenging than what is perceived today. It is also obvious that no niche utilization strategy would work and one will have to look for newer avenues of bulk usage. This paper makes an attempt to capture the physico-chemical characteristics of the Indian fly ashes, their regular pattern of use, problems and prospects of newer applications, present

modes of quality upgradation including mechano-chemical activation and their prospects and limitations.

**Table 1. Fly Ash Generation and Utilization in India**

Year	Generation, Mt	Utilization	
		Mt	Percent of generation
1993-94	40.0	1.2	3.0
2004-05	112.0	42.0	38.0
2006-07	130.0	60.0	46.0
2011-12	170.0	170.0	100% utilization mandated.
2031-32	600.0	---	Not yet planned.

## PHYSICO-CHEMICAL CHARACTERISTICS OF THE INDIAN FLY ASHES

### Chemical composition

The Indian low-lime fly ashes are characterized by relatively higher concentration of  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  and lower contents of  $\text{Fe}_2\text{O}_3$ . This implies higher fusion temperature for these fly ashes and, consequently, the chances of lower glass formation, if the ash is not subjected to relatively high temperature. While in the low-calcium fly ashes the silica content is almost double of the alumina content, in the high-calcium fly ashes the content of these two oxides is by and large comparable or close to each other. The iron oxide content in the high-lime fly ash is significantly higher than in the low-lime variety. Studies involving sieving, sink-float and magnetic separation, carried out to study the heterogeneity of fly ashes, revealed that the variability of composition is more in the high-lime fly ashes [Das 2003].

A study has been conducted to classify the oxide compositions of fly ashes using radial basis function neural network [Nataraj et al 2006]. The following three main groups of fly ashes have been identified from about 80 sets of data :

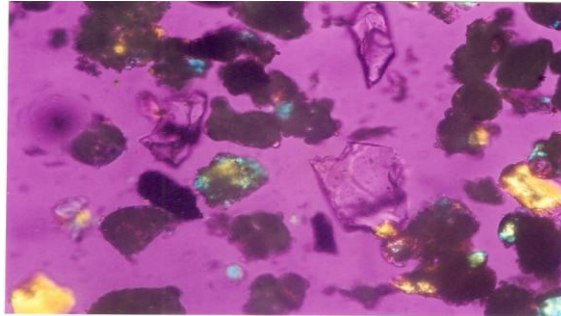
Group Index	$\text{SiO}_2\%$	CaO %	$\text{SO}_3\%$
1	53.0 – 63.1	0.6 – 3.0	NA
2	39.0 – 50.0	3.0 – 16.0	0.8 – 2.5
3	30.0 – 38.0	16.0 – 29.0	2.3 – 3.0

Group Index 1 relates to the proper pozzolanic variety with little or no cementitious properties while the Group Index 2 and 3 cover fly ashes with cementitious properties with progressively increasing heat of hydration.

### Phase composition and glass content

It is widely known that the reactivity of fly ashes is dependent on their glass content and other mineral phases present. It has been observed that the Indian fly ashes are more crystalline than those obtained in other countries, the glass content ranging from 47.0 to 60.9 per cent (Fournier et al 2004). The network theory of glass formation [Zachariassen 1932] provides a theoretical basis to explain the relatively poorer glass content in the Indian fly ashes. Since the ratio of network formers ( $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ ) to network modifiers ( $\text{Na}_2\text{O} + \text{K}_2\text{O} + \text{CaO} + \text{MgO}$ ) in the Indian fly ashes is very high and imbalanced, the glass content is low. Nevertheless, the fly ashes contain spherical

hollow particles (cenospheres), encapsulated hollow particles (plerospheres), angular glass fragments and crystalline grains in varying proportions as shown in Figure 1.

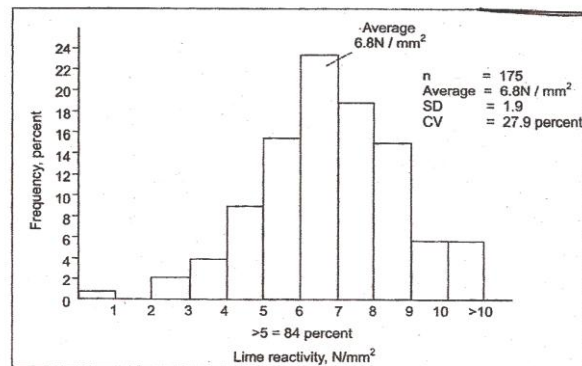


**Fig.1.Fly Ash Particles in Low Magnification (x200) Showing Angular Glass Fragments**

### Specific surface area

It is known that the fly ash is separated from the gases generally with the help of multi-field electrostatic separators and the specific surface areas of the flyashes collected typically vary from about 250m<sup>2</sup>/kg in the first field to along 850 m<sup>2</sup>/kg in, say, the sixth field. Since the final collection hopper contains materials of all the fields, the specific surface areas of the mixed fly ashes as received at the user end are on the coarser side and quite variable [Chatterjee, 2008].

For applications in cement and concrete one of the important quality tests is to check their lime reactivity potential under standard test conditions. The variations in the lime reactivity values of a large number of Indian fly ashes are shown in Figure 2 [Mullick, 2005].



**Fig.2. Lime Reactivity Data on Indian Fly Ashes**

The main requirements, which govern the performance of fly ash in cement and concrete are the Blaine's specific surface area, residue on 45 µm sieve, unburnt carbon content measured as loss on ignition, and reactive silica or the glass content.

It may also be relevant to mention here that the success of fly ash in structural fill and grouting applications depends on the material's ability to be compacted into strong layers. This is primarily a function of particle size distribution and specific surface area. Some road base applications of fly ash depend on the physical effects of the fly ash rather than its reaction with lime.

## BULK USES OF FLY ASHES

### Regular practices

The current regular bulk uses are wellknown and include primarily cement and concrete making.

The present consumption of fly ash in the cement industry alone is estimated at more than 25 million tonnes per year on the assumption that the production of Portland Pozzolana Cement is more than 100 million tonnes per year with incorporation of at least 25 per cent fly ash per tonne of cement produced. If the absorption of fly ash can be enhanced all over the industry to the present permissible limit of 35 per cent, the consumption of fly ash in the cement industry alone may rise to about 60 million tonnes per year by the year 2011-12. The realization of this large potential is dependent, to a large extent, on enhancing the pozzolanicity or reactivity of fly ash by adopting newer beneficiation technologies. It is somewhat doubtful if the present standard specification of fly ash (Table 2) would be adequate to achieve the above target.

**Table 2 Specification of the Indian Low-lime Fly Ash vis-à-vis the US and European Standards**

Parameters	ASTM C-618	EN 450	IS 3812
SiO <sub>2</sub> per cent, min	---	---	35
Reactive SiO <sub>2</sub> , per cent, min	---	25	20
SiO <sub>2</sub> + Al <sub>2</sub> O <sub>3</sub> + Fe <sub>2</sub> O <sub>3</sub> , per cent, min	70	---	70
Total/reactive CaO per cent, max	---	10	10
Residue on 45 µm per cent, max	34	40	34
Blaine's SSA, min, m <sup>2</sup> / kg	---	---	320
Lime reactivity, N/mm <sup>2</sup> , min	---	---	4.5
Cement Reactivity, per cent, min	75	75	80

### Trial application areas

Three potential application possibilities are still at the experimental or trial stage, viz.,

- i. High-Volume Fly Ash Concrete (HVFAC)
- ii. Geopolymer cement
- iii. Fly ash as a multipurpose filler and reinforcement

The status of these trials is outlined below.

### High-volume fly ash concrete

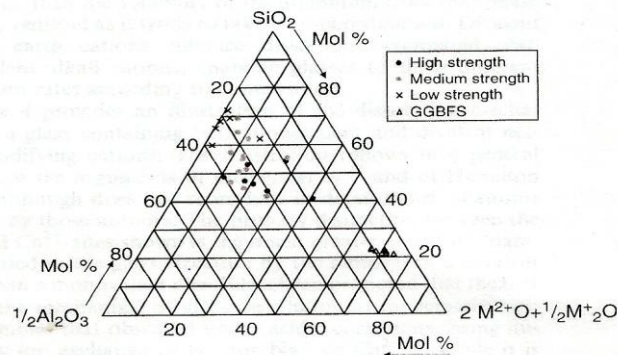
Attempts so far have been to establish the suitability of the Indian cement and fly ashes to meet the product requirements [Basu and Saraswati, March 2006] and to construct concrete pavements on trial basis [Desai 2006]. The present restrictions of the Indian Codal provisions are also being examined [Basu and Saraswati, August 2006]. From the studies carried out it appears that with high-strength cements with 28 days compressive strength of more than 53 N/mm<sup>2</sup> and fly ash of very high reactivity as reflected through their lime

reactivity ( $>6.0 \text{ N/mm}^2$ ) and cement reactivities ( $>90\%$ ), one may achieve upto 70% replacement of cement in HVFAC, using SNF based superplasticisers.

### Geopolymer cements

It is now fairly well known that an alkaline fluid could be used to react with the silicon and aluminum in a source material of geological origin or in by-product materials such as slag or fly ash to produce binders. Since these reactions represent an inorganic polymerization process, the terminology coined was “geopolymer” [Davidovits 1988]. The polymerization process involves a substantially fast chemical reaction under alkaline conditions on Si-Al minerals that result in a three-dimensional polymeric chain and ring structure consisting of Si-O-Al-O bonds [Davidovits 1994]. Low-calcium fly ash has been successfully used to manufacture geopolymer concrete, when the silica and alumina constitutes about 80% by mass with Si-to-Al ratio of about 2. The content of iron oxide usually ranged from 10 to 20% by mass, whereas the calcium oxide content was less than 3% by mass. The carbon content of the fly ash as determined by the loss on ignition, was less than 2%. The designed particle size distribution was found to be 80% particles to be below  $50\mu\text{m}$  [Rangan 2007].

The reactivities of fly ashes with respect to the geopolymeric products have been evaluated with the help of a pseudo-ternary composition diagram shown in Figure 3. [Duxson and Provis, 2008]. From Figure 3 it is clear that ashes with low network modifier content tend to produce poor alkali activation products, with strength generally increasing as a function of network modifier content for low-lime fly ashes.



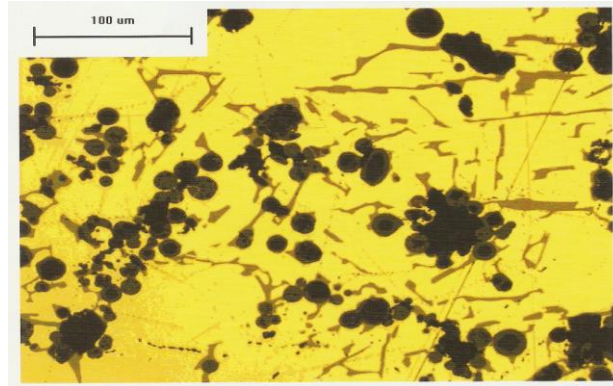
**Fig. 3. Pseudo-Ternary Composition Diagram for Fly Ashes**

The observation that network modifier content is an indicator of the potential inability of a given fly ash in geopolymerization obviously brings attention to the use of high-lime or Class C fly ashes. It has been observed that not only is it possible to utilize Class C fly ash in geopolymers but in fact it can be largely preferable, if the rheology of the mix can be adequately controlled. On the whole it seems that glass chemistry and availability of aluminum to a large degree control the properties of geopolymers.

### Fly ash as a multipurpose filler and reinforcement

Fly ash, in a limited way, is used as a mineral filler in plastics. The results of many experimental studies conducted with fly ash have shown that the addition of fly ash filler increases the stiffness of a plastic formulation, but like most fillers, reduces the impact resistance. However, the purity and particle size are the most important requirements. Apart from polymers, fly ash is often considered as a filler for paints and also as an extender for enamels. Prima facie suitability of as-received fly ashes for these kinds of applications is

rather limited. One of the latest developments is the use of fly ash in metal-matrix composites as shown in Figure 4. [Withers, 2008].



**Fig. 4. Photomicrograph of Ultatite Composite showing Black fly Ash Spheres in Yellow Matrix of Aluminum**

### **CLASSIFIED FLY ASH**

One of the most prevalent technologies adopted by the industry is the classification of fly ash by the high-efficiency cyclone separators into close-range particle size fractions. In a specific study [Chatterjee 2008] pertaining to the improvement in properties of the classified fine fly ash, it was observed that there was significant difference in its particle size and distribution as compared to the initial fly ash (Table 3)

It is evident that the finer fraction of fly ashes display higher pozzolanicity and overall reactivity. This is corroborated by the properties of fly ashes collected from different fields of electrostatic precipitators (Table 4).

**Table 3. Particle Size Characteristics of Processed and Unprocessed Fly Ashes**

Fly Ash	Mean Size, $\mu\text{m}$			Volume (%) above		
	D (v,0.1)	D (v, 0.5)	D (v, 0.9)	10 $\mu\text{m}$	20 $\mu\text{m}$	45 $\mu\text{m}$
Unprocessed	1.9	19.2	76.9	64.7	49.0	24.2
Processed fine traction	0.9	2.0	4.5	0.2	0.00	0.0

**Table 4. Reactivity of Fly ashes collected from different ESP fields**

Characteristics	Field 1	Field 2	Field 3	Field 4
Blain's SSA, $\text{m}^2/\text{kg}$	250	500	600	700
Residue on 45 $\mu\text{m}$	40	15	0.5	Nil
Lime Reactivity, $\text{N}/\text{mm}^2$	5.0	6.2	7.0	7.0

With the help of cyclone separators, one may therefore collect fine fly ash of 600 – 700  $\text{m}^2/\text{kg}$  having lime reactivity of more than 6.0  $\text{N}/\text{mm}^2$ , which may potentially satisfy the demands of increased usage of fly ash in construction but this technology has the shortcoming of generating large volumes of coarse fly ash having disposal problems.

### **PROSPECTS OF CHEMICAL ACTIVATION**

Chemical activation usually refers to the processes of alkali and/or sulfate activation. The alkali activation involves dissolution of glassy phase in fly ash in the increased alkaline environment thereby accelerating the pozzolanic reaction. It is obvious that for glass-deficient fly ash this process of activation may not be effective. On the other hand, the sulphate action involves its ability to initiate reactions with the aluminous phases, thereby generating the strength imparting hydrate phases in cement paste. This, however, has a relation with the order of crystallinity of the alumina bearing phases and their dissolution kinetics. The materials such as  $\text{Na}_2\text{SO}_4$ ,  $\text{Na}_2\text{SiO}_3$ ,  $\text{Ca}(\text{OH})_2$ ,  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  have been tried as activators with some but no spectacular success.

On the other hand, there are certain conceivable routes for manufacturing favourable glasses in fly ash [Duxon and Provis, 2008]. One alternative is to add components into pulverized coal, prior to combustion, to artificially manufacture an ash of predominantly known and desired glass composition; for example, glass phases including lime feldspars may be targeted. The second option is to adopt remelting process to create a clinker of alkali aluminate chemistry. As this process is independent of the initial ash generation, a production facility is necessary to manufacture the highly reactive phases to be blended with otherwise poor-quality ashes. The third route could be the preparation of an ideal glass, using geopolymer as a reaction intermediate; in other words it is a finishing stage of making a geopolymer cement with ideal glass content.

The first two alternatives provide a means for production of value-added raw materials predominantly for two part geopolymeric process, while the third alternative provides a process for production of a one-part geopolymer glass.

## **LIMITATIONS OF NOT REACHING SUBMICROCRYSTALLINE PARTICLE SIZE RANGE IN FLY ASHES**

### **Status of mechanical milling**

In view of shortcomings of the classification process, various milling options in lab and pilot scale have been and are being explored by the Indian cement industry. One of the objectives is the possibility of introducing elastic, plastic and shear deformations to fly ash particles, leading to their fracture and amorphization. The options examined include various mechanical milling systems. It is wellknown that mechanical milling is the most productive method if producing large quantities of nanocrystalline powders of different types of materials such as metals, alloys, intermetallics, ceramics and composites. Milling and mechanical alloying are carried out using high-energy planetary, ball and vibration mills, where the mean size of the produced powders may vary from 200 to 5-10 nm.

Unlike the above experience, the mechanical milling of fly ash has given a different trend of grindability. The typical results of grinding in a small ball mill are shown in Table 5.

By and large, the comparative results of fly ash grinding in the lab-scale ball mills and twin-tube vibratory mills in dry mode as well as in attrition mills in wet mode can be summarized as given in Table 6.

From the table it is evident that in the case of dry milling the vibratory mills displayed significantly lower milling time and better product characteristics. But the attrition mill in wet mode showed even better performance. However, the average particle sizes of fly ashes ground in different milling systems ranged from 3.0 to 9.0  $\mu\text{m}$  and the enhancement of fly ash reactivity was, for all practical purposes, proportional to the levels of fineness achieved

as shown in Figure 5. Since vibro-milling and attrition milling showed some potential to reduce milling time and consequently the energy consumption, the scale-up possibilities of these systems were examined.

**Table 5. Results of Extended Ball Milling of an Indian Fly Ash**

Grinding time, h	Blaine's surface m <sup>2</sup> /kg	Density, g/cm <sup>3</sup>	Lime reactivity, N/mm <sup>2</sup>
0	300	2.10	5.8
1	490	2.30	7.0
2	550	2.35	--
3	630	2.39	--
4	710	2.42	9.5
5	700	2.48	--

**Table 6. Lab-scale Grinding Trials of the Indian Fly Ashes by the Different Milling Systems**

Mill system and mode of milling	Milling time, min	Factor of increase in Blaine surface	Reduction ratio of Median particle size	Factor of increase in Lime Reactivity
Ball mill (Dry)	300-360	3.0 – 3.5	3.0 – 3.8	2.0 – 3.0
Vibratory mill (Dry)	50-60	3.5 – 3.8	5.0 – 6.0	2.5 – 3.0
Attrition mill (Wet)	30-40	3.5 – 4.0	8 - 10	2.5 – 3.0

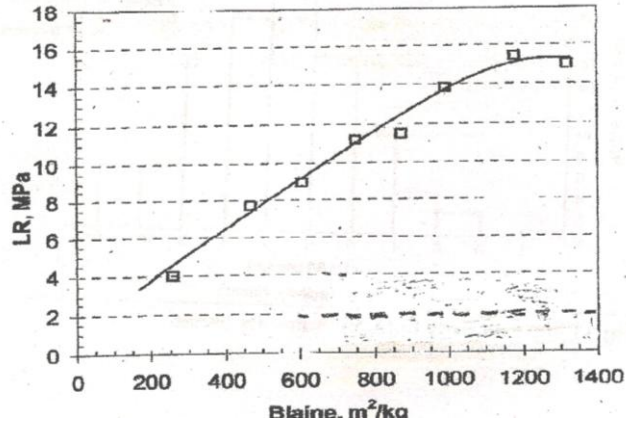
### Scale-up limitations

Two specific attempts have been made in this regards. A pilot vibratory mill of 1.5 tph rated throughput with twin tube of 600 mmφ x 3500 mm length has been installed in an Indian cement plant. The rated mill drive of 90 kW has been provided with 62% volumetric filling of cylpebs of 30 x 30 mm, 25 x 25 mm and 20 x 20 mm sizes. For this model the problems encountered so far have been the non-achievement of rated throughput, desired reduction ratio and the expected specific power consumption. In all the three counts the upscaled version is yet to perform.

Another endeavour has been with a tower mill, as a variant of the attrition mill but in dry mode, keeping in view the fact that the tower mills or vertimills of throughput of upto 100 tph were in industrial use essentially in wet mode. The pilot-scale trial runs with the Indian fly ashes having feed particle size range of 1-100 μm did not yield the d<sub>50</sub> value of even 10 μm with specific power consumption of upto 110 kWh / t of fly ash. Use of coarse media (6 mm), low tip velocity of the stirrer (3 m/s) and dry mode were considered responsible for not achieving the expected performance.

In all the above examples, one feature that comes out quite prominently is that the fineness level of fly ashes has improving effects in various properties of the products in which they are used but the effects can not be seen to be of the level of mechano-chemical activation, which is often caused and characterized by the presence of very large number of surface molecules or atoms, as compared with molecules or atoms in the bulk of the materials. This is perhaps due to the fact that the mean particle size of classified fly ashes still stays in the micron range.





**Fig. 5 : Effect of Increasing Blaine Surfaces on Lime Reactivities of Fly Ashes**

The same reason might be valid when one attempts to compare the performance of classified fine fly ash with silica fume, claimed to be a very effective mineral admixture to make impermeable concrete. Depending on the source, the SiO<sub>2</sub> content may vary between 94-98% in the case of silicon metal and between 86-90% in the case of alloys. The average particle size is about 100-150 nm and the BET specific surface area is 15-25 m<sup>2</sup>/g. Some comparative results are presented in Table 7 [Dattatreya, 2006]

Table 7 has gives an indication that the quantum jump in the properties of the Indian fly ashes can not be expected as long as the mean particle size remains above 1 μm, which has been so far the size reduction limit of the known and potentially upscalesable fine grinding and classification technologies.

Compared to silica fume, the performance of aqueous silica colloid suspensions (known as cembinder) is also worth-noting. [Chandra 2002]. This admixture contains silica colloid particles in an amount of 8-60% by weight of the solutions. The specific surface area of the particles is in the range of 50-200 m<sup>2</sup>/g and the mean size of the particles ranges from 5-200 nm. It is reported that 4% silica colloid consumed 60% more calcium hydroxide than 4%

silica fume in the same hydration period of 7 days. Even in one day the silica colloid reacted more with calcium hydroxide than silica fume. One may therefore observe that the high reactivity of the colloid silica is on account of its nanometric particle size and purity. Another important concrete admixture is metakaolin (mk) containing 51-55% SiO<sub>2</sub> and 40-45% Al<sub>2</sub>O<sub>3</sub>. The BET surface area is in the range of 14-22 m<sup>2</sup>/g with particle sizes of 60-90% under 12 μm and less than 15% above 5 μm. This type of admixture shows a pozzolanic reactivity of about 840 mg CaOg<sup>-1</sup> mk by the Chapelle test [Jones 2002]. This metakaolin with similar particulate dimensions as of fly ash seems to behave in a fairly comparable manner. Finally it may be relevant to compare the behavior of fine ground lime stone filler in cement. It is generally interground with clinker and because of its softness becomes finer than the latter. For an overall specific surface area (Blaine) of 420 m<sup>2</sup>/kg, 50% of the filler can be below 700 nm, compared with 3 μm of clinker. Because of the fineness, it accelerates the hydration of the alite and aluminate phases. Chemically it reacts with the aluminate phase producing a hydrated carboaluminate phase, thus competing with the gypsum [Taylor, 1990].

**Table 7. Performance Comparison of Classified Fine Fly Ash and Silica Fume in Concrete**

	Composition & Properties	Control concrete	Fine fly ash incorporated concrete	Silica fume incorporated concrete
1	Mean particle size, $d_{50}$ , $\mu\text{m}$ , of the mineral admixture	--	3	~ 0.1
2	Addition of mineral admixture (%)	--	10	10
3	Water/cement ratio	0.40	0.40	0.40
4	Cement content in concrete, $\text{kg}/\text{m}^3$	400	357	356
5	Slump, mm	100	80	80
6	Compressive strength, MPa			
	7 day	56.2	54.5	71.0
	28 day	67.9	68.7	80.7
7	Rapid Cl permeability, coulomb			
	7 day	2922	1083	429
	28 day	2340	758	297

From the above examples one may observe that the submicrocrystalline materials behave quite differently from materials coarser than 300 nm. Hence, it is certainly worth looking at the feasibility of bringing down the mean particle size of the crystalline fly ashes in the submicrocrystalline range in order to observe the corresponding improvement in reactivity by the Chapelle test or by the standard lime reactivity test. Using this as the reference point, an exercise needs to be undertaken to optimize the top size of the ultrafine fly ashes particles along with their size distribution pattern so as to achieve the tangible benefits of mechano- chemical activation.

The main scope of this optimization exercise is to answer the following questions for the crystalline fly ashes:

- (a) Is there a sharp and distinctive boundary between the bulk and the submicrocrystalline states ?
- (b) Is there some critical grain size below which the characteristic properties of submicrocrystalline or even nanocrystalline material become observable and above which the material behaves as a bulk one ?

## CONCLUDING REMARKS

It has been observed that by reducing the mean particle size of the fly ashes from 30  $\mu\text{m}$  to below 10  $\mu\text{m}$ , substantial improvement in the flow and strength properties of mortars and concrete are achieved but the enhancement of properties corresponding to further reduction of fly ash particle size to even 3-5  $\mu\text{m}$  is either incommensurate or inconsistent.

The properties of the Indian fly ashes that are highly crystalline, relatively coarse and widely variable, need to be substantially improved for newer and more sophisticated uses. Presently, this improvement in properties is being partially achieved by fine grinding and high-efficiency cyclone separation technologies. Notwithstanding the adoption of such technologies, the size of fly ash particles remains in the range of 3-9 microns. In other words the known and currently practised technologies cannot yield particles in the submicrocrystalline range, which is defined as 300 to 400 nm. Consequently, even with fine

grinding and classification the fly ash properties do not show such changes as demonstrated by the submicrocrystalline or nanocrystalline materials. The difference between the properties of small particles and properties of bulk materials is known and has been utilized in aerosols, pigments, glass coloured by colloidal metal particles etc. A similar approach is, therefore, called for in maximizing the enhancement of properties of fly ashes.

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