

# Study of Characteristics of Ground Coal Fly Ash for Potential Use as Filler in High Opacity Specialty Papers

**Abstract:** Industrial solid waste handling and its processing for reuse in industry is very important for sustainable industrial growth and conservation of natural resources and environment. Pulverized coal fly ash obtained from the burning of pulverized coal in thermal power plants is one of the most prominent solid wastes in most of the countries at global scale. The fly ash can be beneficiated by different mechanical and chemical processes to give suitable properties as fillers. In this direction, a novel experimental study has been done for use of pulverized coal fly ash as filler in colored laminate base paper. The particles of fly ash are higher in size in comparison to conventional fillers. So grinding is one of the best options to improve its property as filler material. The ball mill grinding has been studied for reduction of particle size and improvement of surface area. The mechanical strength properties, optical properties like brightness, opacity and surface properties of paper hand-sheets were studied. Chemical compound analysis of fly ash sample using XRF has shown its potential for use as wet end filler in laminate base paper. Carbon percentage in fly ash using CHN analyzer and LOI method has been carried out to see the effect of grinding on carbon percentage as the brightness of fly ash decreases with increase of carbon percentages and deteriorates its quality as filler. Laser particle size analysis for particle size distribution and density distribution of fly ash along with XRD analysis of fly ash for crystalline structure before and after grinding are done. The opacity of fly ash loaded paper was better than conventional fillers. Strength properties were also comparable.

**Keywords:** Fly ash, Fillers, Pulp, Paper, Opacity.



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## 1. Introduction

The sustainability concepts of industrial ecology advocates that the by-product generated in a particular industrial process should be assimilated by other industrial activities to conserve and optimize natural resource consumption. This minimizes the overall material and energy consumption as well as reduces environmental impacts and economic costs [1]. A large amount of fly ash is dumped in the near-by landfills or impounds of power plants where it mixes in air, water and soil. Current annual production of fly ash from coal based power plants is 112 million

tons in India. More than 65,000 acres of land is occupied by ash ponds for disposal of fly ash. A number of policies have been framed by Govt. of India for utilization and disposal of fly ash [2].

Coal fly ash (CFA) processing provides the perfect opportunity to increase the awareness of the potential for industrial synergy because of the vast number of potential applications that the CFA or the CFA derived products can be used in. However, in order to facilitate some of the further development work that is required to turn some of these research works in commercialized reality, there has to be more attention paid to some of the

driving factors at the research stage. Economic evaluation of the applications would give a greater indication of the areas in the research that should be focused on; currently the economic data within these CFA application areas is scarce [3]. A more up to date estimate would mean that 750 million tonnes of CFA is generated on a global basis each year. Current CFA utilization figures are 39% in the US and 47% in Europe [4, 5]. The global average is estimated to be close to 25% [6]. Current forecasts predict that the next two decades will see the installation of the same amount of power generation capacity as that installed over the whole of the 20th century. This is due to its abundance in

energy intensive countries such as China and India, coal is likely to become an increasingly dominant fuel for power generation [7]. Several reviews of the utilization of CFA have been conducted, but the overviews of multi-component utilization of CFA are only 5 and 10 years old. These overviews address a wide range of sustainability issues within both the fuel and minerals sector. [8-14].

The principle components of fly ash are silica, alumina, ferrous oxide, and calcium oxide with varying amounts of carbon as measured by a Loss on Ignition (LOI) test [15]. The American Society for Testing and Materials (ASTMs) group CFA into two classes: C and F. The class F CFA has a combined SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, and Fe<sub>2</sub>O<sub>3</sub> content of greater than 70% compared to greater than 50% for Class C coal fly ash. It is for this reason that it is sometimes held that class C CFA is derived from lignite and sub-bituminous coals and class F CFA is derived from bituminous and anthracite coals [16]. Inter-particle variations in particle diameter and chemical composition in coal fly ashes are significant [17]. Bituminous and lignite CFAs that contain less than 10% CaO in total often consist mainly of aluminosilicate glass and usually do not contain any crystalline compounds of calcium. CFAs that contain more than 15% total CaO are composed of calcium aluminosilicate glass in addition to substantial proportions of crystalline calcium compounds [18]. One of the possibilities for particle sizes exceeding 90 micro meters is that they are made up of the organic constituent or the un-burnt coal (char) components. It has been shown that the larger fractions of a CFA contain a greater content of carbon particles [19].

The phase-mineral and chemical composition of magnetic (MCs) and char (CCs) concentrates recovered from five fly ashes (FAs) produced in four large Spanish thermo-electric power stations were characterized. The MCs and CCs were isolated by magnetic separation and sieving processes. The data reveal that LOI at 500 OC (16 h) or 815 OC (1 h) can be successfully applied for the faster determination of char contents in the FAs and FA fractions studied according to the ultimate analyses and LOI results [20].

Density classification of an ash is a useful technique in optical characterization because the optical properties (more specifically, the complex refractive index) of a fly ash particle depend on its composition. The scattering and absorption characteristics of a fly ash particle also depend on its geometry [21, 22].

Coal fired power plants generate about 23 % of the electricity consumed worldwide [23]. Significant variations in the bulk density (800-980 kg/m<sup>3</sup>), porosity (45-57%) and surface area (0.138-2.3076 m<sup>2</sup>/g) were recorded in study of fly ashes of different plants in India [24]. Coal fly ash elemental analysis on dry basis shows Si (29.39%), Al (13.40%), Ti (1.01%), Fe (1.58%), Ca (1.71%), Mg (0.77%) and O<sub>2</sub> (50.43%) as the major elements present [25]. The Fly ash sample from Talcher thermal power plant, Orissa has been analyzed for its particle size and chemical composition. It shows that the fly ash is grayish white in color, having bulk density of 1gm/cm<sup>3</sup> and specific gravity of 2.13. The specific gravity of fly ash is lower than its components such as quartz, mullite, magnetite and glass, which indicates that it is compositionally heterogeneous and contains many hollow spheres. It is very fine grained (d<sub>50</sub> = 16.1 micro meter). The major elements present are Si (SiO<sub>2</sub> = 58%) and Al (Al<sub>2</sub>O<sub>3</sub> = 26%), which are corroborated by an abundance of quartz and mullite [26].

Some of the common paper making fillers like kaolin clay (typical composition of 39% Al<sub>2</sub>O<sub>3</sub>, 46% SiO<sub>2</sub>, and 13% H<sub>2</sub>O), hydrated silica (consists of 78% SiO<sub>2</sub>, 5% CaO, and 17% H<sub>2</sub>O), aluminum tri-hydrate (65% Al<sub>2</sub>O<sub>3</sub> and 34% H<sub>2</sub>O), and iron oxide have the same constituents as available in fly ash [27]. The shape and particle size of materials used as filler in paper are very important. The addition of filler affects the pore structure and its interaction with paper properties is found to be important. To produce effective filler-paper interaction, the particle content in the sheet must be high [28].

Physical and chemical characteristics of mechanically treated fly ashes have been investigated by other scientists. The grinding of fly ash using laboratory mill for 10 to 60 minutes time have been studied. This is reported as increase of fineness of the samples with grinding time but loss of effectiveness occurred for grinding time longer than 20 minutes. The ground samples showed higher specific gravity (increases from 2.44 to 2.69 g/cm<sup>3</sup>) probably due to presence of hollow particles like cenospheres in the original fly ash. The mean diameter decreases from 32.19 to 5.93 micro meters and median diameter from 15.64 to 3.58 micro meters (μm) in 60 minutes. The specific surface area increases from 3980 to 8930 cm<sup>2</sup>/g. In few minutes, the majority of particles with diameter greater than 50 micro meters have been crushed; the percentage of particles greater

than 30 μm is negligible after 30 minutes of time. Only a little change in mineralogical composition of fly ash was reported during grinding process. This little change was due to formation of calcium carbonate resulting by reaction of calcium oxide with carbon dioxide [29].

The grinding also results in changes of particle shapes due to breaking down of coarse particles. A large proportion of the fly ash particles remained unaltered and retained their spherical shapes. The bulk specific gravity decreases from 1.54 to 1.13 g/cm<sup>3</sup> [30]. The grinding process of circulating fluidized bed combustion (CFBC) fly ash has been divided into three stages. The increase in fineness of ground CFBC fly ash is very sharp in the first stage (0 to 30 minutes), then slows down in the second stage (30 to 50 minutes), and in the last stage the change is very slow or negligible (50 to 70 minutes). When the particle size diameter is more than 1 micro meter, it is observed that the particle size distribution of the ground CFBC fly ash follow the RPSB distribution [31]. The dry milling of fly ash using tungsten carbide Tema mill has been studied for 2 minutes milling time. The BET surface area has increased from 7.2 to 10.0 m<sup>2</sup>/g and dry loose bulk density decreased from 0.93 to 0.89 ton/m<sup>3</sup>. The mean particle size decreases from 31.3 to 11.6 μm where as D<sub>90</sub> (90% of particles have diameters smaller than this size) particle size decreases from 80 to 30.6 μm [32]. The beneficiation of fly ash can be done by using magnetic separation as the magnetic fraction will constitute of iron oxide and other magnetic constituents to higher extent. The non magnetic fraction will retain lower amount of iron oxide (70-75% reduction). Rice straw, a solid agro waste material has been used as fiber source. Acetic acid based chemical pulping process converts the agro waste in to a resource for natural fibers. Wheat straw and rice straw are the new promising natural fiber sources available in abundant quantities throughout the world [33, 34]. Preliminary study of coal fly ash as filler without grinding has been done resulting in high opacity but lower brightness with comparable strength properties. But the effect of particle size reduction by grinding and its effect on paper properties has not been studied till now [35, 36, 37].

## Materials and Methods

### Materials

The rice straw pulp has been used as major fraction for sheet preparation. This was manufactured in paper technology laboratory using H2SO4



catalyzed acetic acid pulping. The acetic acid pulping provides higher opacity in paper due to nearly 75–80% retention of silica present originally in raw material. This has been earlier studied for optimization of process parameters by Sinha [33]. Bleached rice straw pulp and long fiber southern pine pulp were used in the ratio of 80:20 for paper sheet making. The long fiber southern pine pulp was obtained from nearby paper industry. The rice straw and pine pulp were having brightness of 72.2% and 89% ISO respectively. Coal fly ash samples (From three boilers) were collected from thermal power plant, Bathinda, Punjab (India).

#### Pulverized coal fly ash processing and testing

The sample of fly ash was processed in ball mill for 1 hr for grinding. The ground fly ash was sieved for 30 minutes in a sieve shaker using sieves no. 36, 60, 85, 150, 300, and 400 (B S 410 1986). The finest fraction, passing through 400 mesh no. (Screen opening size of 38 micro meter) was used as filler in paper making. The sieve analysis of particle size of the fly ash sample is shown in table 3. The particle size and density distribution analysis were carried out using HELOS (H1004) AND SUCELL laser particle size analyzer from sophisticated instrumentation center for applied research and testing, Vallabh Vidyanagar. The compound analysis of original and ground fly ash samples were carried out using X-ray fluorescence spectrometer from IIT Mumbai. XRD analysis was done from sophisticated analytical instrument facility, Punjab University, Chandigarh. XPERT-PRO diffractometer system was used with reflection spinner stage maintaining start and end angle position of 5.0084 and 79.9614.

#### Hand sheet preparation

Dilute slurry (1% solid) of titanium dioxide as well as fly ash (non magnetic fraction) was prepared for better mixing in pulp slurry. Preparation of paper hand-sheets were done by taking 200 grams of O.D. pulp. The pulp was treated in disintegrator and then diluted to 2% consistency for beating in lab valley beater up to 40 OSR. Fillers were added in pulp slurry in 0, 10, 20, 30 and 40 wt% ratio based on oven dry pulp amount. The pulp slurry was diluted to 0.8 % consistency and then paper hand sheets of 100 GSM were made on lab sheet former with different wt percentages of fillers. The wet papers were pressed in lab sheet press and then air dried for 24 hrs.

#### Analysis of paper (hand sheet) samples

The paper hand sheet samples were placed in an environmental chamber for three hours which

was maintained at a temperature of 25 OC and 52% relative humidity. Important properties like brightness (ISO), opacity (ISO), burst strength, tear strength, tensile strength, Gurley porosity, Bendtsen roughness were measured as per TAPPI test methods.

#### Experimental design

Experiments were planned to obtain nearly 0%, 5%, 10%, 15%, and 20% ash levels in the paper hand sheets. The following experiments were conducted:

1. Five hand sheets of 100 g/m<sup>2</sup> were prepared without adding any filler
2. Titanium dioxide was added in pulp slurry to make its weight percentages on oven dry basis as 10, 20, 30 and 40 in pulp slurry.
3. Five hand sheets of each percentage were made on semi automated lab Sheet former.
4. The samples of hand sheets (five for each composition) loaded with fly ash were made and stored.

Study of effects of pulverized coal fly-ash addition as wet end filler in paper making

1. Fractionation of fly ash based on particle size
2. Experimental design for beneficiation of fly ash to make it suitable as filler
3. Addition of fly ash as filler in different percentages for paper hand sheet preparation
4. Testing of paper samples
5. Effect of different process parameters for better and economical production

#### Results and Discussion

Chemical analysis of fly ash is shown in table 1. Silica and alumina are the main constituents. The first four components silica, alumina, iron oxide and titanium oxide comprise nearly 96 percent which are common fillers for specialty grade papers and paper boards.

The important physical properties of fly ash (which are relevant for filler) are shown in Table 2. The grinding improves the brightness of fly ash as very fine carbon particles undergo oxidation and reduces in ground fly ash. Carbon particles are the main reason for dark color of fly ash.

Residual Carbon analysis and Loss on ignition of fly ash

Residual carbon percentage in fly ash used as wet end filler was measured by CHN analyzer. Residual carbon in ground fly ash is 4.67% where

as in original fly ash it is 6.35 % .LOI is measured according to ASTM standard C 311. LOI value of ground fly ash and original fly ash are 5.10 % and 6.50 %.

Fractionation of fly ash sample using sieve analysis before and after grinding process for different time interval are shown in Table 3 and Table 4.

The fractions obtained by sieve analysis was further analyzed by laser particle size analyzer to find the comparison of density distribution based on particle size of original fly ash (denoted by FA), ground fly ash passing through 38 micron size sieve (denoted by FA (ground)), after burning for one hour time at 900 OC (denoted by FA-ORG-B), ground fly ash passing through 53 micron size sieve (denoted by FA-G53) and ground fly ash passing through 53 micron size sieve and burned for one hour time at 900 OC (denoted by FA-G53-B).

This was analyzed from graph in Fig.1 that grinding provides sharp increase in the particle density especially for medium size particles (from 8 micron to 64 micron). Density of particles increased to nearly double its original value after grinding. This is due to hollow particles in fly ash initially which become dense after breakage of hollow particles in this size range. The most of the particles are below 64 micron size.

The burning of different fly ash samples does not show any significant change in density distribution. The higher size particles have very low density showing presence of lighter and hollow particles like carbon.

The fractions obtained by sieve analysis was again analyzed by laser particle size analyzer to find the Cumulative mass distribution based on particle size of original fly ash, ground fly ash passing through 38 micron size sieve and ground fly ash passing through 53 micron size sieve.

This comparison is given in graph in Figure 1, which shows that original fly ash has particles size from 4 micron to 256 microns where as particles retained on 53 micron sieve has variation from 4 microns to nearly 100 microns. But after grinding all the particles are below 64 microns. The variations of size in different sieve fractions are mainly because of irregular shapes of particles.

The effect of grinding on particle crystallite size of coal fly ash has been shown in table 5 and 6. The XRD analysis tables show that the size of crystallite decreases as compared with original fly

ash samples (FA-0). Crystallite size of original and ground fly ash: The crystallite size is measured at different angles. The crystallite size of ground fly ash shows significant decrease from original fly ash.

$$\text{Crystallite size} = \frac{(K \cdot L)}{(\text{FWHM} \cdot \cos \theta)}$$

Where K = Shape factor,

L = Wavelength of X-ray used in diffraction = 1.5406,

FWHM = Full width at half maximum,

$\theta$  = Position angle measured as  $2\theta$

#### Effect of Filler percentage on different properties of paper:

Most of the mechanical strength properties like burst, tensile and tear strength are better for ground fly ash loaded paper in comparison to paper loaded with specialty filler titanium dioxide. The brightness of sample paper loaded with fly ash is poor in comparison to titanium dioxide loaded paper as shown in figure 3 but opacity is superior as shown in figure 4. Effect of filler on burst index of paper as shown in figure 5 demonstrates that both the filler particles have similar effects and fly ash has better effect. Similarly, effect of filler on tensile index of paper as shown in figure 6 demonstrates that both the types of fillers have similar effect trends and fly ash loaded paper has superior strength. But, the effect of filler on Gurley porosity of paper as shown in figure 7 demonstrates that both the types of fillers have similar effect trends and fly ash loaded paper has shown lower time requirement for passage of same volume of air through the sample surface in comparison to titanium dioxide loaded paper. Again the effect of filler on tear factor of paper as shown in figure 8 demonstrates that both the types of fillers have similar effect trends and fly ash loaded paper has more tear strength in comparison to titanium dioxide loaded paper.

#### Mechanical Strength properties of Paper from acetic acid pulp without filler loading:

Since rice straw pulp has been used for paper making. So the analysis of paper without filler is important to see the effects of fillers in specialty papers with high filler loadings.

Mechanical Strength properties were determined for pulp obtained by acetic acid pulping after treating in lab valley beater. The beaten pulp was having 40 OSR. 100 GSM paper was made and tested for burst strength, tear strength and tensile

strength. Burst index of paper samples varied from 0.52 to 0.65 kPa.m<sup>2</sup>/g. Tear index of paper samples varied from 3.25 to 3.38 mN.m<sup>2</sup>/g. Tensile index has values in between 22 to 23.90 N.m/g and the average value is 22.99 N.m/g.

#### Optical properties of Paper from acetic acid pulp

Brightness of paper measured at 457 nanometer wavelength of light in terms of ISO brightness has shown that the pulp has brightness in between 25.38 to 26.81 % ISO. This is good and can be easily bleached to 80 – 85 % ISO brightness by conventional bleaching sequences.

Opacity of paper is varying in between 97.9 to 98.8 %. This is very high and makes the paper very good for writing and printing purpose as readability improves with increase of opacity. The unbleached rice straw pulp taken for bleaching has Kappa number 16.53 and brightness of 25.38 % ISO. The final brightness is 81.89 % ISO.

#### Economics & Benefits of fly ash

##### Economic consideration of use of fly ash as filler

Using the Bond's law for dry grinding by an industrial ball mill in either open (approximated in batch tests) or closed circuits and resulting in products that could reach significant fineness,

Calculation of Power consumption, W is as follows:

$$W = 13W_i \left( \frac{1}{\sqrt{P_{80}}} - \frac{1}{\sqrt{F_{80}}} \right) \left( \frac{P_{80} + 10.3}{1.145 P_{80}} \right) K$$

Bond ball milling experiments resulted in a Bond work index  $W_i$  equal to 36.7 kWh/t for fly ash (Cordeiro et al. 2009, Herbst & Lichter 2006).

Power consumption,  $W = 477.1 * (0.169 - 0.089) * (45.3/40.075) * 1$

$= 477.1 * 0.08 * 1.130 = 43.129 \text{ kWh/t.}$

At current price rate of electricity of 7.5 Rs. per kWh,

(One US dollar = 50 Indian Rs. approximately)

Cost of electricity =  $43.129 * 7.5 = 323.46 \text{ Rs./t} = 0.32 \text{ Rs. / kg.}$

(This is equivalent to 6.5 USD for grinding of 1000 kg of fly ash)

So, total cost of fly ash with all the processing will be taken as 1000 Rs. /t of fly ash which includes all the other costs.

Coal fly ash is available at free of cost from thermal power plants.

Considering 22 % of filler in paper,

Amount of actual filler to be added in stock preparation for getting require percentage:

Basis of calculation: 100 tonnes of O.D. paper  
(1 tonne = 1000 kg)

Amount of filler in paper = 22 tonnes

Actual amount of filler required = amount of filler in paper / retention of filler

(For 100 GSM paper, retention is 70.2 % for fly ash and 65 % for Titanium dioxide.)

Fly ash required =  $22 / 0.702 = 31.34 \text{ tonnes}$

Kaolin clay required =  $22 / 0.65 = 33.84 \text{ tonnes}$

Cost of processed fly ash as filler =  $31.34 * 1000 \text{ Rs.} = 31340 \text{ Rs.}$

Cost of kaolin clay =  $33.84 * 80000 \text{ Rs.} = 27,04,200 \text{ Rs.}$

So, saving in terms of cost of filler =  $27,04,200 - 31340 = 26,72,860 \text{ Rs.}$

(This is equivalent to 48,000 USD)

Considering cost of industrial manufacturing of laminate base paper = 100, 000 Rs. /tonne

Percentage reduction in cost of manufacturing =  $(26,72,860 * 100) / (100 * 100,000) = 26.73.$

So approximately 27 % cost reduction can be done by replacement of convention filler

by coal fly ash for a 100 GSM paper with 22% filler content. The cost reduction will be higher for higher filler percentage in paper.

First benefit is the negligible cost of fly ash in comparison to other fillers used in paper, so it will decrease cost of paper. Secondly, it will result in higher opacity of paper for same filler percentage in paper. Mechanical strength properties are superior to that of conventional fillers added paper. Again environmental degradation may be avoided by having proper use of fly ash.

#### Conclusions

The major fraction (75%) of pulverized coal fly ash has size in between 4.5  $\mu\text{m}$  to 105  $\mu\text{m}$ . About 10 % of mass has particle size below 6.61  $\mu\text{m}$  and 16% of fly ash has particle size below 10.30  $\mu\text{m}$ . X50 of fly ash sample is 47.06  $\mu\text{m}$ . Grinding will increase the percentage of particles in smaller range. A grinding time of one hrs results in bringing X50 of particles at 18.74  $\mu\text{m}$ .



The grinding process also affects the shape. The crystallite size decreased with grinding. Silica, alumina and iron oxide are the main chemical constituents making 95% mass fraction of fly ash. Grinding will increase the percentage of particles in smaller range. A grinding time of one hrs results in bringing 90 % of particles in the range below 38  $\mu\text{m}$ .

Titanium dioxide, costly filler is present in fly ash in reasonable quantity, 1.5 % on mass basis. Residual carbon in fly ash is 4.67%. Fly ash has the potential to be very good filler for laminate grade colored base papers where very high opacity is required. Printing opacity of paper can go above 99% for 21.5% filler loading in paper.

Strength properties are good for laminate base special paper. This will decrease the production cost of paper significantly. This is a remarkable beneficiation of two waste materials like pulverized coal fly ash by grinding and rice straw for manufacturing of valuable industrial product like laminate base papers and other fiber-filler composites for industrial purposes.

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