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Experimental Studies and Analyses for Basic Characterization of Fly Ash

Maheboobsab B Nadaf and J. N. Mandal

Enormous quantity of fly ash is being produced as byproduct in the coal industries challenging its safe disposal as land-to-population ratio is very low in country like India. Therefore, it is required to recycle the fly ash effectively in various Geotechnical applications. Before handling the fly ash, its physical, chemical and mineralogical properties should be known. The properties of fly ash may vary from place to place depending on the type of coal burnt. In this paper, an attempt has been made to determine the physical, chemical and mineralogical properties of fly ash collected from Dahanu Thermal Power Station, Thane, India. The physical properties were determined in Geotechnical Engineering laboratory as per the Indian Standards. Results show that the fly ash is non-plastic silt sized material. X-Ray Fluorescence (XRF), Carbon-Hydrogen-Nitrogen-Sulphur (CHNS) and Scanning Electron Microscope (SEM) tests were conducted to determine the chemical properties, C-H-N-S content and surface topography of the fly ash respectively. XRF test results indicate that the fly ash is less cementitious and hence, categorized as Class F. It was observed from SEM that most of the fly ash particles are spherical shaped with very smooth surface. The mineralogical properties of fly ash were also investigated by conducting X-Ray Diffraction (XRD) test. It was observed that the minerals present in fly ash are mainly Quartz (maximum) and Mullite.

Field of Research: Civil Engineering

1. Introduction

Conserving the natural resources and energy is a major concern in recent years. In the direction to conserve the natural materials, several by or waste products have been proposed for use as alternative construction materials. One by product that has shown substantial guarantee as an alternative construction material is fly ash. Fly ash, a byproduct of coal industry and recognized as a pollutant, is generated due to the combustion of pulverized coals in the Thermal Power Plants. The coal-based thermal power plants have been a major source of power generation in India where 75% of the total power obtained is from coal-based thermal power plants (Senapati 2011). In India, near about 120 coal based thermal power stations are producing about 112 million tons fly ash per year. With the increasing demand for power, fly ash generation is expected to reach 225 million tons by 2017 and the use of fly ash is only 45 million tons per year (Kumar et al. 2005). Acquiring a large land area for the disposal of fly ash is not easy in India, where the land-to-population ratio is very low. Therefore, proper utilization and disposal of fly ash without causing any environmental degradation is a foremost concern for a developing country such as India. Many researchers have attempted to convert this waste into useful construction materials by exploring viable avenues for fly ash management. Among the various uses of fly ash, its bulk utilization is possible only in geotechnical engineering applications such as construction of embankments, and as a sub-base material etc.

Maheboobsab B Nadaf, Research scholar, Indian Institute of Technology Bombay, Civil Engineering Department, Powai, Mumbai 400076, Maharashtra, India. Email id: maheboob_nadaf@iitb.ac.in,
Professor: J. N. Mandal, Indian Institute of Technology Bombay, Civil Engineering Department, Powai, Mumbai 400076, Maharashtra, India. Email id: cejnm@iitb.ac.in,

Again, the decreasing availability of good construction sites and increasing construction activities for infrastructural developments have forced Civil engineers to use unsuitable sites, especially low-lying areas after filling them with fly ash. One of the main characteristics of fly ash is its reactivity. The fly-ash particles are spherical with a high surface area and low loss on ignition. Also, the most soluble phase of fly ash is believed to be amorphous silicate (Smith et al. 1982)

There are two classes of fly ash, Class C and Class F (ASTM C618-12), with properties that reflect minerals present in the type of coal that is burnt. The Class C fly ash, produced in the electric generation process, contains calcium aluminates and calcium silicates, which cause the fly ash to form a cementitious matrix when combined with moisture, making it an excellent binding agent for soil stabilization applications. The burning of harder, older anthracite and bituminous coal typically produces Class F fly ash. This fly ash is pozzolanic in nature, and contains less than 10% lime (CaO). Possessing pozzolanic properties, the glassy silica and alumina of Class F fly ash requires a cementing agent, such as Portland cement, quicklime, or hydrated lime, with the presence of water in order to react and produce cementitious compounds. Alternatively, the addition of a chemical activator such as sodium silicate (water glass) to a Class F ash can lead to the formation of a geopolymer. The use of fly ash in soil stabilization has been practiced in the United States for a vast array of construction projects for over 60 years.
Keeping in view the applications of fly ash in various projects, the physical, chemical and mineralogical properties of fly ash have been investigated and reported in this paper. The fly ash used in the present investigation was collected from Dahanu Thermal Power Station, Thane, Maharashtra, India.

2. Literature Review

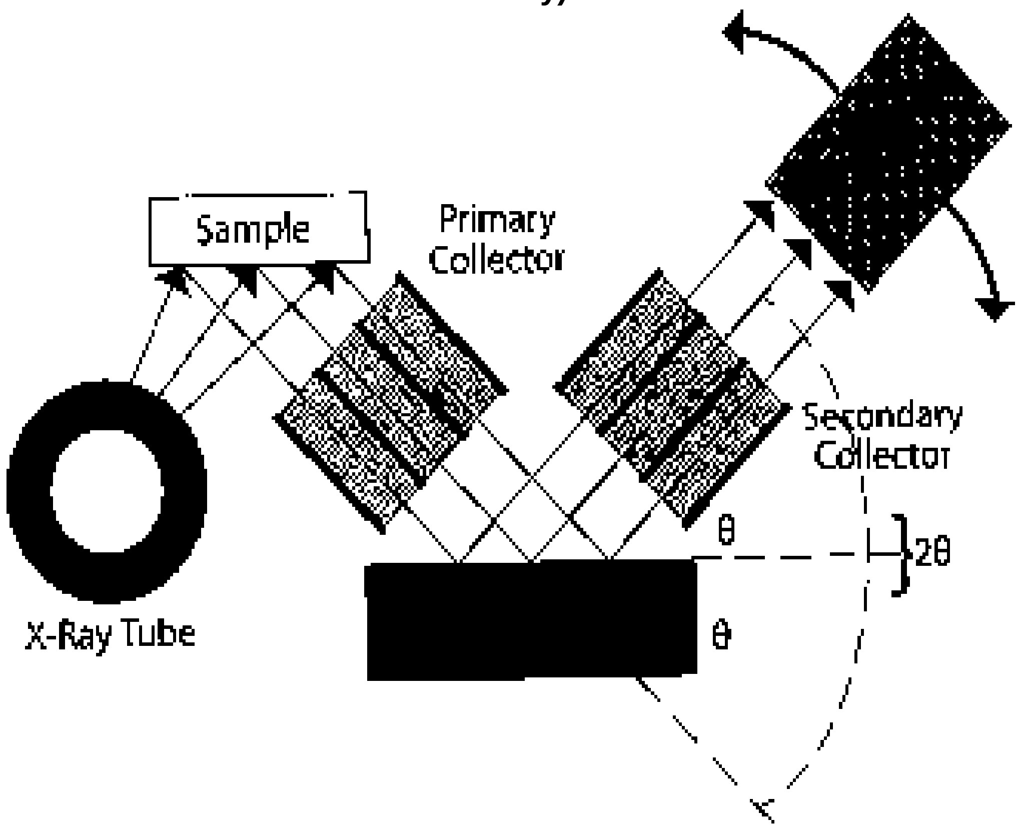
Sridharan et al. (1997) investigated the geotechnical characterization of various ash ponds in India and reported that fly ash has low unit weight, good frictional properties with low compressibility and are also well suited for their applications as a structural fill. Toth et al. (1978) reported similar physical behavior between fly ash and silts. Trivedi & Sud (2002) investigated the engineering properties of coal ash by x-ray diffraction, scanning electron microscope and grain size distribution. Pandian (2004) characterized the coal ash produced in thermal power stations from various parts of India and reported that it is a freely draining material with high angle of internal friction and low specific gravity and can effectively be used in the Geotechnical applications. Its strength can also be improved by adding additives/modifications. Ismail et al. (2007) reported that fly ash consists of inorganic, incombustible matter present in the coal that has been fused during combustion into a glassy, amorphous structure and also contains trace concentrations of the following heavy metals: nickel, vanadium, cadmium, barium, chromium, copper, molybdenum, zinc and lead. Choudhary et al. (2009) conducted experimental study to assess the use fly ash in stabilizing and modifying the undesirable characteristics of fine grained soil and reported that addition of fly ash resulted in the decrease of plasticity, maximum dry density, cohesion and swelling properties of the soil while optimum moisture content and angle of internal friction increased with increasing fly ash content in the mix. Lal & Mandal (2013) conducted laboratory tri-axial tests on unreinforced and cellular reinforced fly ash samples and concluded that inclusion of reinforcements at proper location enhanced the shear strength properties of fly ash. Lal & Mandal (2013) reported that, in india disposal of fly ash is foremost distress and have to be disposed safely to have ecofriendly. Crushed coal is generated from thermal power plants having loose, fine and low unit weight. For the exploitation of fly ash as an substitute backfill material, it is used in the cellular reinforced walls when subjected to uniformly distributed surcharge pressure.

3. Methodology

3.1 X-Ray Diffraction (XRD): X-ray Diffraction technique is based on observing the scattered intensity of an X-ray beam hitting a sample as a function of incident and scattered angle, polarization, and wavelength or energy. The mineralogical composition of the soil was determined with the help of an X-ray Diffraction (XRD) Spectrometer (Phillips 2404, Holland), which employs a graphite monochromator and Cu-Kα radiation. The soil sample was scanned in the range where 2θ value lies from 5° to 80°; θ is the angle of incidence of the reflected X-ray beams. The minerals present in Fly ash are identified with the help of the data files developed by the Joint Committee on Powder Diffraction Standards (JCPDS 1994).

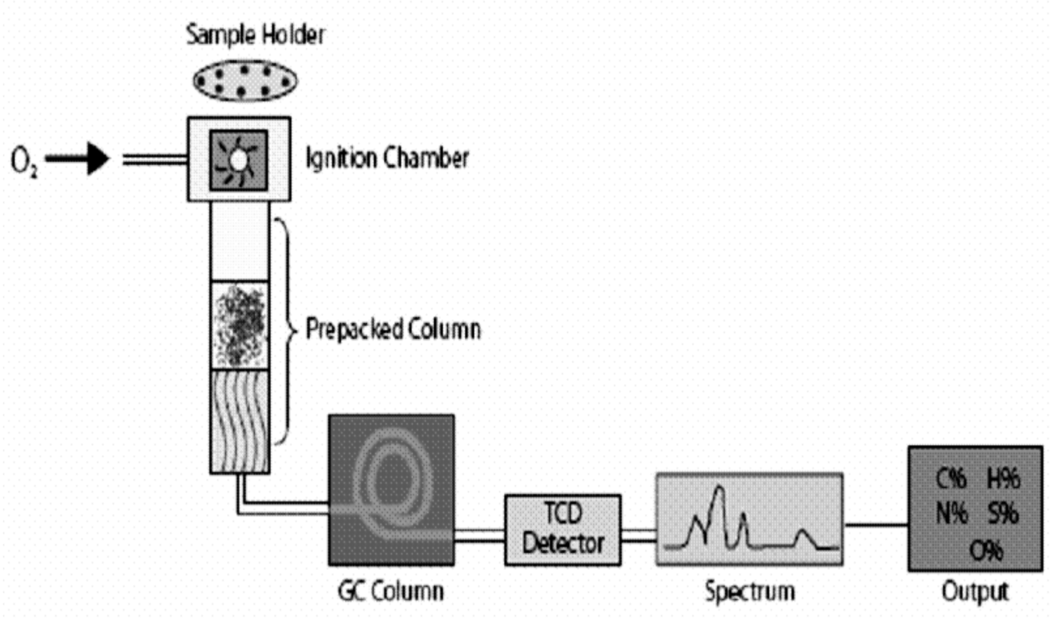
3.2 X-Ray Fluorescence: Chemical composition of the soil sample, in the form of major oxides, was determined using an X-ray Fluorescence setup (Phillips 1410, Holland).
Sample preparation: First 4.0 gm of the fly ash sample was mixed uniformly with 1.0 gm microcrystalline cellulose in a bowl followed by addition of Isopropyl alcohol to the mixture and kept underneath an infrared lamp for slow drying. Two-third of an aluminum dish of inner diameter 33 mm and height 12 mm was filled with a mixture of 70% methyl-cellulose and 30% paraffin wax followed by filling up the remaining portion with the dried sample. Then the dish was placed in a hydraulic compressor and compressed with the help of a hydraulic jack (by applying 15 ton load for 60 sec) to form a pellet, on which the X-Ray Fluorescence test was conducted.
The compressed pellet was mounted in the sample holder of the XRF setup. When an X-ray beam traverses the sample, the crystallites are oriented in such way that they will fulfill the Bragg's condition: $n\lambda=2d\sin\theta$ where, λ=wave length of the exciting X-rays, d = inter planar spacing of atomic planes, θ = angle of incidence, and n= order of diffracted X-ray for reflection from every possible inter planar spacing principle. Figure 1 shows a line sketch to illustrate the principle of X-ray fluorescence test.

Figure 1: Line sketch representing the principle of X-ray fluorescence test (SAIF, IIT Bombay)



3.3 CHNS test: The CHNS(O) Analyzer is of use in determining the percentages of Carbon, Hydrogen, Nitrogen and Sulphur as well as Oxygen in case of organic compounds, based on the principle of "Dumas method" which involves the complete and instantaneous oxidation of the sample by "flash combustion". After combustion, the products are separated by a chromatographic column and detected with the help of a thermal conductivity detector (T.C.D.), which gives an output signal proportional to the concentration of the individual components of the mixture. Schematic diagram of Carbon-Hydrogen-Nitrogen-Sulphur (CHNS) Test is shown in Figure 2.

Figure 2 Schematic diagram of CHNS test (SAIF, IIT Bombay)



3.4 Scanning Electron Microscope (SEM) Test: Scanning Electron Microscope is a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons which interact with the atoms of the sample and produce signals that contain information about the sample's surface topography, composition and other properties such as electrical conductivity. The setup for scanning electron microscope test is shown in Figure 3.

Figure 3 Setup for scanning electron microscope test (SAIF, IIT Bombay)

4. Findings

The fly ash used in the present investigation was collected from Dahanu Thermal Power Station, Thane, India. The study was aimed to find out its physical, chemical and mineralogical properties as well as its surface topography, organic and CHNS content.

4.1 Physical properties:

The physical properties of fly ash have been evaluated in laboratory according to the Indian Standards as mentioned in Table 1. The grain size distribution curve is shown in Figure 4. Standard Proctor test was conducted on fly ash to find out its optimum moisture content and maximum dry density. Figure 5 shows the variation of dry density with change in moisture content.

Table 1 Physical properties of Fly ash

PROPERTIES	STANDARDS	VALUES
Specific gravity	IS: 2720 Part III 1980	2.15
Dry density (g/cm^3)	IS: 2720 Part VII 1980	1.44
Optimum moisture content (OMC) %		18.0
Coefficient of permeability, k (m/sec)	IS: 2720 Part 17	1.11 x10^{-6}
Fine sand size	IS: 2720 Part IV 1977	16%
Silt size		76%
Clay size		8%
Group symbol	Unified soil classification	MS
Group name		Sandy silt
Liquid Limit (%)	IS: 2720 Part V 1987	29.54
Plasticity		NP

Figure 4 Grain size distribution of fly ash

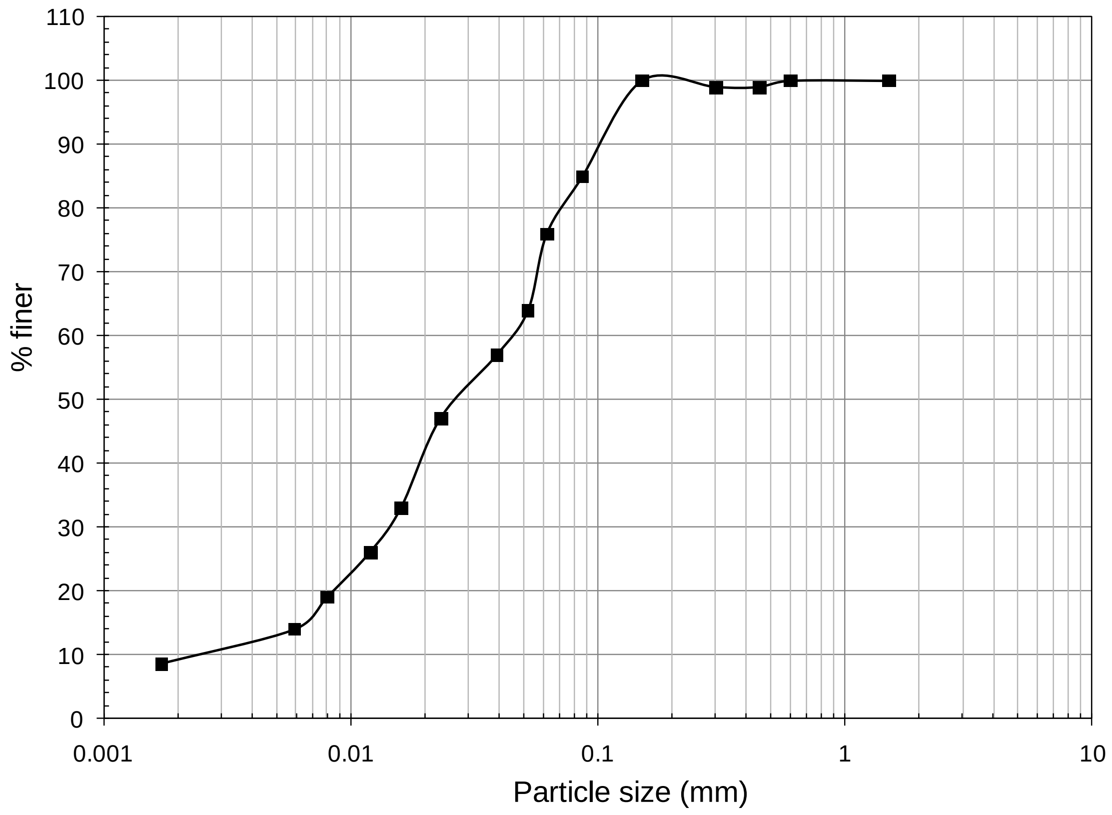
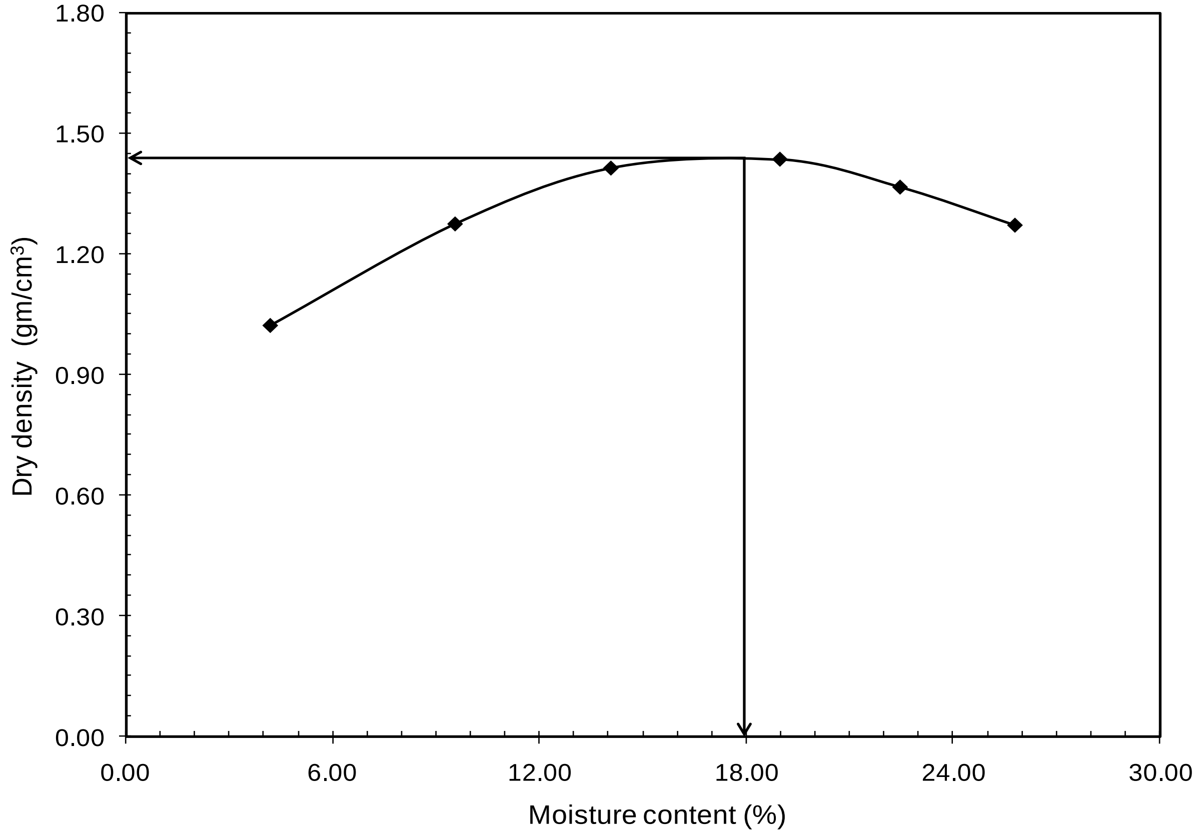


Figure 5 Dry density versus moisture content curve from Standard Proctor Test



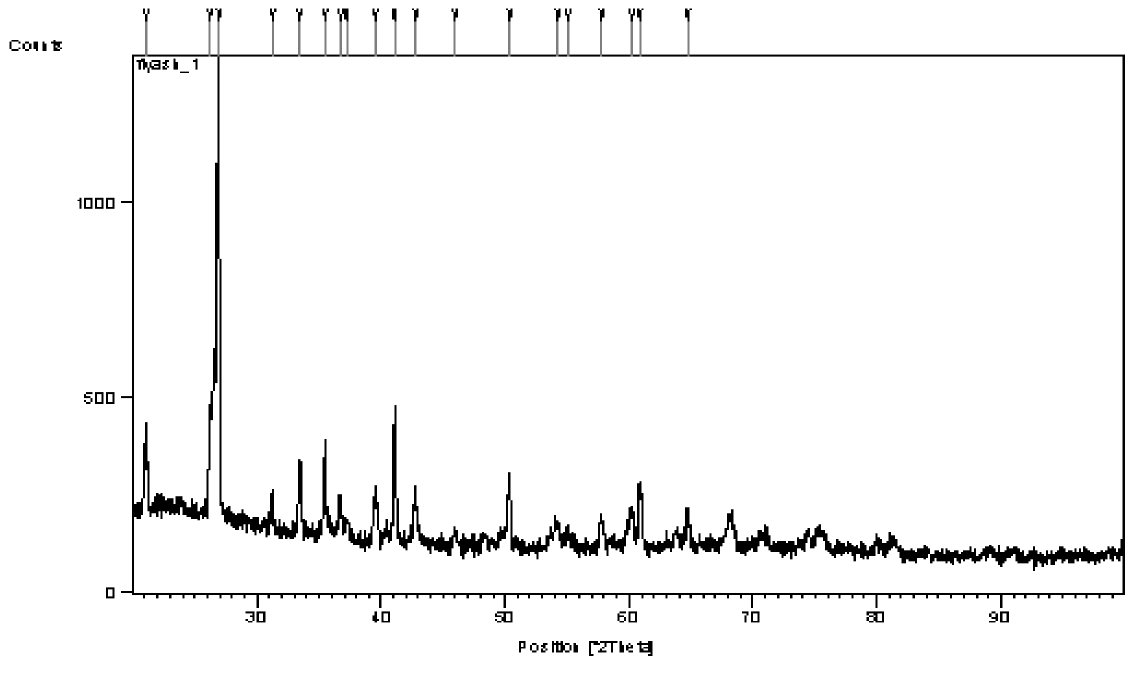
4.2 Mineralogical Composition

X-Ray Diffraction (XRD) test was performed by XRD spectrometer with a scanning range of 5-120º using Cu-Kα radiation to determine the phase compositions of the fly ash. The X-Ray diffraction pattern of fly ash is shown in Figure 6. The minerals present in the fly ash are identified by using database of JCPDS and reported in Table 2.

Table 2 Mineralogical composition of fly ash

Mineral	Value (%)
Quartz	62
Mullite	33
Hematite	4
Sillimanite	1

Figure 6 X-Ray Diffraction pattern of fly ash (MEMS, IIT Bombay)



4.3 Chemical Composition

X-Ray fluorescence (XRF) test was performed on the fly ash sample at Sophisticated Analytical Instrument Facility (SAIF), I.I.T. Bombay to assess its basic chemical constituents. X-Ray fluorescence Spectrometer (make Phillips PW 2404) was used to perform the analysis. Table 3 represents the basic chemical composition of the fly ash. As the CaO content is less than 10%, the fly ash is classified as Class F fly ash (ASTM C618).

Table 3 Chemical composition of fly ash

Chemical constituents	Value (%)
Al_2O_3	26.822
BaO	0.067
CaO	1.128
Fe_2O_3	5.487
K_2O	0.936
MgO	0.955
MnO	0.031
P_2O_5	0.246
SiO_2	61.321
SO_3	0.073
SrO	0.059
TiO_2	1.646
LOI*	1.229
LOI* Loss on ignition	

4.4 Carbon, Hydrogen, Nitrogen, Sulphur (CHNS):

The CHNS(O) analyzer (Make: Thermo Finnigan, Italy; Model: FLASH EA 1112 series) was used to determine the percentages of Carbon (C), Hydrogen (H), Nitrogen (N) and Sulphur (S)present in the fly ash. The test was carried out at Sophisticated Analytical Instrument Facility (SAIF), IIT Bombay. The percentages of CHNS present in the fly ash are reported in the Table 4.

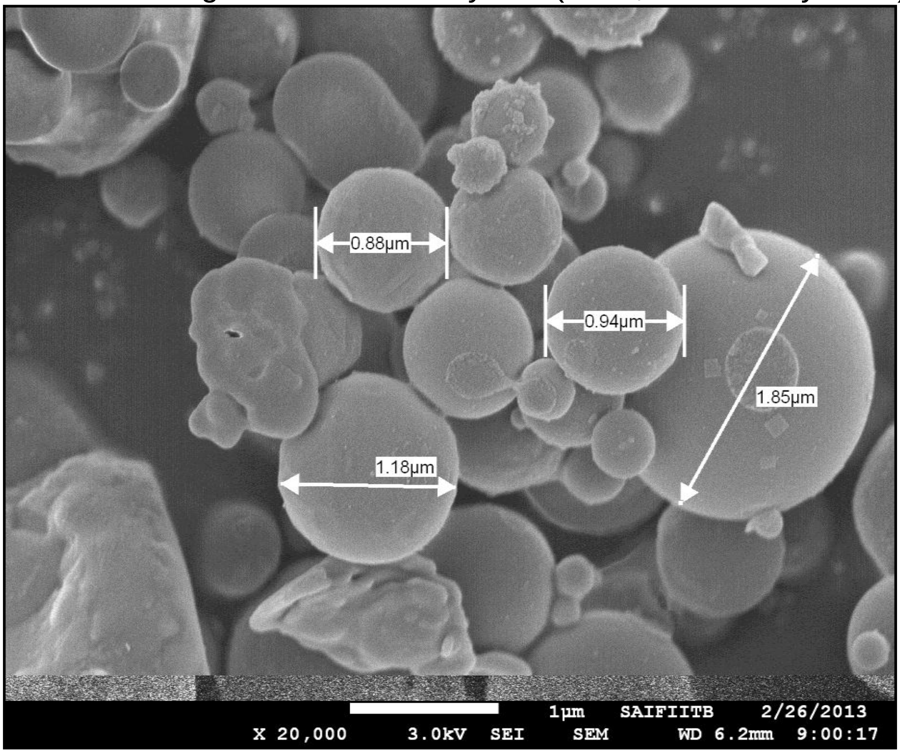
Table 4 Carbon (C), Hydrogen (H), Nitrogen (N) and Sulphur (S) in fly ash

Compound	Value (%)
Carbon	0.00
Hydrogen	0.665
Nitrogen	1.182
Sulphur	0.72

4.5 Scanning Electron Microscope:

Field Emission Gun-Scanning Electron Microscope (SEM) (model JSM-7600F) study was carried out to be familiar with the morphology of the fly ash. The test was also performed at Sophisticated Analytical Instrument Facility (SAIF), IIT Bombay. It was observed that most of the fly ash particles were spherical in shape with surface appeared to be very smooth. Figure 7 shows the scanning electron micrograph of the fly ash particles with two magnification level.

Figure 7 SEM image of the Class F fly ash (SAIF, IIT Bombay 2013)



5. Conclusion

In India, the coals used are generally from the same sources. Therefore, fly ash produced from the burning of the coals in different factories of India will have almost same properties. From the present study, the following conclusions can be drawn.

- Fly ash is silt sized and non-plastic material.
- Major two chemicals in Fly ash are Silica (SiO_2) and Alumina (Al_2O_3). The percentage of silica and alumina are 61.321% and 26.822% respectively. The other chemicals present in the fly ash are less than 6%.
- The fly ash used in the present study is a Class F fly ash as the Calcium Oxide (CaO) content is less than 10%.
- The CHNS test shows that the fly ash is Carbon free.
- SEM confirms that most of the fly ash particles are spherical in shape along with very smooth surface.

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