CHARACTERIZATION OF FLY ASH FOR THEIR EFFECTIVE MANAGEMENT

A THESIS SUBMITTED TO NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA IN
PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF

BACHELOR OF TECHNOLOGY

IN

MINING ENGINEERING

By

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NATIONAL INSTITUTE OF TECHNOLOGY

ROURKELA, ORISSA - 769008

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UNDER THE GUIDANCE OF

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Date:  
Nooka Shashank  
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This is to certify that the thesis on “CHARACTERIZATION OF FLY ASH FOR THEIR EFFECTIVE MANAGEMENT” is submitted by Sri Nooka Shashank & Sri Vaddeti Kalyan Chakravartli to National Institute of Technology, Rourkela under my supervision and is worthy for the partial fulfilment of the degree of Bachelor of Technology (Mining Engineering) of the Institute. He has fulfilled all the prescribed requirements and the thesis, which is based on candidate’s own work, has not been submitted elsewhere.

Date: 

Supervisor

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Department of MINING Engineering
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ABSTRACT

Coal based thermal power plants more or less producing 150 tons of fly ash each year. Endeavours to utilize fly ash powder have come to just a twenty to thirty percent reutilization rate. Around 80 percent of the power generation is from coal based power plants; rest on gas and oil. As most of the power plants are using bituminous coal and sub-bituminous coal leading towards high production of fly ash. Due to high percent of ash in the coal give rise to large volumes of fly ash. Utilization is becoming huge problem in India. The country’s dependence on coal for power generation has unchanged so we need to look at the strategies to encourage and establish technological concepts to utilize fly ash in bulk. We have plenty of uses of fly ash, but we need to analyse the uses such that effective utilization of fly ash takes place. Production of fly ash depends upon the coal source, plant operations and many more factors. The various fly ash characteristics are discussed including classifications, physical characteristics, chemical properties and chemical compositions. In spite of the fact that far reaching examination has been performed on the utilization of fly ash and took environmental concerns also a major problem like mobilization of toxic elements, biota impact, microbial impact, handling dangers, and pertinent regulations. Finally, extensive research has done to reutilization of fly ash and recommendation is provided to cover deficiencies found in the literature.

Key words: Fly ash; pond ash; bottom ash; characterization; utilization; management
CHAPTER -1

INTRODUCTION
1. INTRODUCTION:

The combustion of pulverized coal at high temperatures and pressures in power stations leads to different types of fly ash. Solid residue after combustion includes one is pulverized fuel ash or fly ash and second one is furnace bottom ash. The fine ash small amount is carried upwards with flue gases are captured with electro precipitators. This material is known as fly ash. It is extremely fine, glassy spheres and looks similar to cement. The rest fraction falls into the grates of boilers is known as furnace bottom ash. It has same structure as sand. If these two are mixed together with water and pumped into dumpers to store at ash pond area is known as pond ash. Nearly 70 percent of the solid waste producing from fly ash after coal combustion. Fly ash is primarily producing from coal fired plants rather than volcanoes.

Quality is decided based on grade and rank of the coal. The coal rank arranged in an ascending order of carbon contents is

Peat << Lignite << sub-bituminous coal << bituminous coal << anthracite

There are three categories of coal ashes are

- Dry fly ash
  Gathered from diverse rows of electrostatic precipitators in dry form. The produced fly ash from pulverized coal is fine grained and powdery particulate.

- Bottom ash
  Collected at the bottom of the boiler furnace

- Pond ash
  This is the combination of dry fly ash and bottom ash with water to form slurry which is pumped to the ash pond area. In the ash pond water gets removed and settlement of fly ash takes place.

<table>
<thead>
<tr>
<th>Year</th>
<th>Fly ash generation</th>
<th>Utilization</th>
</tr>
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<tbody>
<tr>
<td>1994</td>
<td>40MT</td>
<td>1MT</td>
</tr>
<tr>
<td>2008-09</td>
<td>160MT</td>
<td>80MT</td>
</tr>
<tr>
<td>2011-12</td>
<td>220MT</td>
<td>110MT</td>
</tr>
<tr>
<td>2031 (rough estimation)</td>
<td>1000MT</td>
<td></td>
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As we can see in 1994, four million tonnes of municipal solid waste and 2 million tons of waste water sludge are produced. Compared to other ashes it creates significantly larger scale disposal problem. Two major concerns for the disposal are without affecting the environment and large area required for disposal. Consequently attempts are being made to use the ash rather than dump it. With the increasing demand of power and coal being the major source of energy, more and more thermal power stations are expected to be increase in their capacities in near future. Fly ash has been considered as a “Pollution Industrial Waste” till about a decade back and was being disposed off in ash ponds. To store expected fly ash we need minimum of 4000 ha of land for the construction of ash ponds. Generally one acre of land is required per megawatt of power generation. Mostly Indian coal is sub bituminous rank followed by bituminous and lignite. The ash content varies from 30 to 50%. In India different collieries consists of different characteristics of coal like calorific value.

Research is going on environmental impacts from fly ash disposal, recycling, and reuse. Europe is reusing forty to fifty percent of their fly ash and Japan is reusing sixty percent whereas the United States has reused only twenty to thirty percent. Although many fly ash reutilization researches have been performed with positive results, most are still perceived as experimental primarily due to the environmental implications. Careful information of the physical characteristics, chemical composition, and properties is essential to understanding any possible environmental impacts. Once the environmental impacts predicted, a larger market can be encouraged to productively reuse fly ash. This project entails the characteristics of fly ash, environmental concerns and existing methods of fly ash.
1.1 Classes of Fly Ash

According to ASTM C-618 Fly ash is classified into two categories: Class F and Class C fly ash. The main difference between these two classes is the amount of calcium, silica, alumina, and iron content. The chemical properties of the fly ash are largely dependent on the chemical content of the coal burned (i.e., anthracite, bituminous, and lignite). By using electrostatic precipitators fly ash is collected. Since the particles solidify while suspended in the exhaust gases, these particles are generally spherical in shape and size around 0.5 μm to 100 μm. They consist mostly of silicon dioxide (SiO2), which is present in two forms: amorphous, which is rounded and smooth, and crystalline, which is sharp, pointed and hazardous; aluminium oxide (Al2O3) and iron oxide (Fe2O3).

Fly ashes are generally highly heterogeneous, consisting of a mixture of glassy particles with various identifiable crystalline phases such as quartz, mullite, and various iron oxides.

Fly ash also contains environmental toxins in varying amounts, including barium (806 ppm); strontium (775 ppm); chromium (136 ppm); arsenic (41.4 ppm); beryllium (5 ppm); boron (311 ppm); cadmium (3.4 ppm); chromium VI (90 ppm); cobalt (35.9 ppm); copper (112 ppm); fluorine (29 ppm); lead (56 ppm); manganese (250 ppm); nickel (77.6 ppm); selenium (7.7 ppm); thallium (9 ppm); vanadium (252 ppm); and zinc (178 ppm).

<table>
<thead>
<tr>
<th>S1.No</th>
<th>Country</th>
<th>Production M</th>
<th>Utilization MT</th>
</tr>
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<tr>
<td>1</td>
<td>India</td>
<td>130</td>
<td>41</td>
</tr>
<tr>
<td>2</td>
<td>China</td>
<td>100</td>
<td>45</td>
</tr>
<tr>
<td>3</td>
<td>USA</td>
<td>75</td>
<td>65</td>
</tr>
<tr>
<td>4</td>
<td>UK</td>
<td>15</td>
<td>50</td>
</tr>
<tr>
<td>5</td>
<td>Germany</td>
<td>40</td>
<td>85</td>
</tr>
<tr>
<td>6</td>
<td>Australia</td>
<td>10</td>
<td>85</td>
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<td>7</td>
<td>Canada</td>
<td>6</td>
<td>75</td>
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<td>8</td>
<td>France</td>
<td>3</td>
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<td>9</td>
<td>Denmark</td>
<td>2</td>
<td>100</td>
</tr>
<tr>
<td>10</td>
<td>Italy</td>
<td>2</td>
<td>100</td>
</tr>
</tbody>
</table>

Table 1.2 Fly ash generation and utilization in different countries
Not all fly ashes meet ASTM C-618 requirements, although depending on the application, this may not be necessary. Ash used as a cement replacement must meet strict construction standards, but no standard environmental standards have been established in the United States. 75% of the ash must have a fineness of 45 μm or less, and have carbon content, measured by the loss on ignition (LOI), of less than 4%. In the U.S., LOI needs to be under 6%. The particle size distribution of raw fly ash is very often fluctuating constantly, due to changing performance of the coal mills and the boiler performance. This makes it necessary that fly ash used in concrete needs to be processed using separation equipment like mechanical air classifiers.

Class ‘F’ fly ash

Class F fly ash is produced from burning of old anthracite and bituminous coal which contains less than 10% lime (CaO). Class F fly ash having pozzolanic properties, the glassy silica and alumina. In order to produce cementious compounds it requires a cementing agent, such as quick lime, Portland cement or hydrated lime with the presence of water. The addition of a chemical activator such as sodium silicate also known as water glass to a Class F ash can lead to the formation of a geopolymer.

Class C Fly ash

Burning of younger lignite or sub bituminous coal produces class C fly ash. It contains more than 20 percent of lime due to the presence of alkali it does not require an activator. The contents of Alkali and sulphate (SO4) are generally higher as compared to the Class F Fly ash.

Fig 1.1 Fly Ash colours of F and C class
CHAPTER -2

LITERATURE REVIEW
2.1 OBJECTIVES

The objective of this study is “characterization of fly ash for their effective management and utilization”. This requires following specific objectives:

Main objective of this report

1. Literature review
   - Fly ash production
   - Utilization of fly ash
   - Disposal methods.

2. Characterization of fly ash on their physical and chemical properties.

3. Effective utilization of fly ash.

Focus is being continued on:

(i) Development of new / incremental technologies
(ii) Adaptation/large scale application of technologies
(iii) Preparation of standards / specifications
(iv) Support & facilitation to other Government Ministries / Department

2.2 GENERATION OF FLY ASH

Fly ash is created by coal-terminated electric and steam creating plants. Normally, coal is pummeled and blown with air into the heater’s ignition chamber where it quickly touches off, producing warmth and creating a liquid mineral deposit. Heater tubes concentrate heat from the evaporator, cooling the pipe gas and creating the liquid mineral builds up to solidify and structure slag. Coarse fiery debris particles, alluded to as base cinder or slag, tumble to the base of the burning chamber, while the lighter fine powder particles, termed fly slag, stay suspended in the pipe gas. Preceding depleting the vent gas, fly fiery debris is evacuated by particulate outflow control gadgets, for example, electrostatic precipitators or channel fabric bag houses.
In the past, fly ash was generally released into the atmosphere, but pollution control equipment mandated in recent decades now requires that it be captured prior to release. In the US, fly ash is generally stored at coal power plants or placed in landfills. About 43 percent is recycled, often used to supplement Portland cement in concrete production. In India the annual production of flyash is nearly 45 million tonnes per year. This is likely to increase to 70 million tons per year by 2010. At present most of the flyash is being dumped. The disposal of the flyash is a serious hazard to the environment that consumes millions of rupees towards the cost of its disposal. About 14000 Hectares of precious land have already been used for dumping it and another thousand of hectares would be required in future.

In India coal/lignite based thermal power stations account for more than 55% of the electricity installed capacity and 65% of electricity generation. The ash content of the coal used at the thermal stations ranges from 30-40%, with the average ash content around 35%. Since low ash, high grade coal is reserved for metallurgical industries; the thermal power plants have to utilize high ash, low grade coal. The thermal power plant ash generation has increased from about 40 million tons during 1993-1994 to 120million tonnes during 2005-06 and is expected to be in the range of 175 million
tonnes per year by 2012, on account of the proposal to double the power generation. Coupled with this, the deteriorating quality (increasing ash quantity) of coal is expected to aggravate the situation.

FIGURE 2.2 Generation of ash at the power plants
2.3 NATURE AND COMPOSITION

Fly ash is a fine, glass powder recovered from the gases of coal fired plants during the production of electricity by electrostatic precipitators. These micron-sized earth elements consist of silica, alumina and iron. When combines with lime and water the fly ash forms a cementitious compound with properties similar to that of Portland cement. There are substantial amount of non-combustible impurities present in coal in the form of limestone, shale, dolomite, feldspar and quartz. As the fuel travels through the high-temperature zone in the furnace, the volatile matter and carbon are burnt off whereas of the mineral impurities are carried off in the form of ash by the flue gas. The ash particles become fused in the combustion zone of the furnace; however on leaving the combustion zone of the furnace the molten ash is cooled rapidly and solidifies as spherical glassy
particles. Some of the fused matter agglomerates to form bottom ash, but most of it flies out with the flue gas stream and is therefore called fly ash.

The fly ash is removed from the flue gas by means of a series of mechanical separators followed by electrostatic precipitators or bag filters. The ratio of fly ash to bottom ash is 72:28 in wet bottom boilers or 84:16 in dry bottom boilers.

Chemical composition of fly ash

<table>
<thead>
<tr>
<th>Component</th>
<th>Bituminous</th>
<th>Sub bituminous</th>
<th>Lignite</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂ (%)</td>
<td>20-60</td>
<td>40-60</td>
<td>15-45</td>
</tr>
<tr>
<td>Al₂O₃ (%)</td>
<td>5-35</td>
<td>20-30</td>
<td>20-25</td>
</tr>
<tr>
<td>Fe₂O₃ (%)</td>
<td>10-40</td>
<td>4-10</td>
<td>4-15</td>
</tr>
<tr>
<td>CaO (%)</td>
<td>1-12</td>
<td>5-30</td>
<td>15-40</td>
</tr>
<tr>
<td>LOI (%)</td>
<td>0-15</td>
<td>0-3</td>
<td>0-5</td>
</tr>
</tbody>
</table>

Table 2.1 chemical composition of fly ash

2.4 Hazards

By virtue of its physical characteristics and sheer volumes generated, fly ash poses problems like:

- It is a very difficult material to handle in dry state because it is very fine and readily airborne even in mild wind.
- It disturbs the ecology of the region, being a source of soil, air and water pollution.
- Long inhalation of fly ash causes silicosis, fibrosis of lungs, bronchitis, pneumonitis etc.
- Flying fine particles of ash poses problems for people living near power stations, corrode structural surfaces and affect horticulture.
- Eventual settlement of fly ash particles over many hectares of land in the vicinity of power station brings about perceptible degeneration in soil characteristics.

2.5 COLLECTION

Ash Collection

1. Dry Fly Ash

Dry fly ash is collected from different rows of electrostatic precipitators. It is available in two different grades of fineness in silos for use as resource material by different users.

2. Bottom Ash

Bottom ash is collected from the bottom of the boiler and transported to hydro bins and then ash mound for use in road embankment.
3. **Conditioned Fly Ash**

Conditioned fly ash is also available in ash mound for use in landfills and ash building products.

### 2.6 TRANSPORTATION

#### Fly Ash Transportation

Fly ash can be supplied in four forms:

- **Dry**: This is currently the most commonly used method of supplying fly ash. Dry fly ash is handled in a similar manner to Portland cement. Storage is in sealed silos with the associated filtration and desiccation equipment, or in bags.

- **Conditioned**: In this method, water is added to the fly ash to facilitate compaction and handling. The amount of water added being determined by the end use of the fly ash. Conditioned fly ash is widely used in aerated concrete blocks, grout and specialist fill applications.

- **Stockpiled**: Conditioned fly ash not sold immediately is stockpiled and used at a later date. The moisture content of stockpiled ash is typically 10 to 15%. This is used mainly in large fill and bulk grouting applications.

- **Lagoon**: Some power stations pump fly ash as slurry to large lagoons. These are drained and when the moisture content of deposited fly ash has reached a safe level may be recovered. Because of the nature of the disposal technique, the moisture content can vary from around 5% to over 30%. Lagoon fly ash can be used in similar applications to stockpiled conditioned fly ash.

### 2.7 FEATURES

- **Spherical shape**: These are spherical in shape and allowing them to flow and combines easily with mixtures.

- **Reduced Sulphate Attack**: Fly ash mixes up free lime that can combine with sulphate can lead to create destructive expansion.

- **Reduced Efflorescence**: Fly ash chemically combines free lime and salts that can create efflorescence and dense concrete holds efflorescence producing compounds on the inside.

- **Ball bearing effect**: Lubricating action of fly ash particles is mainly due to ball bearing effect when concrete in plastic state.
- **Reduced Bleeding**: Fewer bleed channels decreases porosity and chemical attack. Bleed streaking is reduced for architectural finishes. Improved paste to aggregate contact results in enhanced bond strengths.

- **Reduced Segregation**: Improved cohesiveness of fly ash concrete reduces segregation that can lead to rock pockets and blemishes.

- **Reduced Slump Loss**: More dependable concrete allows for greater working time, especially in hot weather.

- **Higher Strength**: Fly ash continues to blend with free lime that increases the structural strength.

- **Decreased Permeability**: Increased density and long term pozzolanic action of fly ash, which ties up free lime, results in fewer bleed channels and decreases permeability.

- **Increased Durability**: Dense fly ash concrete helps keep aggressive compounds on the surface, where destructive action is lessened. Fly ash concrete is also more resistant to attack by sulphate, mild acid, soft (lime hungry) water, and seawater.

- **Reduced Shrinkage**: Because of lubricating action of fly ash reduces water content and drying shrinkage.

- **Reduced Heat of Hydration**: The reaction between fly ash and lime generates less heat and reduced thermal cracking. That is the main reason to replace Portland cement with fly ash.

- **Reduced Alkali Silica Reactivity**: Fly ash combines with alkalis from cement that might otherwise combine with silica from aggregates, causing destructive expansion.

- **Workability**: Concrete is easier to place with less effort, responding better to vibration to fill forms more completely.

- **Ease of Pumping**: Pumping requires less energy and longer pumping distances are possible.

- **Improved Finishing**: Sharp, clear architectural definition is easier to achieve, with less worry about in-place integrity.

### 2.8 UTILIZATION

The reuse of fly ash as an engineering material primarily stems from its pozzolanic nature, spherical shape, and relative uniformity. Fly ash recycling, in descending frequency, includes usage in:

1. Portland cement
2. Embankments
3. Waste stabilization & solidification
4. Raw feed for cement clinkers
5. Mine reclamation
6. Stabilization of soft soils
7. Road sub base
8. Aggregate
9. Flow able fill
10. Mineral filler in asphaltic concrete
11. Structural fill

Other applications include cellular concrete, geopolymers, roofing tiles, paints, metal castings, and filler in wood and plastic products.

Figure 2.4 Underground mine backfilling

2.9 CHALLENGES IN HANDLING FLY ASH
Many challenges have been reported in the handling and utilization of fly ash. Some of these difficulties include:

- The wet system of fly ash collection/disposal is the most common practice in India. Fly ash is mixed with bottom ash in slurry form before transporting it to ash ponds/lagoons. This process of fly ash dumping is largely unsuitable for all purposes where pozzolonic
properties are essential to its use. Fineness and lime reactivity are seriously imparted and the ash from the ponds is unsuitable for use in most applications needing strength.

- Variations in ash composition are unavoidable and it largely depends on the quality of coal utilizes. Customer therefore can never be sure of the quality of ash available from a particular source.
- There is no system of testing, labelling and packing of coal ash. Most of the ash producers are not equipped to certify the quality or specifications of an ash. This undermines the confidence of the end users of the fly ash from a particular source compelling them to set up such testing and other facilities at their own cost. This obviously makes them somewhat reluctant to use it.
- Most thermal stations are located in remote areas and the user industry faces difficulty in lifting and transporting the fly ash.

2.10 PHYSICAL CHARACTERISTICS

2.10.1 Specific gravity

Specific gravity is one of the important physical properties needed for the use of coal ashes for geotechnical and other applications. In general, the specific gravity of coal ashes varies around 2.0 but can vary to a large extent (1.6 to 3.1). Because of the generally low value for the specific gravity of coal ash compared to soils, ash fills tend to result in low dry densities. The reduction in unit weight is of advantage in the case of its use as a backfill material for retaining walls since the pressure exerted on the retaining structure as well as the foundation structure will be less. The other application areas include embankments especially on weak foundation soils, reclamation of low-lying areas, etc. The variation of specific gravity of the coal ash is the result of a combination of many factors such as gradation, particle shape and chemical composition. It is known that coal ash comprises mostly glassy cenospheres and some solid spheres. The reason for a low specific gravity could either be due to the presence of large number of hollow cenospheres from which the entrapped air cannot be removed, or the variation in the chemical composition, in particular iron content, or both. The investigations show that the specific gravity generally lies between 1.46 and 2.66. In most of the cases, fly ash will have higher specific gravity compared to pond and bottom ashes of the same locality. When the particles are crushed, they show a higher specific gravity compared to the uncrushed portion of the same material.
2.10.2 Morphology

The morphology of a fly ash particle is defined as the form or structure of the particle. Knowing the particle morphology can be helpful to understanding the physical properties and leaching behaviour of the fly ash. Fisher defined eleven major morphological classes with the use of light microscopy. The classes based were based on opacity, shape and types of inclusions. The classes are as follows.

- Amorphous, non-opaque
- Amorphous, opaque
- Amorphous, mixed non-opaque and opaque
- Rounded, vesicular, non-opaque
- Rounded, vesicular, mixed non-opaque and opaque
- Angular, lacy, opaque
- Cenosphere (hollow sphere), non-opaque
- Plerosphere (sphere packed with other spheres), non-opaque
- Solid sphere, non-opaque
- Sphere, opaque
- Sphere with either surface or internal crystals, non-opaque.

2.10.3 Surface area

The surface area of particles is important because it may control the total adsorption capacity but not necessarily the desorption rate. The surface areas of fly ash particles generally vary inversely with the particle size. The smaller the particle size the larger the surface area. After a series of tests determining the surface area and porosity of western coal fly ash, they found, for the 0 - 75 gm particle size range, the surface area did in fact decrease with increasing particle size. However, for particles over 75 gm the shape deviates from the spherical shape and forms irregular sponge like shapes which in turn increases the surface area. The larger particle sizes are usually carbonaceous which indicates they are likely the result of incomplete combustion.

2.10.4 Permeability/Hydraulic Conductivity

Permeability and Hydraulic Conductivity (K) are terms that are often used interchangeably for the same property. It appears hydrologists and physicists use the term hydraulic conductivity
whereas geotechnical engineers use coefficient of permeability. Both terms will be used as referenced in the literature. However, coefficient of permeability (L/T) should not be confused with intrinsic permeability (L²). The coefficient of permeability or hydraulic conductivity (K) takes into account the fluid properties and the medium properties, whereas intrinsic permeability (k) only refers to the effectiveness of the porous medium alone (Gupta, 1989). They are related by the following equation.

\[ K = k \times \text{specific weight of fluid/dynamic viscosity of fluid} \]

Permeability (K) is defined as a measure of the amount of fluid that will flow through a sample for a given time without causing displacement. It should be noted that the fluid is actually flowing through the void spaces, not the particulate matter. Therefore, porosity can have a controlling influence on permeability. Porosity is a value that portrays the amount of voids in a sample which is representative of the water bearing capacity. Porosity is usually represented by n. It is determined by calculating the ratio of the volume of the voids to the total volume (multiply by 100 to express as percent). Obviously the porosity and therefore permeability can be affected by compaction (density) since this reduces the amount of void space for a given total volume. Normally the higher porosity samples will have a higher hydraulic conductivity but it is not necessarily true for all types of soils. Fly ash compacted in a laboratory to 95 percent maximum density can achieve a permeability of \(1 \times 10^{-5}\) cm/sec. A higher density results in a lower permeability. This is beneficial since a low permeability will restrict leachate from migrating away from the site. Similar results were found in other studies: a compacted fly ash generally has a low hydraulic conductivity at 9cm/day (1 x 10⁴ cm/sec), whereas un-compact fly ash can be as high as 70cm/day (8.1 x 1 0 4 cm/sec). A study of compacted fly ash samples revealed permeability’s ranging from 1.2 x 10⁻⁴ to 1.8 x 10⁻⁵ cm/sec. They also cited a previous study of forty-one samples with a mean saturated permeability of 1.3 x10⁻⁵ cm/sec with a standard deviation equal to a half order of magnitude.

- Bituminous 1x 10⁻⁴ to 1 x 10⁻⁷ cm/sec
- Subbituminous 1 x 10⁻⁵ to 3 x 10⁻⁶ cm/sec
- Lignite 9 x 10⁻⁶ to 1 x 10⁻⁷ cm/sec

When fly ash was used to amend soil for plant growth, the hydraulic conductivity in the soil increased until the fly ash accounted for 10 to 20 percent by volume. Once this point was reached, the hydraulic conductivity decreased. This seems to be a result of the fly ash's pozzolanic reaction which tends to cement when in contact with water. A smaller amount of fly ash is needed in acidic soils to reach this turning point. The low hydraulic conductivity can be very desirable if the fly ash is to be re-used. Even in a disposal situation, low hydraulic conductivity would discourage water from seeping through and forming leachate, especially if a higher hydraulic conductivity layer surrounds the fly ash (path of lower resistance).
2.10.5 Density

Density is defined as mass per unit volume. Again, density is also affected by compaction. In a laboratory test of eastern bituminous coal fly ash, a 95 percent maximum density of 1.3 g/cm$^3$ was achieved. The disposed state was 85 percent maximum density with a 1.1 g/cm$^3$ density. Since fly ash generally has a low bulk density, fly ash addition to soil reduces the bulk density of soil.

2.10.6 Grain size distribution

Grain size distribution indicates if a material is well graded, poorly graded, fine or coarse, etc. and also helps in classifying the coal ashes. Coal ashes are predominantly silt sized with some sand-size fraction. Leonards and Bailey have reported the range of gradation for fly ash and bottom ashes which can be classified as silty sands or sandy silts. The extensive investigation carried out on Indian coal ashes demonstrates that the fly ashes consist predominantly of silt-size fraction with some clay-size fraction. The pond ashes consist of silt-size fraction with some sand-size fraction. The bottom ashes are coarser particles consisting predominantly of sand-size fraction with some silt-size fraction. Based on the grain-size distribution, the coal ashes can be classified as sandy silt to silty sand. They are poorly graded with coefficient of curvature ranging between 0.61 and 3.70. The coefficient of uniformity is in the range of 1.59–14.0.

2.10.7 Free swell index

Free swell index in soil engineering serves as a tool to identify swelling soils. The free swell test method proposed by Holtz and Gibbs to estimate the swell potential suffers from certain limitations. Sridhar modified the definition of free swell index itself to take care of the limitations. There is hardly any information on the free swell index of coal ashes in published literature. Hence, experiments were carried out at IISc to study the free swell index of coal ashes. The results indicate that 70% of the coal ashes show negative free swell index which is due to flocculation. Since the clay-size fraction in coal ashes is very less, the free swell index is negligible.
2.10.10 Index properties

Index properties are extensively used in geotechnical engineering practice. Among them, liquid limit is an important physical property for use in classification and for correlations with engineering properties. While a number of studies have been made on the liquid limit of fine-grained soils not much work has been done on coal ashes. Currently, two methods (Percussion cup and fall cone methods) are popular for the determination of liquid limit of fine-grained soils. In the Percussion cup method it is very difficult to cut a groove in soils of low plasticity and the soils have tendency to slip rather than flow. Hence, this method is not suitable for fly ashes which are non-plastic in nature. Even in the cone penetration method, there is a tendency for the fly ash in the cup to liquify at the surface. There is also a variation of water content in the cup with depth and it is very difficult to get a smooth level surface of the ash in the cup.

A new method of determining liquid limit called “Equilibrium water content under Ko stress method” has been found to be effective for the determination of liquid limit of coal ashes. The proposed method is simple, reasonably error free, less time consuming and has good reproducibility. However, it is not suitable for class C fly ashes which gain strength with time. The results obtained using the proposed method show that fly ashes have liquid limit water content ranging from 26 to 51%, 22 to 64% for pond ashes, and 45 to 104% for bottom ashes. The liquid limit values exhibited by coal ashes are not due to their plasticity characteristics but are due to their fabric and carbon content. All the coal ashes tested are non-plastic and hence plastic limit could not be determined. It was also not possible to carry out shrinkage limit tests since the ash pats crumbled upon drying. Since the amount of shrinkage is very less, the shrinkage limit will be quite high. Hence shrinkage will not be a constraint.

2.10.11 Specific surface

The study of specific surface of soils is widely recognized as a means to understand their physical and engineering behaviour. Even though coal ashes are primarily silt/sand-sized particles and their specific surface is expected to be very low, results need be obtained for completeness and for use in certain cases. With this in mind, the surface area measurements were made using Desiccator method and Blaines Air Permeability method.
2.10.12 Classification

For an effective and efficient use of coal ashes in geotechnical engineering practice, their classification from geotechnical engineering point of view is important. While a number of studies have been made on the physical and engineering properties of coal ashes and their utilization in geotechnical engineering practice, no information is available with respect to their classification.

<table>
<thead>
<tr>
<th>Component</th>
<th>Bituminous</th>
<th>Sub-bituminous</th>
<th>Lignite</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO$_2$</td>
<td>20 – 60</td>
<td>40 – 60</td>
<td>15 – 45</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>5 – 35</td>
<td>20 – 30</td>
<td>10 – 25</td>
</tr>
<tr>
<td>Fe$_2$O$_3$</td>
<td>10 – 40</td>
<td>4 – 10</td>
<td>4 – 15</td>
</tr>
<tr>
<td>CaO</td>
<td>1 – 12</td>
<td>5 – 30</td>
<td>15 – 40</td>
</tr>
<tr>
<td>MgO</td>
<td>0 – 5</td>
<td>1 – 6</td>
<td>3 – 10</td>
</tr>
<tr>
<td>SO$_3$</td>
<td>0 – 4</td>
<td>0 – 2</td>
<td>0 – 10</td>
</tr>
<tr>
<td>Na$_2$O</td>
<td>0 – 4</td>
<td>0 – 2</td>
<td>0 – 6</td>
</tr>
<tr>
<td>K$_2$O</td>
<td>0 – 3</td>
<td>0 – 4</td>
<td>0 – 4</td>
</tr>
<tr>
<td>LOI</td>
<td>0 – 15</td>
<td>0 – 3</td>
<td>0 – 5</td>
</tr>
</tbody>
</table>

Table 2.2 Range of chemical composition of Indian Coal Ashes.

2.11 Chemical properties

The chemical properties of the coal ashes greatly influence the environmental impacts that may arise out of their use/disposal as well as their engineering properties. The adverse impacts include contamination of surface and subsurface water with toxic heavy metals present in the coal ashes, loss of soil fertility around the plant sites, etc. Hence this calls for a detailed study of their chemical composition, morphological studies, pH, total soluble solids, etc.

2.11.1 Chemical composition

Chemical composition suggests the possible applications for coal ash. The investigations carried out on Indian fly ashes show that all the fly ashes contain silica, alumina, iron oxide and calcium oxide. The silica content in fly ashes is between 38 and 63%, 37 and 75% in pond ashes,
and 27 and 73% in bottom ashes. The alumina content ranges between 27 and 44% for fly ashes, 11 and 53% for pond ashes and 13 and 27% for bottom ashes. The calcium oxide is in the range of 0 to 8% for fly ashes, 0.2 to 0.6% for pond ashes and 0 to 0.8% for bottom ashes. It is found that all the Indian coal ashes satisfy the chemical requirements for use as a pozzolonic. According to ASTM classification, only Neyveli fly ash can be classified as Class C fly ash and all other coal ashes fall under Class F.

2.11.2 X-ray diffraction

X-ray diffraction studies are carried out primarily to identify the mineral phases. The studies carried out indicate that coal ashes predominantly consist of quartz and feldspar minerals. The studies carried out at IISc reveal that the major mineral found in coal ashes is quartz with lesser proportions of feldspars, carbonates and chlorites. The coal ashes exhibit both crystalline and amorphous phases.

Table 2.3 Minerals present in different coal ashes

<table>
<thead>
<tr>
<th>Compounds</th>
<th>Fly ash</th>
<th>Pond ash</th>
<th>Bottom ash</th>
<th>Soils</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>38-63</td>
<td>38-75</td>
<td>22-24</td>
<td>43-62</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>27-44</td>
<td>11.7-54</td>
<td>13-26.7</td>
<td>12-39</td>
</tr>
<tr>
<td>TiO₂</td>
<td>0.4-1.9</td>
<td>0.2-1.4</td>
<td>0.2-1.8</td>
<td>0.2-2</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>3.3-6.4</td>
<td>3.5-34.6</td>
<td>4-10.9</td>
<td>1-14</td>
</tr>
<tr>
<td>Mgo</td>
<td>0.01-0.5</td>
<td>0.1-0.8</td>
<td>0.1-0.7</td>
<td>0.2-3.0</td>
</tr>
<tr>
<td>MnO</td>
<td>0-0.5</td>
<td>bd-0.6</td>
<td>bd-0.3</td>
<td>0-0.1</td>
</tr>
<tr>
<td>CaO</td>
<td>0.2-8</td>
<td>0.2-0.6</td>
<td>0.1-0.8</td>
<td>0-7</td>
</tr>
<tr>
<td>Na₂O</td>
<td>0.07-0.43</td>
<td>0.05-0.31</td>
<td>bd-0.3</td>
<td>0.2-3</td>
</tr>
</tbody>
</table>

2.11.3 pH

In general, fly ash can be classified as an amorphous ferro-alumino silicate mineral. The amorphous iron aluminium oxides as well as manganese oxides present on the surface of fly ash particles act as a sink, adsorbing the trace elements. It is this quantity of trace element which is available for leaching. The degree of solubility of these oxide sinks determines the release of the elements associated with them into the aqueous medium. The pH of the aqueous medium affects the solubility of these oxides and hence their physico-chemical characteristics. Further, the mobilization of trace elements in aqueous medium is often regulated by the solubility of hydroxide and carbonate salts which also depends on the pH of the aqueous media. The investigations carried on Indian coal
ashes at IISc show that fly ash has higher pH values compared to pond and bottom ashes. The fly ash with higher free lime and alkaline oxides exhibits higher pH values. Since all the coal ashes tested are nearly alkaline, they can be used in reinforced cement concrete which will be safe against corrosion.

2.11.4 Total soluble solids

The presence of soluble solids is an important aspect requiring examination since the water soluble solids greatly influence the engineering properties. Further, the solubility of nutrient elements such as calcium, magnesium, iron, sulphur, phosphorus, potassium and manganese affect the crop yield to a great extent. The present investigations showed that the soluble solids range between 400 and 17600 ppm for fly ashes, 800 and 3600 ppm for pond ashes, and 1400 and 4100 ppm for bottom ashes.

2.11.5 Lime reactivity

The strength of fly ash generally improves with time due to pozzolanic reactions. Reactive silica and free lime contents are necessary for pozzolanic reactions to take place. Lime reactivity is a property which depends on the proportion of reactive silica in coal ash. Based on the work at IISc, the lime reactivity is found to be high for coal ashes with high silica content. It was also found to be high for fly ash compared to bottom and pond ashes. The results indicate that a high percentage of free lime in coal ash plays an important role in increasing its lime reactivity.

2.11.6 Compaction behaviour

The density of coal ashes is an important parameter since it controls the strength, compressibility and permeability. Densification of ash improves the engineering properties. The compacted unit weight of the material depends on the amount and method of energy application, grain size distribution, plasticity characteristics and moisture content at compaction. The variation of dry density with moisture content for fly ashes is less compared to that for a well-graded soil, both having the same median grain size. The tendency for fly ash to be less sensitive to variation in moisture content than for soils could be explained by the higher air void content of fly ash. Soils normally have air void content ranging between 1 and 5% at maximum dry density, whereas fly ash contains 5 to 15%. The higher void content could tend to limit the build-up of pore pressures during compaction, thus allowing the fly ash to be compacted over a larger range of water content.
2.11.7 **Strength behaviour**

An important engineering property that is necessary for using fly ash in many geotechnical applications is its strength. The compressive strength characteristics of fine ash are higher than those of coarser ash specimen. Also most of the shear strength of the fly ash specimen is due to internal friction. Effects of additives – various research works carried out on the fly ashes in different part of the country have shown that the strength increases with the addition of gypsum and lime. In few cases addition of gypsum alone has no effects on the strength characteristics of fly ash.

2.11.8 **Permeability behaviour**

Permeability is an important parameter in the design of liners to contain leachate migration, dykes to predict the loss of water as well as the stability of slopes and as a sub-base material. The coefficient of permeability of ash depends upon the grain size, degree of compaction and pozzolanic activity. The permeability of fly ashes is in the range of $8 \times 10^{-6}$ cm/s to $1.87 \times 10^{-4}$ cm/s, $5 \times 10^{-5}$ cm/s to $9.62 \times 10^{-4}$ cm/s for pond ashes, and $9.9 \times 10^{-5}$ cm/s to $7 \times 10^{-4}$ cm/s for bottom ashes.

2.11.9 **Leaching behaviour**

If water contacts or passes through a porous media, each constituent present in the matrix dissolves into pore water at some finite rate because there is no such thing as a completely insoluble material (Conner 1990). Permeation of the contaminated pore water out of the porous matrix due to any driving force is called “leaching.” The contaminated water that is generated as water passes through a porous matrix is called “leachate.” The capacity of the waste material to leach is called its “leachability.” Depending on the sources of coals used in thermal power plants, fly ash may contain various toxic elements. Due to serious environmental problems involved, the leaching of these toxic elements from ash ponds is gaining considerable importance. The review of literature reveals that the release of contaminants from fly ash and their subsequent influence on ground-water quality is governed by several factors including quality of coal, sources of water, pH, time, temperature, etc. The leachate characteristics are highly variable and even within a given landfill site, leachate quality varies over time and space.

2.12 **Disposal and market sources of fly ash**
Cellular Light Weight Concrete (CLC) Blocks

These are substitute to bricks and conventional concrete blocks in building with density varying from 800 kg/m$^3$ to 1800 kg/m$^3$. The normal constituents of this Foaming Agent based technology from Germany are cement, Fly Ash (to the extent 1/4th to 1/3rd of total materials constituent), sand, water and foam (generated from biodegradable foaming agent). Using CLC walling & roofing panels can also be manufacture. Foaming agent and the Foam generator, if used for production of CLC with over 25% fly ash content invites concession on import duty by Govt. of India

Development of Fly Ash Based Polymer Composites as Wood Substitute

Fly ash based composites have been developed using fly ash as filler and jute cloth as reinforcement. After treatment, the jute cloth is passed into the matrix for lamination. The laminates are cured at specific temperature and pressure. Number of laminates is used for required thickness. The technology on fly ash Polymer Composite using Jute cloth as reinforcement for wood substitute material can be applied in many applications like door shutters, partition panels, flooring tiles, wall panelling, ceiling, etc. With regard to wood substitute products, it may be noted that the developed components / materials are stronger, more durable, resistant to corrosion and above all cost effective as compared to the conventional material i.e. wood. One commercial plant has also been set up based on this technology near Chennai.

![Figure 2.4 fly ash as wood substitute](image)

Portland Pozzolana Cement
Up to 35% of suitable fly ash can directly be substituted for cement as blending material. Addition of fly ash significantly improves the quality & durability characteristics of resulting concrete. In India, present cement production per annum is comparable to the production of Fly Ash. Hence even without enhancing the production capacity of cement; availability of the cement (fly ash based PPC) can be significantly increased.

**Ready mixed Fly Ash concrete**

Though Ready Mix concrete is quite popular in developed countries but in India it consumes less than 5 percent of total cement consumption. Only recently its application has started growing at a fast rate. On an average 20% Fly ash (of cementitious material) in the country is being used which can easily go very high. In ready mix concrete various ingredients and quality parameters are strictly maintained/controlled which is not possible in the concrete produced at site and hence it can accommodate still higher quantity of fly ash

![Figure 2.5 road mixed fly ash concrete](image)

**Fly Ash- Sand-Lime-(Gypsum /Cement) Bricks /Blocks**

Fly Ash can be used in the range of 40-70%. The other ingredients are lime, gypsum /cement, sand, stone dust/chips etc. Minimum compressive strength (28 days) of 70 kg/cm2 can easily be achieved and this can go up to 250 Kg/cm2 (in autoclaved type)
Fly Ash in Road Construction

Fly ash can be used for construction of road and embankment. This utilization has many advantages over conventional methods. Saves top soil which otherwise is conventionally used, avoids creation of low lying areas (by excavation of soil to be used for construction of embankments). Avoids recurring expenditure on excavation of soil from one place for construction and filling up of low lying areas thus created. Fly Ash may be used in road construction for:

i) Stabilizing and constructing sub-base or base.

ii) Upper layers of pavements.

iii) Filling purposes. Concrete with Fly Ash (10-20% by wt) is cost effective and improves performance of rigid pavement.

iv) Soil mixed with Fly Ash and lime increases California Bearing Ratio (CBR), increased (84.6%) on addition of only Fly Ash to soil. Addition of Fly Ash has not shown any adverse effects on the ground water quality in the vicinity of experimental plots. National Highway Authority of India (NHAI) is currently using 60 lakh m$^3$ of Fly Ash and proposed to use another 67 lakh m$^3$ in future projects.

Fig 2.6 fly ash bricks

Fig 2.7 fly ash in road construction
Roller compacted concrete
Another application of using fly ash is in roller compacted concrete dams. Many dams in the US have been constructed with high fly ash contents. Fly ash lowers the heat of hydration allowing thicker placements to occur. Data for these can be found at the US Bureau of Reclamation. This has also been demonstrated in the Ghatghar Dam Project in India

Embankment
Fly ash properties are somewhat unique as an engineering material. Unlike typical soils used for embankment construction, fly ash has a large uniformity coefficient consisting of clay sized particles. Engineering properties that will affect fly ash use in embankments include grain size distribution, compaction characteristics, shear strength, compressibility, permeability, and frost susceptibility. Nearly all fly ash used in embankments are Class F fly ashes.

Asphalt concrete
Asphalt concrete is a composite material consisting of an asphalt binder and mineral aggregate. Both Class F and Class C fly ash can typically be used as a mineral filler to fill the voids and provide contact points between larger aggregate particles in asphalt concrete mixes. This application is used in conjunction or as a replacement for, other binders (such as Portland cement or hydrated lime). For use in asphalt pavement, the fly ash must meet mineral filler specifications outlined in ASTM D242. The hydrophobic nature of fly ash gives pavements better resistance to stripping. Fly ash has also been shown to increase the stiffness of the asphalt matrix, improving rutting resistance and increasing mix durability
CHAPTER-3
EXPERIMENTAL INVESTIGATION
3 EXPERIMENTAL INVESTIGATIONS

3.1 Moisture content

Coal due its tendency, origin and event is constantly connected with same measure of dampness, which is both physically and chemically bound. It is standard to separate in the middle of outside and inalienable dampness. At the point when a wet fly ash remains is presented to air, the outer climate vanishes, yet the obviously dry fly ash still contains some dampness, which can be uprooted just on warming over 100°C. Outside dampness is additionally called inadvertent or free dampness while intrinsic dampness is termed as harmony or air dried or hygroscopic dampness. The amount of outside dampness depends primarily on the method of event and treatment of fly ash, yet the air-dried dampness is identified with the inalienable hygroscopic nature of the fly ash.

Test procedure

Around 1 gm of finely powdered (-212 micron) air-dried fly ash example is said something a silica crucible and after that set inside an electric hot air broiler, kept up at 108°C±2°C. The crucible with the fly ash test is permitted to stay in the stove for 1.5 hours and is then brought out with a couple of tongs, cooled in desiccators for around 15 minutes and after that weighed. The misfortune in weight is accounted for as dampness (on % premise). At that point figuring is done according to the accompanying.

\[
\text{% moisture} = \frac{Y-Z}{Y-X}
\]

Where X= weight of empty crucible, gram 
Y= weight of crucible + fly ash sample before heating, gram 
Z= weight of crucible + fly ash sample after heating, gram 
Y–X= weight of fly ash sample, gram 
Y–Z= weight of moisture.

MOISTURE CONTENT OF FLY ASH

<table>
<thead>
<tr>
<th>Weight of empty crucible (gm)</th>
<th>Weight of fly ash (gm)</th>
<th>Weight of crucible and fly ash before heating (gm)</th>
<th>Weight of crucible and fly ash after heating (gm)</th>
<th>Moisture content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>36.376</td>
<td>1.014</td>
<td>37.390</td>
<td>37.239</td>
<td>0.148</td>
</tr>
<tr>
<td>36.420</td>
<td>1.026</td>
<td>37.446</td>
<td>37.279</td>
<td>0.162</td>
</tr>
</tbody>
</table>

Table 3.1 Moisture content of fly ash
3.2 TRUE DENSITY

True density of fly ash is the weight every unit volume of finely powdered example. Thusly, the volume of pores spaces and the interspaces is excluded here. To focus the genuine true density, fly ash test is scattered in water. The measure of water scattered every gram of fly ash gives the true density of fly ash. The thickness of the fly ash, which goes from 2-2.8, decides the volume it will involve for a given mass. Density changes may demonstrate an alternate coal source.

Test procedure

Take a measuring container graduated in ml. clean it altogether with immaculate water. Take water into the jar up to certain level and note down its level (starting perusing). Drop gradually 20 grams of the supplied fly ash example into the jug. Shake the jug for quite a while. Presently note down the level of eater in the container (last reading). Repeat this for 4 specimens and classify the outcomes. Isolate the distinction of the last and introductory perusing by weight of the specimen to acquire genuine thickness.

TRUE DENSITY OF FLY ASH

<table>
<thead>
<tr>
<th>Sl.No.</th>
<th>Amount of fly ash (gm)</th>
<th>Initial reading in (ml)</th>
<th>Final reading in (ml)</th>
<th>Difference in (ml)</th>
<th>True Density in (gm/cc)</th>
<th>Average true density</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>20</td>
<td>50</td>
<td>59</td>
<td>09</td>
<td>2.23</td>
<td></td>
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<tr>
<td>2</td>
<td>20</td>
<td>50</td>
<td>60</td>
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<td>2</td>
<td>2.07</td>
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<tr>
<td>3</td>
<td>20</td>
<td>50</td>
<td>60</td>
<td>10</td>
<td>2</td>
<td></td>
</tr>
</tbody>
</table>

Table 3.2 True density of fly ash

3.3 SPECIFIC GRAVITY

Specific gravity is the ratio of the mass of unit volume of soil at a stated temperature to the mass of the same volume of gas-free distilled water at a stated temperature. The specific gravity of a fly ash is used in the phase relationship of air, water and solids in a given volume of the fly ash.

Test procedure

Determine and record the weight of the empty clean and dry pycnometer, WP. Place 10g of a dry fly ash sample (passed through the sieve No. 10) in the pycnometer. Determine and record the
weight of the pycnometer containing the dry fly ash, WPS. Add distilled water to fill about half to three-fourth of the pycnometer. Soak the sample for 10 minutes. Apply a partial vacuum to the contents for 10 minutes, to remove the entrapped air. Stop the vacuum and carefully remove the vacuum line from pycnometer. Fill the pycnometer with distilled (water to the mark), clean the exterior surface of the pycnometer with a clean, dry cloth. Determine the weight of the pycnometer and contents, WB. Empty the pycnometer and clean it. Then fill it with distilled water only (to the mark). Clean the exterior surface of the pycnometer with a clean, dry cloth. Determine the weight of the pycnometer and distilled water, WA. Empty the pycnometer and clean it.

Calculate the specific gravity of the fly ash using the following formula:
Specific Gravity, \( G_s = \frac{W_0}{W_0 + (WA - WB)} \)
Where:
\( W_0 = \) weight of sample of oven-dry fly ash, g = WPS – WP
\( WA = \) weight of pycnometer filled with water
\( WB = \) weight of pycnometer filled with water and fly ash.

### SPECIFIC GRAVITY OF FLY ASH

<table>
<thead>
<tr>
<th>Sl.no</th>
<th>Mass of empty, pycnometer (WP), (grams)</th>
<th>Mass of empty pycnometer + dry fly ash (WPS), (grams)</th>
<th>Mass of pycnometer + dry fly ash + water (WB), (grams)</th>
<th>Mass of pycnometer + water (WA), (grams)</th>
<th>Specific gravity (GS)</th>
<th>Average Specific gravity</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>43.1</td>
<td>74.83</td>
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<td>140.69</td>
<td>2.21</td>
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<td>2</td>
<td>47.2</td>
<td>76.88</td>
<td>154.5</td>
<td>139.67</td>
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</table>

Table 3.3 specific gravity of fly ash

### 3.4 SETTLING PROPERTIES
Tests on settling rates establish the ease with which solid-liquid separation takes place in slurries during filling activity, and the tests also provide a means of determining the recycled water quality.

**Experimental procedure:**

Take a measuring container graduated in ml. clean it through with impeccable water. Take water into the flask up to certain level (x ml) and a short time later incorporate fly ash(100-x ml) steadily into the jug. Mix the water and fly ash totally with a stirrer for a long time. Note down upper and lower meniscus of the mixture. By then note down time for each ml settling of fly fiery debris stays in water. Repeat the other illustration and orchestrate the results.

<table>
<thead>
<tr>
<th>Time (hrs)</th>
<th>Upper level reading (ml)</th>
<th>Lower level reading (ml)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.3</td>
<td>71</td>
<td>71</td>
</tr>
<tr>
<td>2.35</td>
<td>71</td>
<td>70</td>
</tr>
<tr>
<td>2.42</td>
<td>71</td>
<td>69</td>
</tr>
<tr>
<td>2.47</td>
<td>71</td>
<td>67</td>
</tr>
<tr>
<td>2.56</td>
<td>71</td>
<td>66</td>
</tr>
<tr>
<td>3.10</td>
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<td>63</td>
</tr>
<tr>
<td>3.20</td>
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<td>52</td>
</tr>
<tr>
<td>5.00</td>
<td>71</td>
<td>52</td>
</tr>
</tbody>
</table>

Table 3.4 Settling characteristics of fly ash

**3.5 SEM (Scanning Electron Microscope)**

The **scanning electron microscope** (SEM) is a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain...
information about the sample's surface topography, composition and other properties such as electrical conductivity.

The types of signals produced by an SEM include secondary electrons, back-scattered electrons (BSE), characteristic X-rays, light (cathode luminescence), specimen current and transmitted electrons. Secondary electron detectors are common in all SEMs, but it is rare that a single machine would have detectors for all possible signals. The signals result from interactions of the electron beam with atoms at or near the surface of the sample. In the most common or standard detection mode, secondary electron imaging or SEI, the SEM can produce very high-resolution images of a sample surface, revealing details about less than 1 to 5 nm in size. Due to the very narrow electron beam, SEM micrographs have a large depth of field yielding a characteristic three-dimensional appearance useful for understanding the surface structure of a sample. A wide range of magnifications is possible, from about 10 times (about equivalent to that of a powerful hand-lens) to more than 500,000 times, about 250 times the magnification limit of the best light microscopes. Back-scattered electrons (BSE) are beam electrons that are reflected from the sample by elastic scattering. BSE are often used in analytical SEM along with the spectra made from the characteristic X-rays. Because the intensity of the BSE signal is strongly related to the atomic number (Z) of the specimen, BSE images can provide information about the distribution of different elements in the sample. Characteristic X-rays are emitted when the electron beam removes an inner shell electron from the sample, causing a higher energy electron to fill the shell and release energy. These characteristic X-rays are used to identify the composition and measure the abundance of elements in the sample. Chemical analysis in the scanning electron microscope is performed by measuring the energy or wavelength and intensity distribution of x-ray signal generated by a focused electron beam on the specimen. With the attachment of the energy dispersive spectrometer (EDS) or wavelength dispersive spectrometer (WDS) the precise elemental composition of material can be obtained with high spatial resolution. When we work with bulk specimen in the SEM very precise accurate chemical analysis (relative error- 1-2%) can be obtained from larger areas of the solid (0.5-3 μm dia) using an EDS or WDS.
SEM MICROPHOTOGRAPHS OF FLY ASH UNDER DIFFERENT MAGNIFICATION

fig 3.1 Magnification at 1300x

fig 3.2 Magnification at 5000x

Fig 3.3 Magnification at 8000x

fig 3.4 Magnification at 10000
3.6 PROCTOR TEST

Objective
To determine the optimum moisture content (OMC) and the maximum dry density (MDD) of the given soil sample.

Apparatus Required
- Proctor mould having a capacity of 974 cc with an internal diameter of 10 cm and a height of 12.7 cm. The mould shall have a detachable collar assembly and a detachable base plate.
- Rammer: A mechanical operated metal rammer having a 5.08 cm diameter face and a weight of 2.5 kg. The rammer shall be equipped with a suitable arrangement to control the height of drop to a free fall of 30 cm.
- Sample extruder.
- A balance of 15 kg capacity.
- Sensitive balance.
- Straight edge.
- Graduated cylinder.
- Mixing tools such as mixing pan, spoon, towel, spatula etc.
- Moisture tins.

**Procedure**

1. Take a representative oven-dried sample, approximately 5 kg in the given pan. Thoroughly mix the sample with sufficient water to dampen it to approximately four to six percentage points below optimum moisture content.
2. Weigh the proctor mould without base plate and collar. Fix the collar and base plate. Place the soil in the Proctor mould and compact it in 3 layers giving 25 blows per layer with the 2.5 kg rammer falling through.
3. Remove the collar, trim the compacted soil even with the top of the mould by means of the straight edge and weigh.
4. Divide the weight of the compacted specimen by 974 cc and record the result as the wet weight $g_{\text{wet}}$ in grams per cubic centimetre of the compacted soil.
5. Remove the sample from the mould and slice vertically through and obtain a small sample for moisture determination.
6. Add water in sufficient amounts to increase the moisture content of the soil sample by one or two percentage points and repeat the above procedure for each increment of water added. Continue this series of determination until there is either a decrease or no change in the wet unit weight of the compacted soil.

**Observation & Calculation**

Weight of empty mould – 2080 g
Height of the mould – 12.7 cm
Internal diameter of the mould – 10 cm
Volume of the mould – \( (\pi \times 10^2 \times 12.7 \div 4) \)
Wet density (gm/cc) = weight of compacted soil/vol of mould
Moisture content (%) = (weight of water/weight of dry soil)*100
Dry density (gm/cm$^3$) = Wet density (gm/cm)/ (1+M.C/100)
Plot the dry density against moisture content and find out the maximum dry density and optimum moisture for the soil
Results

Fly ash sample

Optimum Moisture content = 37.53 %

Maximum dry density = 0.983 g/cm3, 983 kg/m3

Fig 3.6 graph between optimum moisture content and dry density

3.7 FTIR Spectroscopy study of fly ash

Fourier Transform Infrared (FTIR) IR Prestige-21 model was used for this investigation. FTIR spectroscopy bases its functionality on the principle that almost all molecules absorb infrared light. Most FTIR spectroscopy uses a Michelson interferometer to spread a sample with the infrared light spectrum and measure the intensity of the infrared light spectrum not absorbed by the sample. FTIR spectroscopy is a multiplexing technique, where all optical frequencies from the source are observed simultaneously over a period of time known as scan time. The spectrometer measures the intensity of a specially-encoded infrared beam after it has passed through a sample. The resulting signal, which is a time domain digital signal, is called an Interferogram and contains intensity information about all frequencies present in the infrared beam. This information can be extracted by switching this signal from a time domain digital signal to a frequency domain digital signal, which is accomplished by applying a Fourier transform over the interferogram and producing what is called a single beam spectrum. Fly ash samples both raw and modified by additives were analysed.
in the laboratory by preparing samples by using a hydraulic press. The results are reported and discussed in the respective sections.

**FTIR analysis**

FTIR (Fourier Transform Infrared Spectrometry) investigation has been carried out to obtain information regarding functional groups of materials/compounds. The quality or consistencies of the fly ash-additive mixture after treatment have been significantly depicted. The FTIR monograph in Figure shows that the untreated fly ash has peaks at 1612.50 cm\(^{-1}\), 797.50 cm\(^{-1}\), and 463.75 cm\(^{-1}\). These peaks attribute to T-O-Si (internal linkage; T = Si or Al); Si-O-Si (external linkage); Si-O-Si or O-Si-O groups of fly ash that are mainly responsible for strength behaviour of the material.

![FTIR analysis of fly ash](image)

**Fig 3.7 FTIR analysis of fly ash**
Chapter -4

RESULTS

AND

DISCUSSION
4. RESULTS & DISCUSSION

4.1 SEM (Scanning Electron Microscope)

The SEM data indicated intermixing of Fe and Al-Si mineral phases and the predominance of Ca non-silicate minerals. The fly ash samples consisted mainly of amorphous alumino-silicate spheres with a lesser number of iron-rich spheres. The majority of the iron-rich spheres consisted of two phases: an iron oxide mixed with amorphous alumino-silicate. The calcium-rich material was distinct in both elemental composition and texture from the amorphous alumino-silicate spheres. It was clearly a non-silicate mineral possibly calcite, lime, gypsum or anhydrite. In spite of the inherent variability of fly ash samples, this analysis indicated that the primary mineral/morphological structures are fairly common. Quartz and alumino-silicates are found as crystals and as amorphous particles.

4.2 SPECIFIC GRAVITY

The specific gravity of the fly ash collected from NACLO Plant was found to be 2.25.

4.3 TRUE DENSITY

The true density of fly ash collected from NALCO plant was found to be 2.07.

4.4 MOISTURE CONTENT

The moisture content of fly ash collected from NACLO was found to be 0.155%. The moisture content of the sample were found out to be around 0.155% indicating that all the moisture have been evacuated and they are suitable for the construction works etc.

4.5 SETTLING CHARACTERISTICS OF FLY ASH

Fly ash: 40 gm and water: 60ml

Upper reading: 79 ml, lower reading: 78 ml

The total time taken for settling of fly ash in the mixtures was found to be 2 hours 47 minutes at the reading 45ml of the mixtures in the flask.
**Fly ash: 50 gm and water: 50 ml**

Upper reading: 74 ml, lower reading: 72 ml

The total time taken for settling of fly ash in the mixtures was found to be 3 hours and 15 minutes at the reading 55ml of mixtures in the flask.

**Fly ash: 35 gm and water: 65 ml**

Upper reading: 82 ml, lower reading: 80ml

The total time taken for settling of fly ash in the mixtures was found to be 2 hours 40 minutes at the reading 39ml of the mixtures in the flask.

**FTIR ANALYSIS**

The FTIR monograph in Figure shows that the untreated fly ash has peaks at 1612.50 cm\(^{-1}\), 797.50 cm\(^{-1}\), and 463.75 cm\(^{-1}\). These peaks attribute to T-O-Si (internal linkage; T = Si or Al); Si-O-Si (external linkage); Si-O-Si or O-Si-O groups of fly ash that are mainly responsible for strength behaviour of the material.
CHAPTER-5
SUMMARY AND CONCLUSION
5. SUMMARY AND CONCLUSION:

- From the compositions of fly ash sample collected, it can be concluded that the fly ash sample belongs to ASTM class F.
- As the fly ash belongs to Class F category which acts as pozzolanic in nature, so it needs alkaline substance for becoming the strengthening.
- Visual observations of the SEM images show a distinct spherical nature for the grains for the fly ash samples.
- The specific gravity attribute to the mineralogical composition i.e presence of silica content and CaO.
- The moisture content of the samples were found out to be around 0.155% indicating that all the moisture have been evacuated and they are suitable for the construction works etc
- Due to the fine grained nature of the solid constituents, the fly ash slurries exhibit marked sluggishness for settling and also did not provided clear supernatant solutions.
- The composition of fly ash: 25 gm and water: 75ml is the good parameter for the separation of solid-liquid in slurries during the filling activity.
- Pozzolanic properties of fly ash can be identified by presence/absence of calcium oxide. So class F fly ash is the weak in pozzolanic as very less amount of calcium oxide present.
- Strengthening of composite is due to dispersion strengthening, particle reinforcement and solid solution strengthening.
- The chemical, physical and mineralogical properties of fly ash had appreciable effects on performance of fly ash in filling low lying and mine void areas.
- Huge amount of fly ash is available for mine void filling and frequently in the vicinity of mines.

    Bituminous (pozzolanic) fly ash is more frequently used to construct embankments and structural backfills than sub-bituminous or lignite (self-cementing) fly ash. This is due in part to the self-cementing characteristics of the latter type, which hardens almost immediately after the addition of water.
REFERENCES

15. www.uic.edu/classes/Experiment 204-Specific Gravity.pdf, Reddy, K