

# Effectiveness of Some Techniques for Fly Ash Beneficiation

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**ABSTRACT-** Fly ash can be used as cement admixture. However, fly ash having high un-burned carbon content is not suitable for use as cement admixture. Beneficiation of fly ash is done in order to obtain carbon and fly ash fractions of high purity for their use as value-added products. Beneficiation is based on the difference in physical and surface properties of the carbon and ash. In the present study, effectiveness of froth floatation and fluidized bed separation processes is investigated. Froth floatation alone could reduce the LOI (loss on ignition) of fly ash to ~10% from an industrial fly ash sample having initial LOI value of ~ 15%. The effectiveness of the combination of screening and froth floatation (froth floatation after screening) gave encouraging results. A reduction in LOI to ~ 8% could be achieved using this combined process. Fluidized bed separation's results showed that this dry process can be an attractive alternative. This process showed LOI reduction to ~ 11.5% in a shallow bed.

**Keywords:** Fly ash, beneficiation, Loss on ignition, froth floatation, fluidized bed separation, cement admixture, screening.

## 1.INTRODUCTION

Pulverized coal combustion, for power generation, results in the production of different types of ash in large volumes. The 'fine' ash fraction carried by the flue gas is separated from the flue gas using highly efficient electro static precipitators. This fraction of ash is known as 'fly ash'. In the coal-fired power plant, the coal before burning is ground to powder fineness to improve its combustion. Fly ash, carries the unburned carbon particles and the mineral content of the coal residue, is captured from the exhaust gases of the power plant. This capture fly ash can be a useful resource.

The pollution control norms have mandated the capture of the fly ash as contrary to the past practice of releasing it into the atmosphere. The quality of the captured fly ash considerably varies depending upon the source of the coal.

However, the main constituents of the fly ash are silicon dioxide ( $\text{SiO}_2$ ) (amorphous and crystalline) and calcium

oxide ( $\text{CaO}$ ). Fly ash is commonly used to supplement Portland cement in concrete production, where it can bring both technological and economic benefits. The hardened fly ash concrete shows increased strength together with a lower permeability, where the later leads to a higher resistance toward aggressive admixtures (Taylor, 1997; Manz, 1998; Maroto-Valer et al., 2001). In addition, partial replacement of cement with fly ash reduces the production costs of concrete due to the lower price of fly ash compared to cement (Manz, 1998). Fly ash particles spheroids, and can be conveniently blended with other fine solids. Its flow-ability and easy blending properties make fly ash a desirable admixture for concrete.

When fly ash is used as admixture in concrete it affects the dose of air entraining admixtures (AEAs) required to entrain the proper amount of the air in concrete. The carbon present in the fly ash adsorbs AEAs and the stabilization of the air-water/cement interface is adversely affected. Also, discoloration and segregation of mix components occurs due to high values of LOI. Due to this reason, the high un-burned carbon content in the fly ash makes it unsuitable for use as cement admixture. Usually the carbon content in fly ash is in the range of 2-12% (Hwang et al., 2002). The problem of high un-burned carbon has increased with the implementation of low- $\text{NO}_x$  combustion technologies (Pedersen et al., 2008). For meeting the  $\text{NO}_x$  emission regulation the power companies have installed low  $\text{NO}_x$  burners. As a result, the carbon content of fly ash increases significantly due to combustion at low temperature or using less oxygen conditions which are suitable for low  $\text{NO}_x$  combustion units. When fly ash is to be used as a cement admixture, the maximum permissible concentration of unburned carbon in the fly ash should be 6.0 (wt%). Carbon content in the fly ash is measured by loss on ignition (LOI).

If the carbon content in the fly ash were lowered, more fly ash will qualify to meet the requirement of use as cement admixture. Beneficiation processes are therefore used for the selective separation of unburned carbon from the fly ash. Beneficiation processes are used to extract high purity carbon and ash, which can be subsequently used for the production of value-added products. Beneficiation techniques are based on the differences in size, density,

electrostatic, and physical properties. Un-burned carbon fraction can be used as a fuel or as a catalyst/adsorbent. The purified inorganic fraction can be used as a cement admixture.

Many methods have been attempted to classify the unburned carbon content of ash, including, sieve classification, electrostatic classification (Shilling, 1999), density gradient centrifugation (Maroto-Valer et al., 1999), oil agglomeration (Rubio et al., 2008), wet classification (Walker and Wheelock, 2006), vibration classification, jet mill classification. Each has an advantage and a disadvantage but none has been used in common practice or with great success.

Gray et al. (2002) cleaned an industrial fly ash sample using tribo-electrostatic separation, ultrasonic column agglomeration, and column flotation. The unburned carbon concentrates were collected at purities ranging up to 62% at recoveries of 62%. The authors concluded that the column flotation was the most effective process for the collection of carbon concentrates at a LOI value of 61% and a carbon recovery of 62% with 90% of the ash reporting to the tails with LOI values <8% for the Shawville fly ash. Ban et al. (1997) showed that dry tribo-electrostatic separation of fly ash has the potential to be an ineffective method of separating unburned carbon from fly ash. They performed laboratory tests on a simple parallel flow separator and showed that 60-80% of ash could be recovered at carbon contents below 5%, and 50% of carbon could be recovered at carbon concentrations over 50%.

Michigan Technological university developed a froth flotation process to separate unburned carbon from fly ash with a target of less than 1% carbon in the separated ash fraction with  $\geq 90\%$  recovery of carbon or  $\geq 80\%$  of LOI in the carbon concentrate with  $\geq 70\%$  recovery of total carbon. In this process, a collector is used which selectively coats the surfaces of certain mineral particles and make them hydrophobic. These particles then attach to the air bubbles and rise to the surface of the slurry. Hwang et al. (2002), studied the separation techniques namely, gravity separation, electrostatic separation, and froth flotation. They could generate carbon concentrate with a LOI value of 67 to 80% from these processes. Levy and Sarunac (1997) studied the carbon removal from fly ash in a long horizontal fluidized bed and in a fluidized bed with acoustic speaker. They concluded that the fluidized bed separation process has good commercial potential.

In the literature, froth floatation has been proposed as most promising technique. On the other hand, dry

separation processes (electrostatic, fluidized bed) are being investigated, as these processes do not cause downstream pollution that is caused by wet processes (froth floatation, oil agglomeration). In the present work, the effectiveness of froth floatation and fluidized bed separation techniques for the fly ash beneficiation are investigated.

## 2. MATERIALS AND METHODS

### 2.1 Loss of Ignition

Loss on Ignition consists of strongly heating ("igniting") a sample of the material at a specified temperature, allowing volatile substances to escape, until its mass ceases to change. The loss on ignition of a fly ash sample gives the amount of un-burnt fuel in the fly ash.

#### *LOI determination:*

The crucible was dried at 105 °C for at least 1 hour, and then cooled in a desiccator for 30 minutes. The weight of dried crucible ( $W_C$ ) was noted, fly ash sample (about half full) was added and the crucible was kept in an oven at 105 °C (overnight). Crucible was then cooled in a desiccator and reweighed ( $W_S$ ). The crucible was placed in a muffle furnace at 750°C and left for 2 hours, followed by cooling in a desiccator to room temperature. The final weight ( $W_A$ ) of the crucible was noted. The LOI was calculated as,

$$\text{LOI (\%)} = \frac{(W_S) - (W_A)}{(W_S) - (W_C)} \times 100$$

### 2.2 Froth Floatation

A mixture of fly ash (30 g) and collector (kerosene) was prepared in a 1 l beaker, and agitated thoroughly to ensure uniformity. The air pump was switched on, and the slurry was added into the froth floatation cell. Carbon rich fraction is collected from the top and ash settled at the bottom. The samples collected from top and bottom were filtered and analyzed for LOI.

### 2.3 Fluidized Bed Separation

A cylindrical glass tube (height = 2' and diameter = 1.5") was used for fluidizing the fly ash particles. A sintered disk is provided at the bottom of the column for proper air distribution and to avoid choking of the air inlet

connection. A regulated air supply was connected to the air inlet. The particle collection arrangement was designed so as to recover all the particles from the exhaust air. Fly ash sample was charged into the column. Airflow was increased steadily using a pressure regulator till the bed was uniformly fluidized. The fine particles escaping from the bed were collected in a flask. The particles that were escaping with the air were collected, by contacting the outgoing air with water. LOI values of the fraction elutriated and the fraction retained in the bed were determined.

### 2.4 Sieve Analysis

Sieve analysis involves a nested column of sieves with wire mesh cloth (screen). A stack of four sieves having sizes 90 $\mu$ , 75 $\mu$ , 45 $\mu$  and 25 $\mu$  and a pan were used for sieve analysis. A 100 g sample was put into the top sieve which has the largest openings. Each lower sieve in the column has smaller openings than the one above. At the base was a round pan, called the receiver. The column was placed in a mechanical shaker. The sieving was done for 10 min. After the shaking was complete the material on each sieve is weighed. The weight of the sample of each sieve was then divided by the total weight to give a percentage retained on each sieve.

## 3. RESULTS AND DISCUSSION

The fly ash sample, obtained from NFL Nangal, Punjab, was taken for LOI determination. All the fly ash was well mixed before subjecting to LOI determination and beneficiation. The well mixed sample of fly ash showed good reproducibility for LOI determination as shown in Table 2. These results show that the LOI of the fly ash is 14.9 $\pm$  0.2 (wt%).

**Table-1:** LOI of fly ash sample obtained from NFL, Nangal

Trail no.	LOI (wt%)
1	15.08
2	14.79
3	14.72

### 3.1 Froth Floatation Process

The froth floatation process is shown to be very promising for fly ash LOI reduction (Hwang et al., 2002). However,

the success of this process depends on the doses of collector/frother. In the present study, few initial experiments were done without the use of collector. In all the experiments 15 g fly ash sample was taken in beaker and slurry was prepared. This slurry was slowly added into the floatation cell and the overflow was collected in a beaker. Separation was not obtained as the carbon particles did not attach with the rising air bubbles. Figure 1 shows the top surface of the floatation cell.



**Figure-1:** Froth floatation separation without collector dose

A second set of experiments was conducted by using a collector (kerosene) dose of 5 ml in 15 g fly ash sample. In this case the carbon particles got coated with the collector and rose to the surface after attaching with the air bubbles. Figure 2 shows the top surface of the floatation cell and the collecting beaker when the collector was added to the fly ash sample. The color difference in figures 1 and 2 shows the collection of carbon at the top surface in Figure 2.



**Figure-2:** Froth floatation separation with collector dose

The LOI results of top and bottom fractions for these experiments were reproducible and are reported in Table 2. The results show that with the froth flotation process when collector dose is 5ml/15 g of fly ash sample, the LOI of bottom fraction was reduced to 10±0.5 (wt%) and the carbon concentrate in the top fraction had had LOI 48±1 (wt%).

**Table-2:** LOI of top and bottom fraction of froth flotation cell

Trail no.	LOI of top fraction(%)	LOI of bottom fraction(%)
1.	48.30	10.42
2.	48.87	10.37
3.	46.91	9.64

Figures 3 and 4 show the fractions obtained from froth flotation. The difference in the color of top and bottom fractions is due to the difference in carbon content of these fractions.



**Figure-3:** Bottom fraction from froth flotation



**Figure-4:** Top fraction from froth flotation process

In the following set of experiments, the collector dose was optimized. For these experiments 30 g sample of fly ash with varying dose of collector was subjected to separation by froth flotation. The LOI of top and bottom fractions were determined and are given in Table 3. The results show that a collector dose of 0.5 ml/30 g fly ash was sufficient for the separation. Therefore, for further experimentation the optimum collector dose of 0.5 ml/30g of fly ash sample was used.

**Table 3:** LOI of froth flotation fractions with and varying collector dose

Collector dose (ml)	Fly ash sample (g)	LOI of top fraction(wt%)	LOI of bottom fraction(wt%)
4	30	46.04	10.42
3	30	42.35	10.77
2	30	47.73	9.91
1	30	46.21	10.09
0.5	30	51.62	10.13
0.25	30	48.72	13.71

### 3.2 Screening Followed by Froth Flotation

The effectiveness of froth floatation for various size fractions is investigated. For this purpose, the fly ash samples were first sieved and the various fractions obtained were then subjected to separation by froth

flotation. Four sieves having sizes 90 $\mu$ , 75 $\mu$ , 45 $\mu$  and 25 $\mu$  were used for sieve analysis. Table 4 shows the LOI obtained for different sieve sizes. As expected, the sieve size < 25 $\mu$  gave least LOI value.

**Table-4:** Sieve analysis results

Particle size	Weight obtained (g)	Mass fraction(%)	LOI (%)
>90 $\mu$	74.89	27.33	15.74
<90 $\mu$ &> 75 $\mu$	21.72	7.93	22.13
<75 $\mu$ &> 45 $\mu$	81.95	29.91	11
<45 $\mu$ &> 25 $\mu$	67.28	24.55	10.02
< 25 $\mu$	28.12	10.28	8.07

### 3.3 Screening Followed by Froth Flotation

Initially 20 g sample of fly ash was taken for these experiments, but the results were not reproducible. Then 10 g sample of fly ash (shallow bed) was subjected to fluidized bed separation. For these experiments, the reproducible results were obtained. Table 6 shows the results obtained from the fluidized bed separation process, the LOI reduced to 10.4  $\pm$  1% in top fraction.

**Table-6:** LOI of fly ash fractions obtained from fluidization bed separation

Trail no.	LOI of top fraction(%)	LOI of bottom fraction(%)
1.	11.39	20.25
2.	10.96	18.76
3.	9.52	17.91
4.	11.25	20.61
5.	11.18	20.26

### 3.4 Fluidized Bed Separation

Figures 5 and 6 show the fractions obtained from fluidized bed separation process. Color difference in the bottom(high LOI) and top(low LOI) fractions is visible.



**Figure-5:**Top fraction from fluidized bed separation



**Figure 6:** Bottom fraction from fluidized bed separation

### 4. CONCLUSIONS

The froth flotation process could reduce the fly ash LOI from ~15% to ~10%. Screening of the fly ash sample before subjecting to separation by froth flotation showed promising results in LOI reduction. For all three size fractions below 75 $\mu$ , the LOI could be reduced to ~8%.

Fluidized bed separation process studied for LOI reduction showed promising results. In a shallow bed, the

effectiveness of the fluidization process was close to froth flotation process. The major advantage of using fluidized bed process is its environmental friendliness as the wet process (froth flotation) causes several other environmental issues.

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