STRENGTH DEVELOPMENT OF FLY ASH BASED COMPOSITE MATERIAL

A THESIS SUBMITTED IN PARTIAL FULFILLMENT
OF THE REQUIREMENTS FOR THE DEGREE OF

BACHELOR OF TECHNOLOGY
IN
MINING ENGINEERING

By

PRASHANT KUMAR NAYAK
V.SRIKRISHNAN

DEPARTMENT OF MINING ENGINEERING
NATIONAL INSTITUTE OF TECHNOLOGY
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CERTIFICATE

This is to certify that the thesis entitled **"STRENGTH DEVELOPMENT OF FLY ASH BASED COMPOSITE MATERIAL"** submitted by **Sri Prashant Kumar Nayak** and **Sri V.Srikrishnan** in partial fulfillment of the requirements for the award of Bachelor of Technology degree in Mining Engineering at the National Institute of Technology, Rourkela (Deemed University) is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/Institute for the award of any Degree or Diploma.

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Prashant Kumar Nayak	**V.Srikrishnan**
Date:	**Date:**

ABSTRACT

The term ‘fly ash’ is often used to describe any fine particulate material precipitated from the stack gases of industrial furnaces burning solid fuels. The amount of fly ash collected from furnaces on a single site can vary from less than one ton per day to several tons per minute. The characteristics and properties of different fly ashes depend on the nature of the fuel and the size of furnace used. Pulverization of solid fuels for the large furnaces used in power stations creates an immediate, urgent problem; dry fly ash has to be collected from the stack gases and disposed of quickly and safely.

Fly ashes generally fall into one of two categories, depending on their origin and their chemical and mineralogical composition. Combustion of anthracite or bituminous coal generally produces low-calcium fly ashes; high-calcium fly ashes result from burning lignite or sub-bituminous coal. Both types contain a preponderance of amorphous glass.

Composite material made of fly ash is used in many ways and is subject to a variety of different loading conditions, and so different types of stress develop. The compressive strength of concrete, one of its most important and useful properties and one of the most easily determined, is indicated by the unit stress required to cause failure of a specimen.

In addition to being a significant indicator of load-carrying ability, strength is also indicative of other elements of quality concrete in a direct or indirect manner. In general, strong concrete will be more impermeable, better able to withstand severe exposure, and more resistant to wear. On the other hand, strong concrete may have greater shrinkage and susceptibility to cracking than a weaker material. Finally, the concrete-making properties of the various ingredients of the mix are usually measured in terms of the compressive strength.

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1. INTRODUCTION

Whether used in buildings, bridges, pavements, or any other of its numerous areas of service, composite must have strength, the ability to resist force. The forces to be resisted may result from applied loads, from the weight of the concrete itself or, more commonly, from a combination of these.

Therefore, the strength of concrete is taken as an important index of its general quality. Hence, tests to determine strength are the most common type made to evaluate the properties of hardened concrete, because (a) the strength of concrete. in compression, tension, shear, or a combination of these, has in most cases a direct influence on the load-carrying capacity of both plain and reinforced structures; (b) of all the properties of hardened concrete, those concerning strength usually can be determined most easily; and (c) by means of correlations with other more complicated tests, the results of strength tests can be used as a qualitative indication of other important properties of hardened concrete.

The results of tests on hardened concrete usually are not known until it would be very difficult to replace any concrete which is found to be faulty. These tests, however, have a policing effect on those responsible for construction and provide essential information in cases where the concrete forms a vital structural element of any building. The results of tests on hardened concrete, even if they are known late, help to disclose any trends in concrete quality and enable adjustments to be made in the production of future concrete.

1.1 BACKGROUND

Fly ash was recognized as pozzolanic ingredient for use in concrete as early as in 1914. However, the earliest comprehensive study on the use of fly ash in concrete was conducted by Davis et al. (1937). Abdun Nur (1961) compiled data on the properties and uses of fly ash from the literature from 1934 to 1959 including an annotated bibliography. Several other extensive review papers on the use of fly ash in concrete have also been published over the years (Synder 1962. Joshi 1979, Berry and Malhotra 1985 and 1987, Swamy 1986).

Much of the literature concerning use of fly ash in concrete before 1980 dealt with fly ashes resulting from the burning of bituminous coal, designated as Class F pozzolans in ASTM specifications. During the early period of 1940 to 1960, usefulness of only Class F fly ash was investigated since that was the generally available ash at that time. Uses of fly ash were established for a number of applications and the advantages and disadvantages were identified. Co-operative tests were conducted by ASTM Committee C-9 (1962) and studies on the fundamental characteristics of Class F fly ashes were reported by Minnick (1959) during this period. The early studies concluded that substantial amount of the Portland cement in concrete could he replaced with fly ash without adversely affecting the long term strength of concrete (Timms and Grieb 1956).

These were basically sub-bituminous ashes. Since 1970s a number of studies have been reported dealing with the characteristics of the fly ashes from sub-bituminous. These self hardening fly ashes generally contained larger amounts of calcium as compared to the bituminous ashes. A new class of ash was therefore added to the ASTM specifications which included ashes with more than 15% calcium oxide and combined silica, alumina, and iron oxide content less than 75%. These ashes exhibit many other useful properties.
The most use and, therefore, most research have been on the use of fly ash in cement and concrete.

1.2 OBJECTIVE

This literature review focuses on important factors affecting strength gain in lime-cement - gypsum - fly ash composite and on laboratory procedures for preparing test specimens and determining the strength of lime-cement- - gypsum - fly ash composite mixtures.

Chapter 2

PURPOSE AND SCOPE OF ANALYSIS

OBJECTIVES OF THE PRESENT STUDY

EXPERIMENTAL STUDY PLAN

2. PURPOSE AND SCOPE

The purposes of this research are to identify factors that contribute to strength gain in lime-cement - gypsum - fly ash composite and to determining the strength of lime-cement- - gypsum - fly ash composite mixtures. The scope of the research is described as follows.

2.1 OBJECTIVES OF THE PRESENT STUDY

Objectives of the present study include:

1. Investigation into the engineering properties and strength characteristics of the fly ash samples collected.
2. Investigation into the strength gain of composite material aspects associated with the one of the fly ash specimen collected.
3. Establishment of better suited combinations of fly ash- gypsum-lime-cement compositions for compressive strength test under laboratory scale/conditions.

2.2 EXPERIMENTAL STUDY PLAN

In order to achieve the objectives outlined, the study plan is divided into the following stages.

1. Collection of the fly ash samples from the thermal power plants.
2. For the samples collected, determination of physicochemical properties of relevance using standard physical and chemical analysis procedures and using of different instruments such as SEM, XRD.
3. Characterization of the fly ash samples with respect to the engineering properties of composite material.

LITERATURE REVIEW

Literature pertaining to laboratory testing methods for lime-cement- gypsum - fly ash mixtures were gathered and examined. Altogether, many papers and several reports were collected, including final reports from the two previously mentioned studies.

Application of laboratory procedure

Fly ashes from seven sites were studied: for lime-cement- gypsum - fly ash composite application. Once the characterization study was completed, the laboratory procedure was used to perform a compressive strength test study of the given fly ash.

Analysis of Results

The results of the laboratory test program were analyzed in order to:

- Identify the major factors affecting strength gain in lime-cement- gypsum - fly ash composite.
- Verify that the laboratory procedure could accurately and reproducibly study the effects of the identified variables.
- Identify the differences in laboratory procedures that would explain the difference in strength gain determination.
- Assess the suitability of the recommended laboratory test lime-cement- gypsum - fly ash composite mix design.

Chapter 3

LITERATURE REVIEW

CHARACTERIZATION OF FLY ASH
ORIGIN OF COAL
BURNING CONDITIONS
TYPES AND PROPERTIES OF FLY ASH

This chapter focuses on important factors affecting strength gain in lime-cement- gypsum - fly ash composite columns and on laboratory procedures for preparing test specimens and determining the strength of lime-cement- gypsum - fly ash composite mixtures. The following topics are covered: properties of the fly ash, preparation prior to mixing, stabilizing agents, dose rates and proportions of stabilizer, mixing of the fly ash with additives and stabilizer, sample production, curing, sample extraction, and strength testing.

3.1 CHARACTERIZATION OF FLY ASH

3.1.1 ORIGIN OF COAL

Coal is a complex, heterogeneous material, in widespread use as an energy source throughout the world. It is the end product of a series of biological and physicochemical processes which have resulted in the wide variety of minable materials currently utilized in industry.

When pulverized coal is burnt to generate electrical power, extremely large quantities of fly ash and bottom ash are produced. Fine grade fly ash has acquired considerable importance in the building materials sector.

Coals are formed in the earth’s interior over periods in the order of 300 to 400 million years. Over such long periods, the different kinds of plant material from which coal is formed undergo complex transformations, so that the nature and properties of the great variety of coals we now utilize are dependent on the class of plants which have been transformed and on the depth to which these have been buried. Together with the depth of burial, high temperatures and pressures play an important role in determining coal composition and characteristics.

Coal attains its final stale in combination with a range of different compounds, and can he sub-divided into various classes or groups such as peat, lignite, sub-bituminous and bituminous coals and anthracite.

The quantity of water present in these different classes of coal decreases in proportion to their ascending rank, ranging from 90 % for peat's to 1.5 % for anthracites.

Characterization of coals demands knowledge of the following parameters:

- Moisture,
- Ash content,
- Volatile matter,
- fixed carbon,
- sulphur content (organic, pyritic and sulphatic sulphur),
- Calorific or heating value.

3.1.2 BURNING CONDITIONS

Coal is burned in power stations in order to generate the heat required to turn water into steam which can be used to drive steam turbines. The energy of the coal is finally converted into electrical power. In accordance with the ranking noted above, anthracite has the highest and lignite the lowest calorific value of the coals used as power station fuels.

Three different processes are employed for the combustion of pulverized coal in power station boilers:

- High temperature combustion: here, combustion occurs at furnace temperatures of some 1500 - 1700 °C. The resulting ash melts and falls into water, where it collects in the form of solid, mainly vitreous particles. Only a small quantity of fine particles escapes to electrostatic precipitators in the form of fly ash. Furnaces of this type are generally referred to as slag-tap furnaces.

- Dry combustion: in this case, the pulverized coal is burnt at furnace temperatures of 1100 to 1 400 °C. Roughly 90 % of the ash collected from the process is in the form of ultra-fine particles retained by electro filters or precipitators. Since the temperature decreases slowly, the percentage of vitreous particles is low.
- Fluidized-bed combustion: the furnace temperature in the fluidized beds is less than 900 C, excluding melting. Ashes are irregularly shaped, with a high percentage of crystalline particles. These are not genuine fly ashes, and are of little interest for building material applications.

Coal is used as fuel for about 40 to 50% of electric power generation all over the world.

3.2 COLLECTION OF FLY ASH

During the combustion of pulverized coal in suspension-fired furnaces of modern thermal power plants, the volatile matter is vaporized and the majority of the carbon is burned off. The mineral matter associated with the coal, such as clay, quartz and feldspar disintegrate or slag to varying degree. The slagged particles and unburned carbon are collected as ash. The coarser particles fall in the bottom of the furnace and are collected as bottom ash or boiler slag. The finer particles that escape with flue gases are collected as fly ash using cyclone separators, electrostatic precipitators or bag houses.

Depending upon the collection system varying from mechanical to electrical precipitators or bag houses and fabric filters, about 85 to 99.9% of the ash from the flue gases is retrieved in the form of fly ash. Fly ash, accounts for 75 to 85% of the total coal ash, and the remainder is collected as bottom ash or boiler slag. Fly ash because of its mineralogical composition, fine particle size and amorphous character is generally pozzolanic and in some cases also self cementitious. The bottom ash and boiler slag are much coarser and are not pozzolanic in nature. It is thus important to recognize that all the ash is not fly ash and the fly ashes produced by different power plants are not equally pozzolanic and, therefore, are not always suitable for use as mineral admixture in concrete.

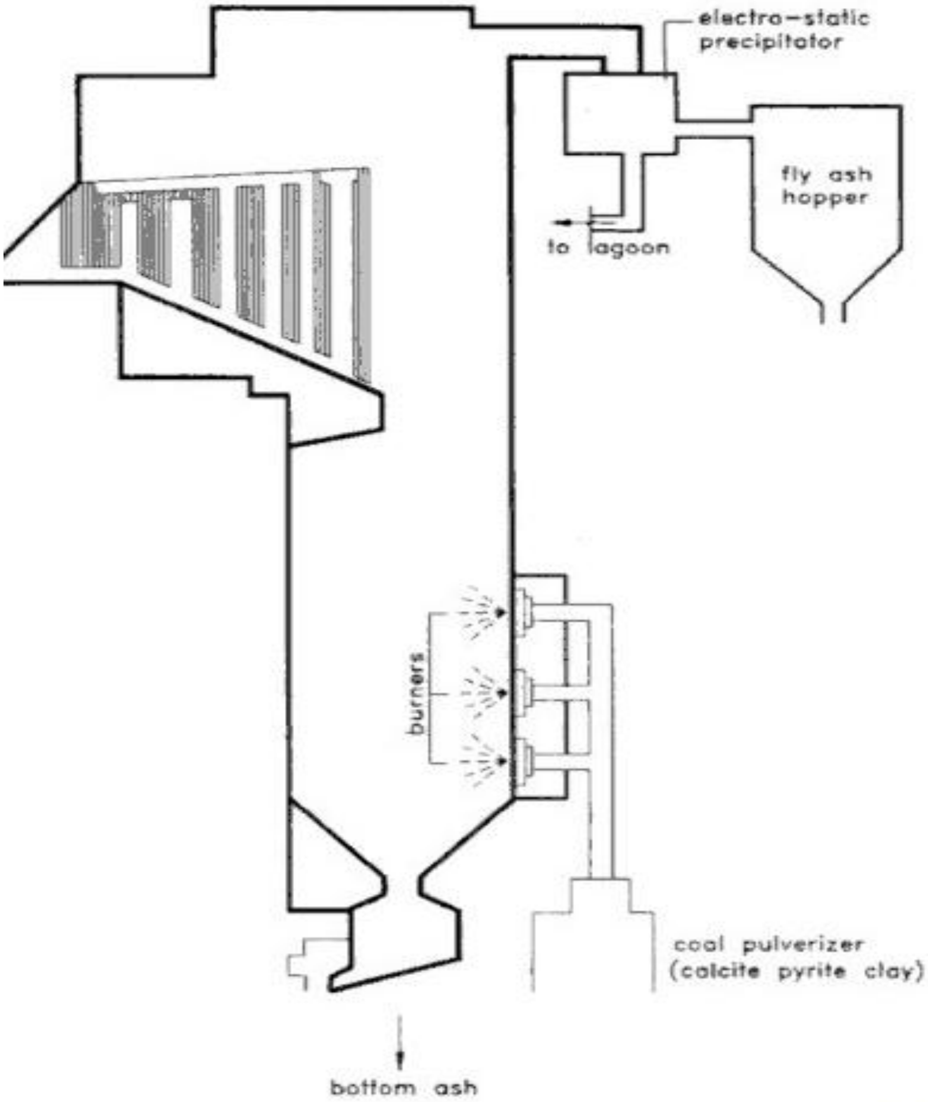


Fig 3.2 Schematic diagram showing fossil fuel furnace and fly ash collection system

Fly ash generated in coal burning power plants is an inherently variable material because of several factors. Among these are the type and mineralogical composition of the coal, degree of coal pulverization, type of furnace and oxidation conditions including air-to-fuel ratio, and the manner in which fly ash is collected, handled and stored before use. Since no two utilities or plants may have all of these factors in common, fly ash from various power plants is likely to be different. The fly ash properties may also vary within the same plant because of load conditions over a twenty four hour period. Non uniformity of fly ash is a serious disadvantage and sometimes is the main hurdle in the effective and wide scale utilization of fly ash as a pozzolan or a cementitious component in cement and concrete.

3.3 TYPES AND PROPERTIES OF FLY ASH

3.3.1 DEFINITIONS AND SPECIFICATIONS

Pozzolans are siliceous or siliceous and aluminous materials which, though themselves possessing little or no cementitious value, will, in finely divided form and in the presence of moisture, react chemically with calcium hydroxide at ambient temperature to form compounds with cementitious properties (ASTM Standard C618-80).

Fly ash is a solid, fine-grained material resulting from the combustion of pulverized coal in power station furnaces. The material is collected in mechanical or electrostatic separators. The term fly ash is not applied to the residue extracted from the bottom of boilers.

Fly ashes capable of reacting with $Ca(OH)_2$ at room temperature can act as pozzolanic materials. Their pozzolanic activity is attributable to the presence of $Si0_2$ and $A1_20_3$ in amorphous form.

According to ASTM C618-93 specification (1993) for “F1y Ash and Raw or Calcined Natural Pozzolan “for use as Mineral Admixture in Portland Cement Concrete,” pozzolans are defined as “siliceous and aluminous materials which in themselves possess little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties.”

ASTM C618-93 categorizes natural pozzolans and fly ashes into the following 3 categories.

- Class N: Raw or calcined natural pozzolans such as some diatomaceous earths, opaline, chert and shale, stuffs, volcanic ashes and pumice are included in this category. Calcined kaolin clay and laterite shale also fall in this category of pozzolans.
- Class F: Fly ash normally produced from burning anthracite or bituminous coal falls in this category. This class of fly ash exhibits pozzolanic property but rarely, if any, self hardening property.
- Class C: Fly ash normally produced from lignite or sub-bituminous coal is the only material included in this category. This class of fly ash has both pozzolanic and varying degree of self cementitious properties. (Most Class C fly ashes contain more than 15% CaO. But some Class C fly ashes may contain as little as 10% CaO).

Many other forms of classification can be accepted, e.g. classification according to carbon content, SiO2 reactivity, Si02 solubility, pozzolanic activity etc.

Requirements	Mineral admixture class		
	N	**F**	**C**
Chemical requirements			
S03 + Al203 + Fe203,min%	70.0	70.0	50.0
SO3, max%	4.0	5.0	5.0
Moisture content, max%	30	3.0	3.0
Loss on ignition, max%	10.0	6.0	6.0
Physical requirements			
Amount retained when wet sieved on 45 µm sieve,	34	34	34

max%			
Pozzolanic y activity index, with Portland cement at 28 days ,min% of control	75	75	75
Pozzolanic activity index, with lime at 7days,min(MPa)	5.5	5.5	-
Water requirement, max% of control	115	105	105
Autooc1ave expansion or contraction, max %	0.8	0.8	0.8
Specific gravity, max variation from average %	5	5	5
Percent retained on 45 µm sieve ,max variation percentage points from average	5	5	5

Table 3.3.1 presents chemical and physical requirements for fly ash and natural pozzolans for use as a mineral admixture in Portland cement concrete. The table also includes a list of procedures and materials used for assessing the quality of fly ash/natural pozzolans to meet the requirements of ASTM C618.

3.3.2 CLASSIFICATION

As according to ASTM C-618, two major classes of fly ash are recognized. These two classes are related to the type of coal burned and are designated Class F and Class C in most of the current literature. Class F fly ash is normally produced by burning anthracite or bituminous coal while Class C fly ash is generally obtained by burning sub-bituminous or lignite coal. The important characteristics of these two types of ashes are discussed below.

Presently, no appreciable amount of anthracite coal is used for power generation. Therefore, essentially all Class F fly ashes presently available are derived from bituminous coal. Class F fly ashes with calcium oxide (CaO) content less than 6%, designated as low calcium ashes, are not self hardening but generally exhibit pozzolanic properties. These ashes contain more than 2% unburned carbon determined by loss on ignition (LOI) test. Quartz, mullite and hematite are the major crystalline phases identified fly ashes, derived from bituminous coal. Essentially, all the fly ashes and, therefore, most research concerning use of fly ash in cement and concrete are dealt with Class F fly ashes.

In the presence of water, the fly ash particles produced from a bituminous coal react with lime or calcium hydroxide to form cementing compounds similar to those generated on the hydration of Portland cement. Previous research findings and majority of current industry practices indicate that satisfactory and acceptable concrete can be produced with the Class F fly ash replacing 15 to 30% of cement by weight.

When Class F fly ash is used for producing air entrained concrete to improve freeze-thaw durability, the demand for air entraining mixtures is generally increased.

Use of Class F fly ash in general reduces water demand as well as heat of hydration. The concrete made with Class F fly ash also exhibits improved resistance to sulphate attack and chloride ion ingress.

Class C fly ashes, containing usually more than 15% CaO and also called high calcium ashes, became available for use in concrete industry only in the last 20 years in the 1970s. Class C fly ashes are not only pozzolanic in nature but are invariably self cementitious. When mixed with water, Class C ashes hydrate almost in the same way as Portland cement does. In many cases this initial hardening occurs relatively fast. The degree of self hardening generally varies with the calcium oxide present in the fly ash and cementitious.

Joshi (1982) also conducted studies on the effect of coarse fraction larger than the 45 µm on the setting lime and strength development of concrete. The data suggested an increase in setting time and only a slight decrease in strength.

3.3.3 SPECIFIC GRAVITY

The specific gravity of fly ash is reported to be related to shape, colour as well as chemical composition of fly ash particle. It is adopted as an indirect performance parameter for determining the performance of fly ash in concrete. In ASTM C618, for quality control of fly ash, the uniformity of the fly ash is monitored by limiting the variability of the specific gravity and fineness as measured by the amount retained on 45 μm mesh sieve. The requirement is that any sample tested shall not deviate from the average of 10 previous tests or the total of all tests if the number is less than 10, by more than 5%.

In general specific gravity of fly ash may very from 1.3 to 4.8 (Joshi 1968). However, the Canadian fly ashes have specific gravity ranging from 1.91 to 2.94 whereas those of the American ashes have specific gravity between 2.14 and 2.69. Coal particles with some minerallic impurities have specific gravity between 1.3 and 1.6. Opaque spherical magnetite (ferrite spinel) and hematite particles, light brown to black in colour, when present in sufficient quantity in fly ash increase the specific gravity to about 3.6 to 4.8. As the amount of quartz and mullite increases, the specific gravity decreases. Fly ash pulverization releases some of the gases trapped, during quenching inside the large hollow spherical particles, and increases the bulk specific gravity of the fly ash (Joshi 1968, 1979).

3.3.4 DENSITY

The particle density of fly ash is typically 1.5 - 2.5 mg/m3, the lower density associated with a high a LOI .There is some variability in the density of particles, with smaller ones having higher densities. This is due to air voids within many of the particles, and between 1% and 5% contains sufficiently large voids that they float on water. The variation in particle density means that sedimentation techniques for determining the particle size distribution are not suitable and more appropriate methods arc now used, e.g. laser scattering. The heavy compaction will give higher maximum dry densities and lower optimum moisture content values, but they are only slightly different from the light compaction; the latter produces more realistic target values for site control.

3.4 BASIC CONCEPT OF FLY ASH AS A POZZOLAN IN CONCRETE

Setting or hardening of ordinary Portland cement concrete occurs due to hydration reaction between water and the cementitious compounds in cement which give rise to several types of hydrates of calcium silicate (CSH) and calcium aluminate (CAH) besides calcium hydroxide (CH). These hydrates arc generally referred as tobermorite gel. Adhesive and cohesive properties of the gel hind the aggregate particles. With time the cement paste exhibits setting and hardening which impart concrete its properties in fresh and hardened state.

Calcium hydroxide is really a by-product of cement hydration (Neville, 1981). When fly ash in incorporated in concrete. The calcium hydroxide liberated during hydration of Portland cement reacts slowly with the amorphous alumino-silicates, the pozzolanic compounds present in the fly ash. The products of these reactions, termed as pozzolanic reaction products are time dependent but are basically of the same type and characteristics as the products of the cement hydration. Thus additional cementitious products become available which impart additional strength to concrete. Because the pozzolanic reactions are much slower than the cement hydration reactions, partial replacement of the cement in concrete generally reduces early strength, but may be equal or increase the long term strength. The rate of strength gain, however, depends upon properties of the fly ash and cement used mix proportions, as well as the curing conditions of the fly ash concrete (Joshi 1979).

Concrete mixes are designed with more water than needed for cement hydration to obtain proper workability. This excess water is present in capillary channels of the hydrated cement paste and is commonly referred as capillary water. In a properly cured concrete, the calcium hydroxide dissolved in the capillary water would react with the fly ash to form the solid reaction products that will fill partially or completely the capillary channels. The blocking of the capillary pore/channels both by physical action due to fine particle size and due to the formation of new products of pozzolanic reaction results in lower permeability of concrete. The reduced permeability also reduces the aggressive and deleterious action of the salt solutions such as chloride or sulphate solutions.

Another important aspect is that the cement hydration reactions are exothermic iii that a portion of the latent energy required to combine the elements is released by the hydration reaction. As a result the temperature of concrete is raised. Initial setting or hydration of cement rapidly increases the temperature within a large mass of concrete since the heat is not dissipated quickly enough. The subsequent cooling of the concrete introduces internal stresses of sufficient magnitude to cause cracking. With partial replacement of cement by fly ash in concrete the heat of hydration is reduced. Further the heat is released over a long period of time because of the reduced amount of cement and slower pozzolanic reactions. Thus the temperatures in mass concrete, in particular, remain lower because heat is dissipated as it develops.

As a result of extensive laboratory and field research the use of fly ash as pozzolan in cement concrete has been well established and recognized by concrete industry. When the fly ash also has cementitious properties, as is the case with Class C fly ashes, additional strength producing reactions also occur. These reactions although complex but are generally considered similar to normal hydration reactions of Portland cement Therefore, the heat of hydration may not reduce significantly when high calcium Class C fly ash is used for partial replacement of cement in concrete.

3.5 CEMENT EFFECTS ON CONCRETE STRENGTH

To produce higher strength concretes several parameters have to be optimized in addition to mixture design. Although the design of the concrete mixture is a mayor factor in achieving the desired strength. In general, high strength concrete contains strong aggregates, a higher Portland cement content and a low water/cement or cementitious ratio. The addition of water reducing admixtures, super plasticizer, polymer, and blast furnace slag or silica fume is common today.

Following are the major factors that have to be taken into account:

1. Cement characteristics and content
2. Water/cement, liquid/cement, and water/cementitious ratios
3. Aggregate quality and interaction with cement paste
4. Chemical admixture
5. Mineral admixture.
6. Procedure and mixing time of constituent (%)
7. Quality control and assurance

These factors control strength, bond between the fly ash particles and the aggregate and the resulting interlock between the aggregate particles in the concrete. The strength of the coarse aggregate %should at least be equialent to the % strength of the binding matrix in order to achieve the high compressive strength levels that are needed. If not, failure would result by fracture lines passing through the coarse aggregate while the higher-strength binding matrix remained intact.

3.6 EFFECTS OF FLY ASH ON THE PROPERTIES OF FRESH CONCRETE

The present state of knowledge recognizes the usage of fly ash in cement and concrete as raw material in cement production, as an ingredient in blended cement, and as mineral admixture in concrete. Sometimes fly ash is also used as partial replacement of fines. Based on laboratory investigations as well as field applications of fly ash concrete over the last 50 years, several comprehensive reviews and other publications present the accepted views related to the advantages and disadvantages of incorporating fly ash in concrete.

The majority of the recognized effects of fly ash on concrete properties tend to improve concrete performance in field use. The addition of fly ash to concrete affects its properties both in the fresh and hardened states favorably. The nature and degree of effect on a specific concrete property however, depends upon several factors such as type and amount of fly ash, mix proportion, chemical admixtures, curing conditions, and other job requirements including construction practices.

In fresh concrete, fly ash plays an important role in the fluidity of concrete which is commonly expressed in such phenomenological measurements as workability, pump ability, compactability, water demand, bleeding and segregation and finish-ability. Addition of fly ash has significant influence on the rate of hydration reactions as well as on the effectiveness of the chemical admixtures, particularly air entraining agent and water reducer or super plasticizer.

Low calcium Class F fly ash normally acts as a fine aggregate of spherical form in early stages of hydration whereas high calcium Class C fly ash may contribute to the early cementing reactions in addition to its presence as fine particulate in the concrete mix Hydration of cement is an exothermic reaction and the released heat causes a rise of temperature of fresh concrete. For producing high strength concrete, high range water reducer or super plasticizer is added to maintain the given workability of concrete at a low water-cement ratio. Furthermore, air entrainment of adequate amount, usually 6 ± 1 % is obtained by using air entraining admixture to improve freeze-thaw durability of concrete. As fly ash forms one of the components of concrete its effects on the general properties of fresh concrete need better understanding.

Chapter 4

METHODS AND MATERIALS

4. METHODS & MATERIALS

The purposes of this research are to identify factors that contribute to strength gain in composite specimen. This section summarizes the procedures and materials used in performing the investigation. The sections are the fly ash characterization methods and the method for preparing lime-cement- gypsum-fly ash mix specimens.

4.1 FLY ASH CHARACTERIZATION

Fly ash characterization test were performed on the fly ashes that were investigated in this project. Tests included moisture contents, density, specific gravity, pH, mineralogical analyses and compressive strength.

4.2 MOISTURE CONTENT (ASTM D 2216)

About 1 g of finely divided powdered air dried fly ash sample is weighed in a crucible. The crucible is placed inside an electric hot air oven, maintained at 105° C. The crucible is allowed to remain in the oven with lid open for 24 hours and then taken out (with a pair of tongs), cooled in a desiccator and weighed. Loss in weight is reported as moisture (on % basis).

Percentage of moisture = (loss in weight / wt of coal taken) * 100

4.3 SPECIFIC GRAVITY (ASTM D 854)

Values of the specific gravity of the fly ash samples were determined by first weighing a 250 ml flask empty (W F) and then full of water (W FW). A known weight of air-dried fly ash (W S) was placed in the flask, which was then filled to the 150 ml mark and weighed again (W FWS). The weight of water displaced by the fly ash can be calculated as Ww = W S+ W FW -W FWS. Specific Gravity can then be calculated as Gs = W S / Ww.

4.4 PH (ASTM D 4972)

Fly ash samples were adjusted to 100% water content by adding distilled water, and pH values were measured using a calibrated pH probe.

4.5 MINERALOGICAL ANALYSIS

Mineralogical analysis consisted of x-ray diffraction (XRD) and scanning electron microscope on the fly ash samples.X-ray diffraction was used to determine mineral suites present by analyzing oriented. Samples were scanned at a fixed counting time of 4 seconds at 0.075° of 2 per step using CuKa radiation (20 mA, 40 kV). Mineral quantities were estimated as integrated intensities of their respective XRD peaks. The fly ash samples were analyzed at the Laboratory using the SEM equipped with back scattered and secondary electron detectors. The SEM provides detailed imaging information about the morphology and surface texture of individual particles, as well as elemental composition of samples.

4.6 SEM (SCANNING ELECTRON MICROSCOPY)

The purpose of this experiment is to use scanning electron microscopy (SEM) to characterize the morphology of a random group of fly ash samples from a large number of widely dispersed sources.

An experiment is performed in the laboratory of SEM to characterized 7 fly ash samples collected from 7 thermal power plants of states namely Orissa, Jharkhand and Tamil Nadu, using scanning electron microscopy (SEM) instrument. Both the surface and internal structure of fly ash particles were analyzed. The fly ash samples were analyzed at the Laboratory using the SEM equipped with back scattered and secondary electron detectors. The SEM provides detailed imaging information about the morphology and surface texture of individual particles, as well as elemental composition of samples.

A representative portion of 7 fly ash samples was sprinkled on to double-sided carbon tape mounted on a SEM stub. This grain mount enables the analyst to determine the particle morphology, external surface structure and external elemental distribution of individual fly ash particles.

Seven of the samples were also studied in polished cross section in order to examine the internal structure and composition of individual particles. Each fly ash sample was characterized by selecting 1 field of view and examining all the fly ash particles observed within the selected field.

4.7 XRD (X-RAY DIFFRACTION)

X-ray diffraction (XRD) is an instrumental technique that is used to identify minerals, as well as other crystalline materials. XRD provides the researcher with a fast and reliable tool for routine mineral identification.

The minerals are defined by regular, repeating planes of atoms that form a crystal lattice. When a focused X-ray beam interacts with these planes of atoms, part of the beam is transmitted, part is absorbed by the sample, part is refracted and scattered, and part is diffracted.

Diffraction of an X-ray beam by a crystalline solid is analogous to diffraction of light by droplets of water, producing the familiar rainbow. X-rays are diffracted by each mineral differently, depending on what atoms make up the crystal lattice and how these atoms are arranged.

In X-ray powder diffractometry, X-rays are generated within a sealed tube that is under vacuum. A detector detects the X-ray signal; the signal is then processed either by a microprocessor or electronically, converting the signal to a count rate.

Changing the angle between the X-ray source, the sample, and the detector at a controlled rate between preset limits is an X-ray scan.

The geometry of an XRD unit is designed to accommodate this measurement .The characteristic set of d-spacings generated in a typical X-ray scan provides a unique "fingerprint" of the mineral or minerals present in the sample. When properly interpreted, by comparison with standard reference patterns and measurements, this" fingerprint" allows for identification of the material.

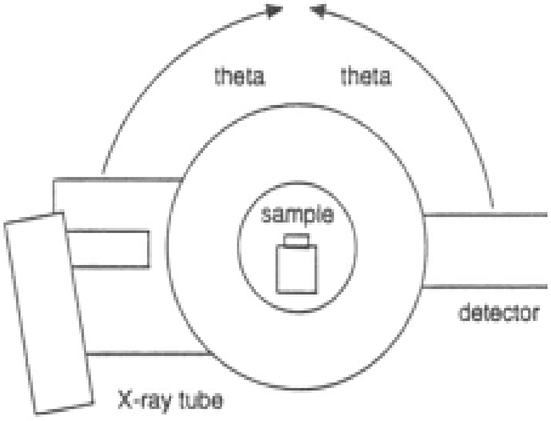


Fig. 4.7 Diagram shows a simplified sketch of one possible configuration of the X-ray source (X-ray tube), the X-ray detector, and the sample during an X-ray scan. In this configuration, the X-ray tube and the detector both move through the angle theta and the sample remains stationary.

4.8 PREPARATION OF FLY ASH COMPOSITE MATERIAL

The fly ash is chosen for its low lime content as well as its availability in abundance. On the basis of the literature reviewing, different lime proportions (0, 2, 4 and 6) % of fly ash (by weight) were selected. Similarly, percentages of gypsum were (0, 1, 2, 3, and 4 and 5) % of fly ash (by weight). And that of cement are (0, 2, 4, 6, 8 and 10) % of fly ash (by weight).

The additives selected are commercially available which are lime, gypsum and cement. The addition of lime enhances the pozzolanic reactivity of fly ash containing insufficient free lime required for pozzolanic reaction with its reactive silica.

Gypsum is chosen for avoiding the interference of impurities because impurities may retard the initial hydration process.

Depending on the sample dimension, required quantities of fly ash, lime, gypsum, cement and water quantity (30 %) of the weight of fly ash sample and are thoroughly mixed by hand. Then it was kept inside a polythene bag for one hour for moisture homogenization. The samples were cast to NX size core i.e. 54 mm diameter and 108 mm length for compressive strength tests.

The samples were taken out of mould after 72 hours and kept in moist proof containers that were in turn placed inside humidity control chambers where the temperature was maintained at about 30˚C ±1%.

4.9 COMPRESSIVE STRENGTH TEST (ASTM D 2166)

Compression strength tests were performed on specific specimens. Using a strain rate equal to 1% of initial specimen length per second (equaling 0.1 mm/sec), a data acquisition system was used to record the deflection measured and load applied. The test proceeded until failure occurred. The data was then downloaded into a spreadsheet so that area corrections could be made and the true unconfined compressive strength calculated. Failure was defined as the peak stress, which typically occurred at 2 to 8 percent strain**.**

Concrete is used in many ways and is subject to a variety of different loading conditions, and so different types of stress develop. Very often the dominant stress is compressive in nature, since this material has long been known to exhibit its best strength characteristics when subjected to compressive loading. The compressive strength of concrete, one of its most important and useful properties and one of the most easily determined, is indicated by the unit stress required to cause failure of a specimen.

Concrete also exhibits tensile and shear strength, in which compressive strength is frequently used as a measure of these properties. The tensile strength of concrete is roughly 10 to 12 percent of the compressive strength and the flexural strength of plain concrete, as measured by the modulus of rupture, is about 15 to 20 percent of the compressive strength.

In addition to being a significant indicator of load-carrying ability, strength is also indicative of other elements of quality concrete in a direct or indirect manner. In general, strong concrete will be more impermeable, better able to withstand severe exposure, and more resistant to wear. On the other hand, strong concrete may have greater shrinkage and susceptibility to cracking than a weaker material.

Finally, the concrete-making properties of the various ingredients of the mix are usually measured in terms of the compressive strength.

Chapter 5

RESULT AND SUMMARY

5.1 SPECIFIC GRAVITY

Specific Gravity of the fly ash sample collected from PTPS is found to be 2.35.
Specific Gravity of the fly ash sample collected from ETPS is found to be 2.20

5.2 MOISTURE CONTENT

Percentage of moisture = (loss in weight / wt of coal taken) * 100
Moisture content of the fly ash sample collected from PTPS is found to be 0.15 %
Moisture content of the fly ash sample collected from ETPS is found to be 0.2 %

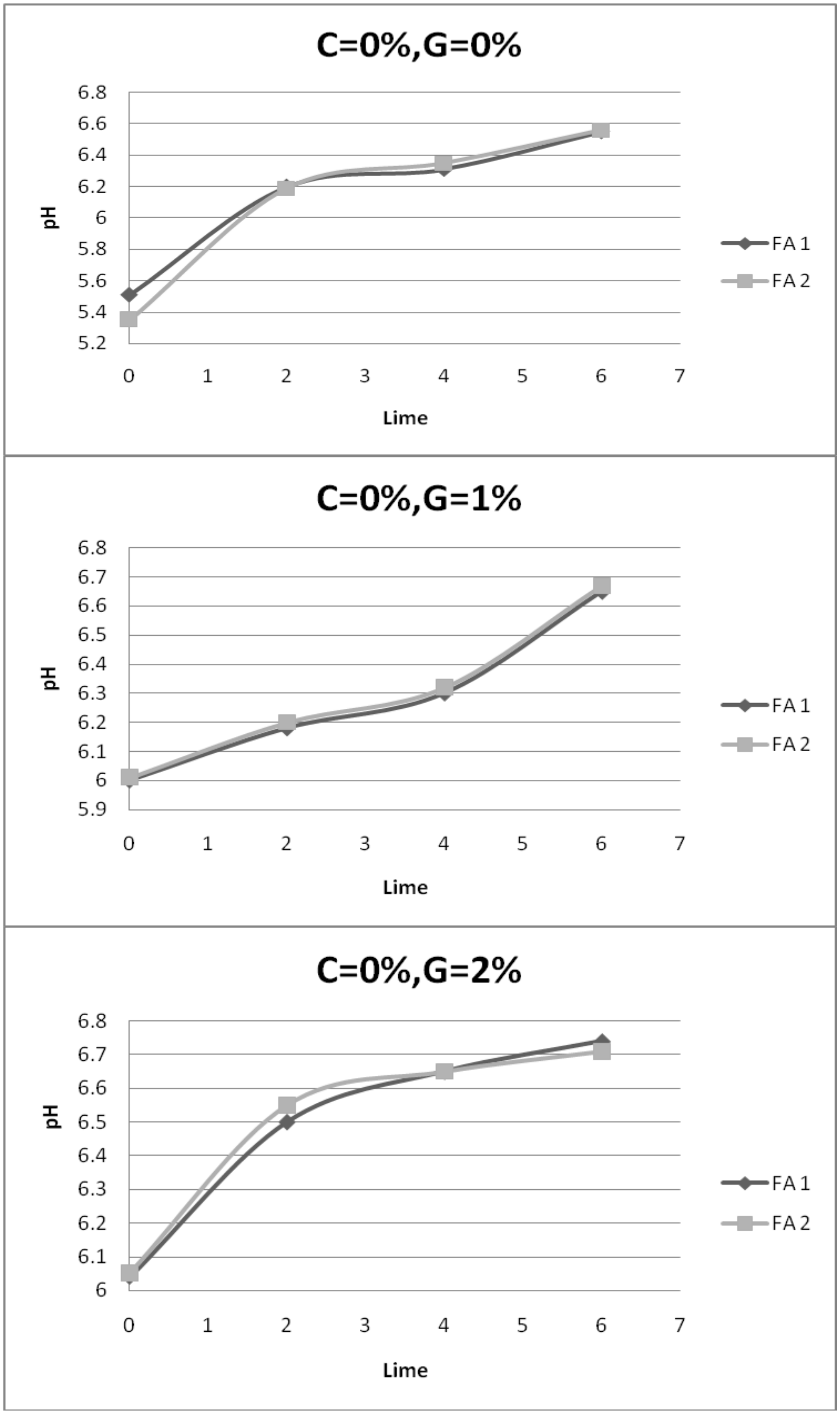
5.3 pH DETERMINATION

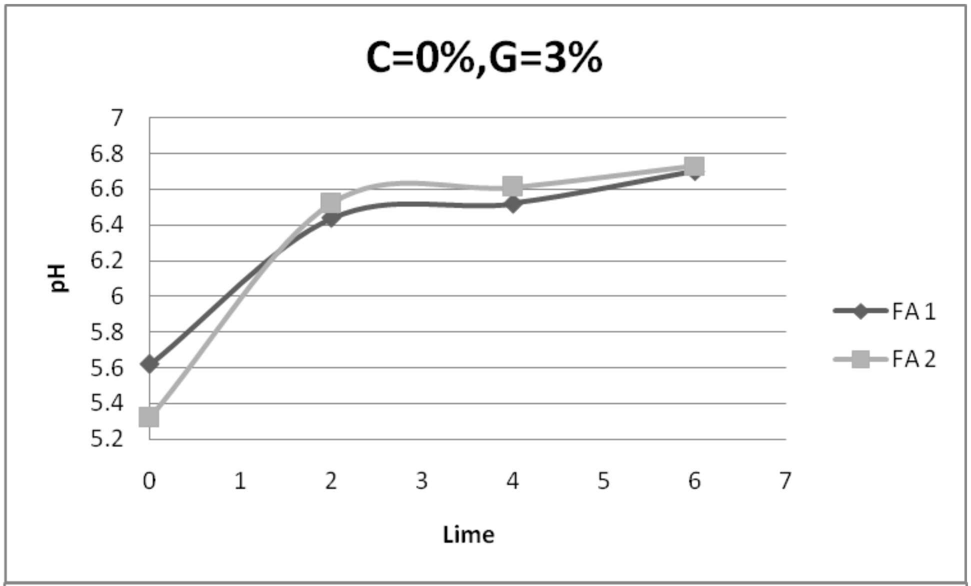
5.3.1 RESULTS

When water is added to fly ash, it initially has a low pH as the sulfate deposited on the surface of the particles is brought into solution as sulfuric acid. This is a transient situation and the pH rapidly rises as calcium is leached into solution with variable amount of lime ,gypsum and cement by percentage of weight of fly ash. The pH is typically 9-11 for fly ash, although the pH for those ashes with higher free calcium oxide contents can rise to 12. Only a very small quantity of free calcium is required to achieve the higher pH. Because most of the water-soluble material that influences pH has been washed out of lagoon fly ash, the pH is lower, typically around 9.

Note :
FA1 – Patratu thermal power plant
FA2 – Ennore thermal power plant

Fig 5.3.1 pH characteristics of Fly ash mixtures

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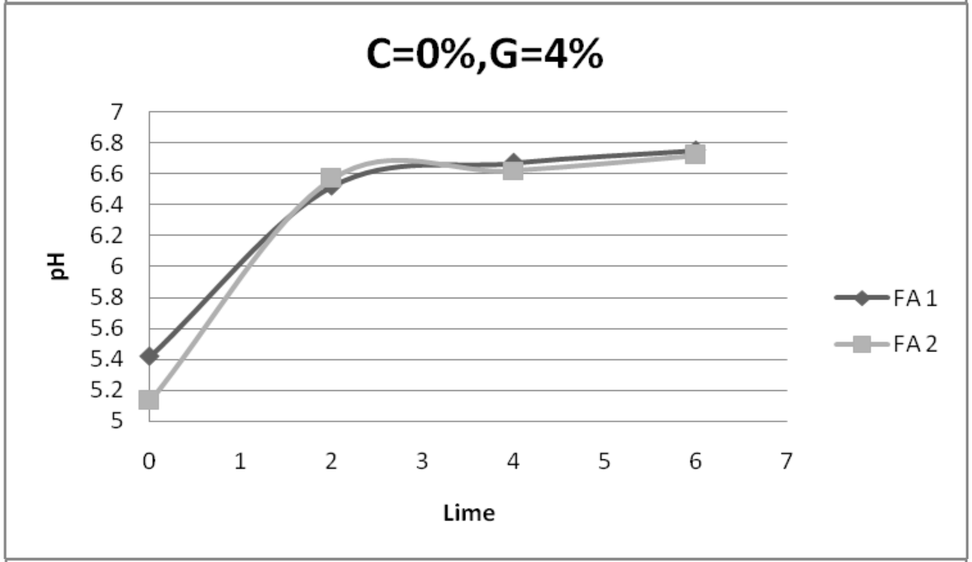
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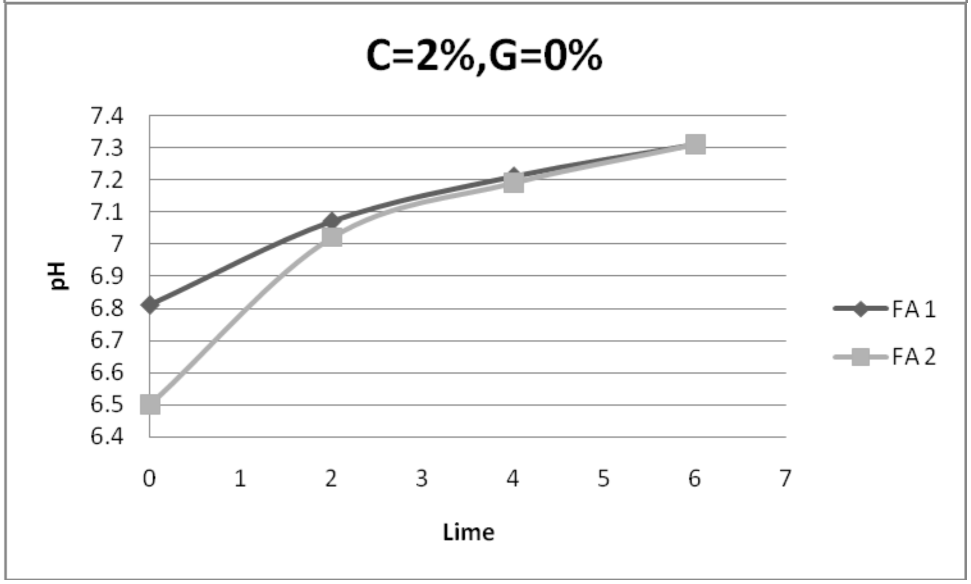
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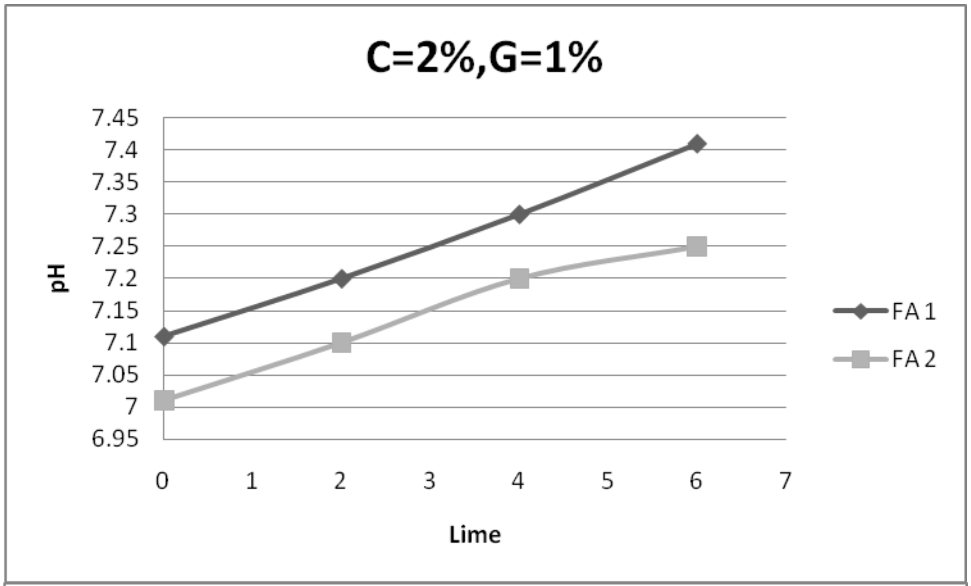
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Lime
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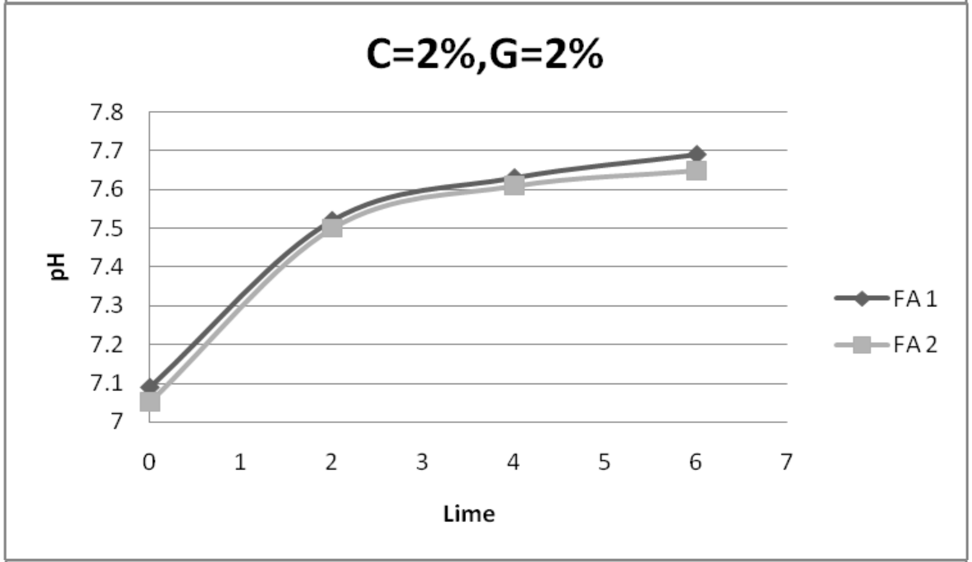
FA 2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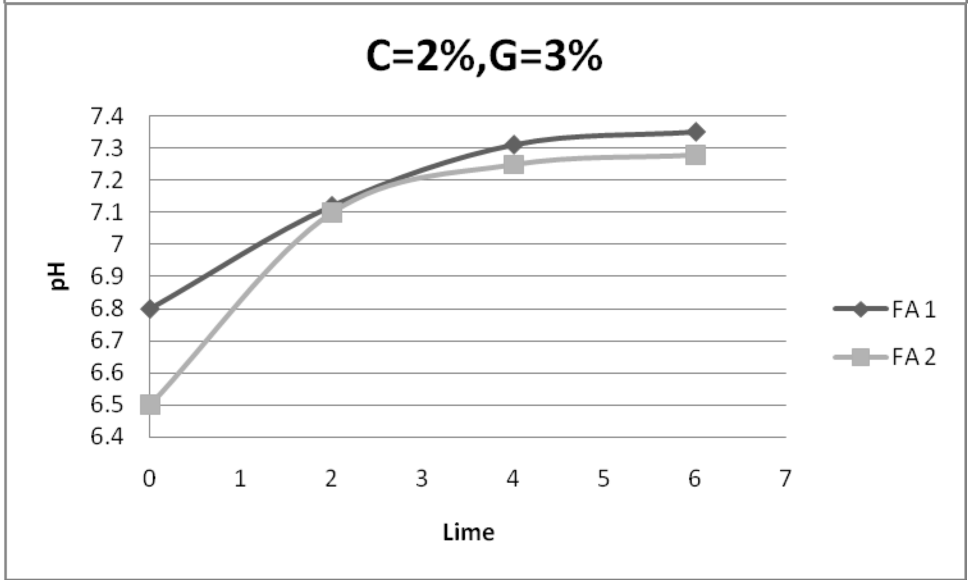
FA 2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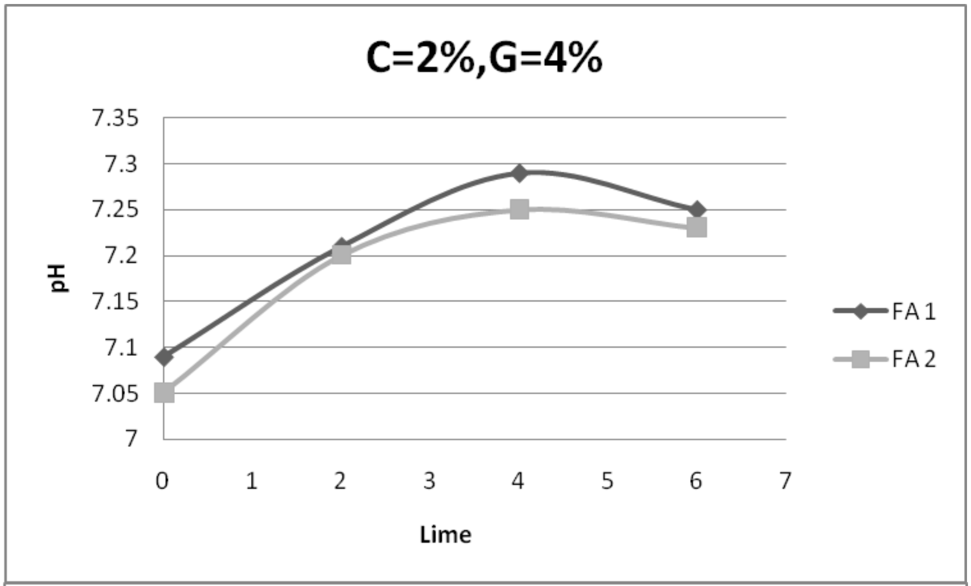
FA 1
FA 2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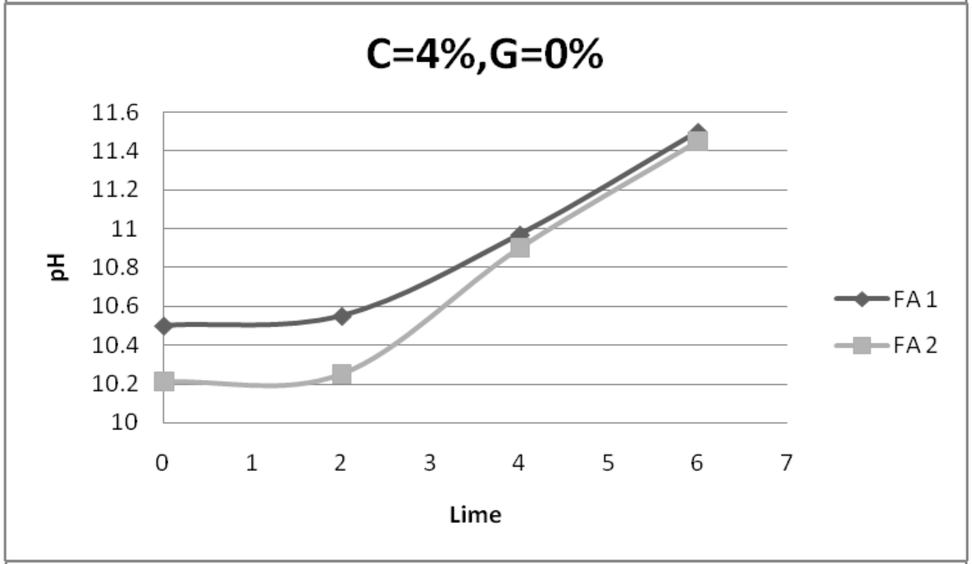
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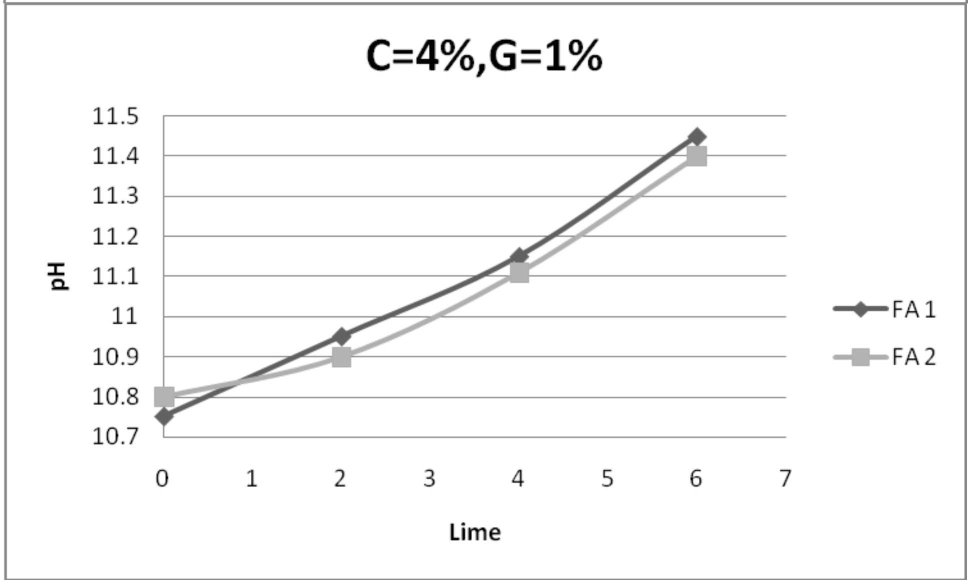
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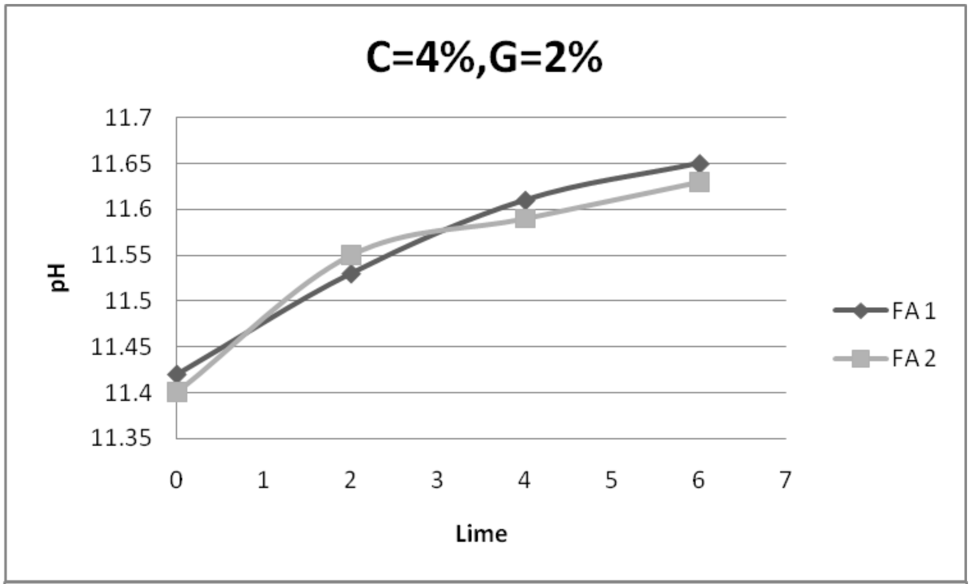
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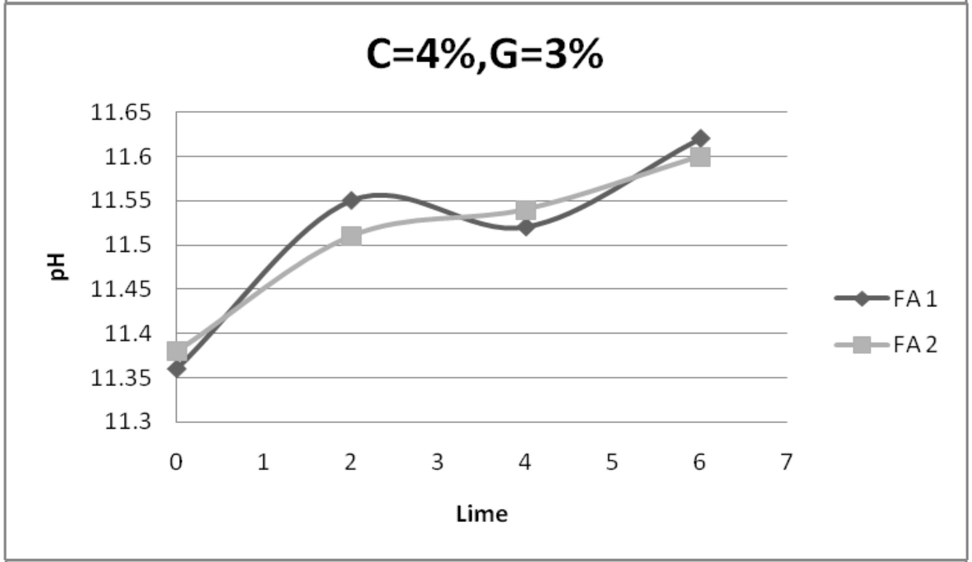
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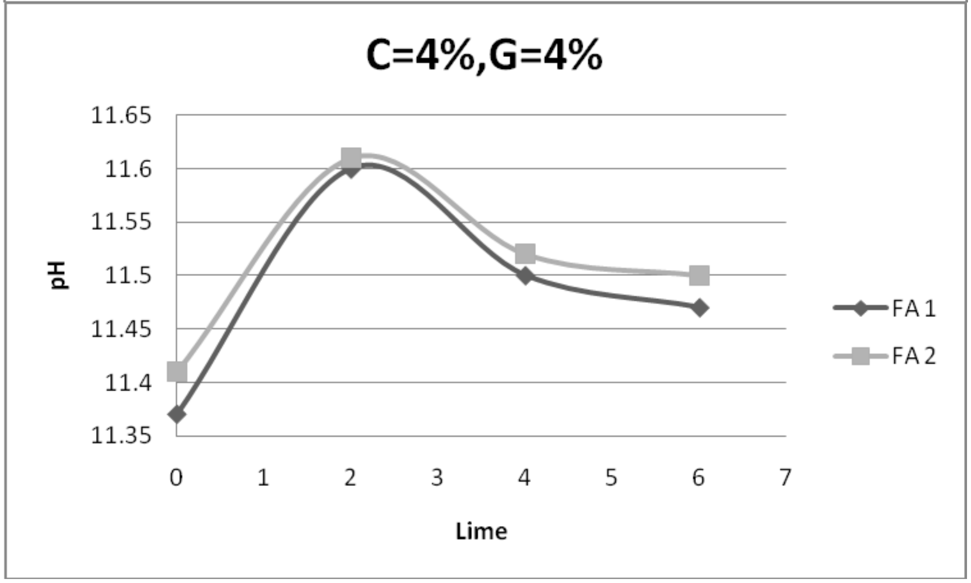
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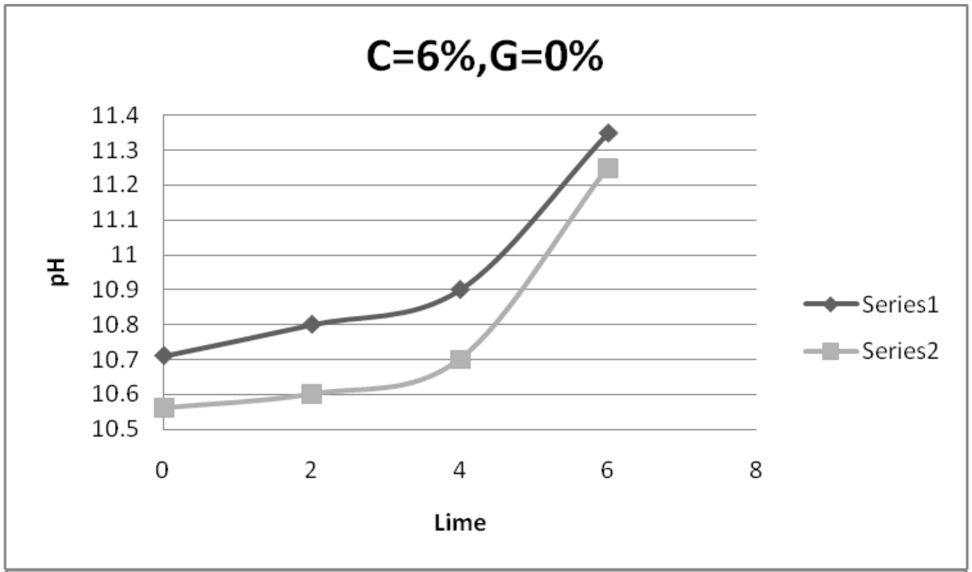
FA 1
FA 2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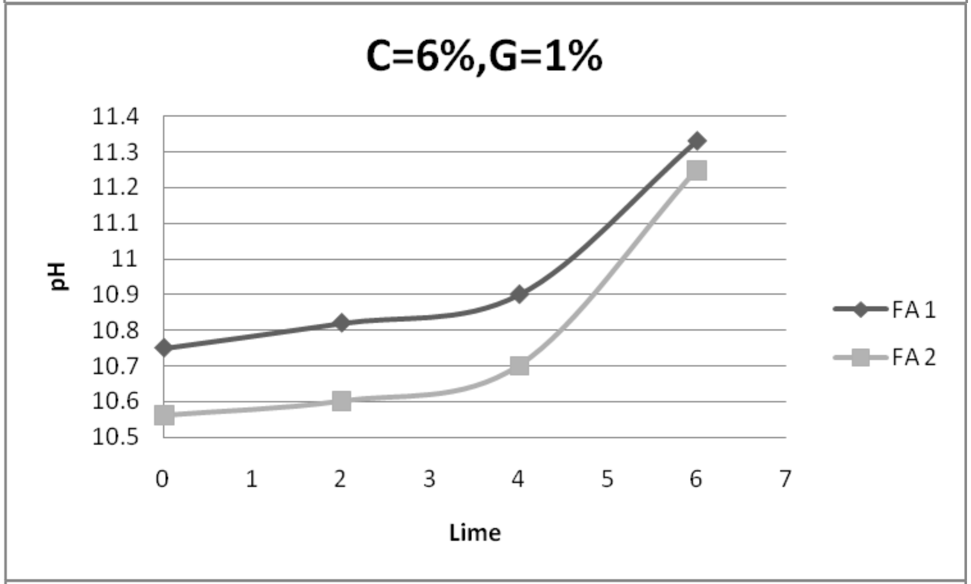
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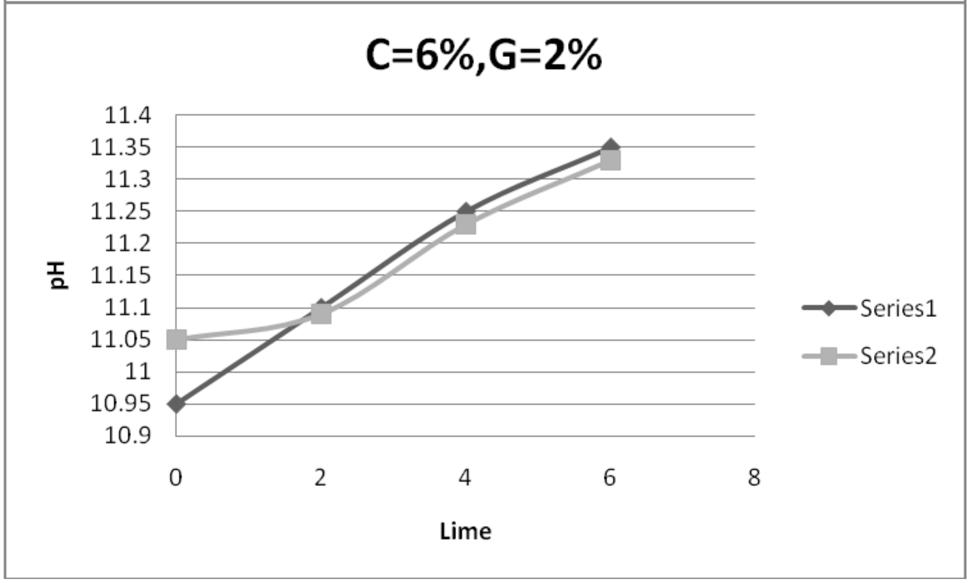
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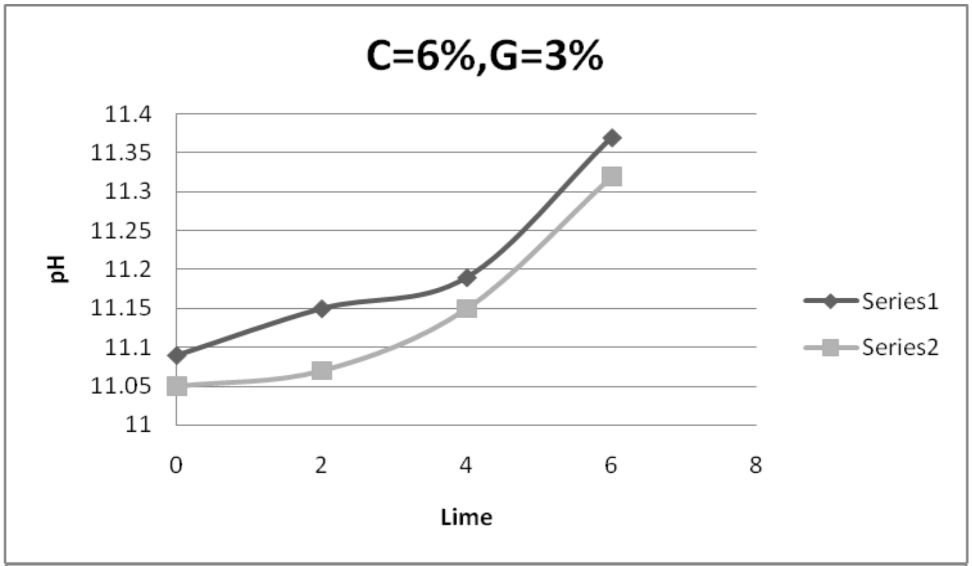
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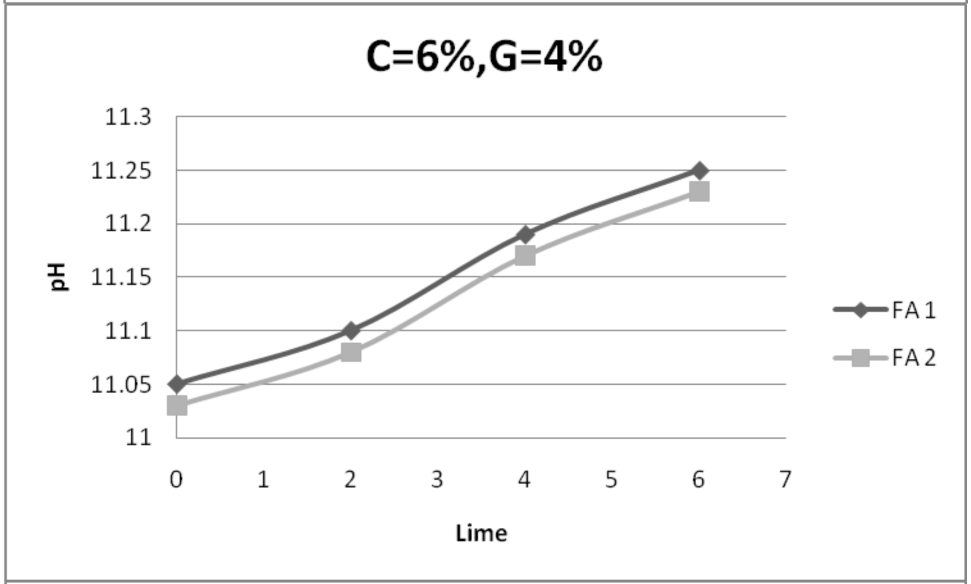
8
Lime
Series1
Series2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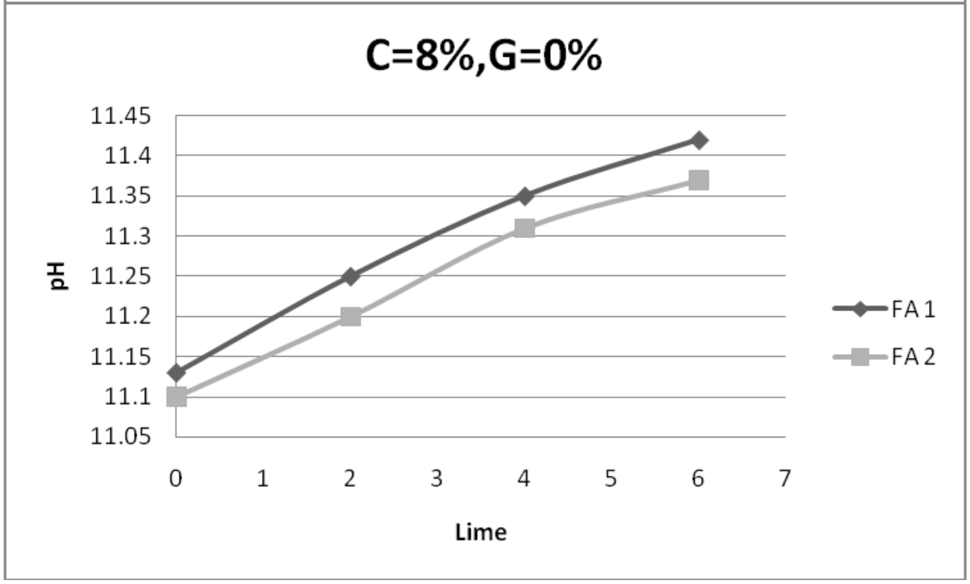
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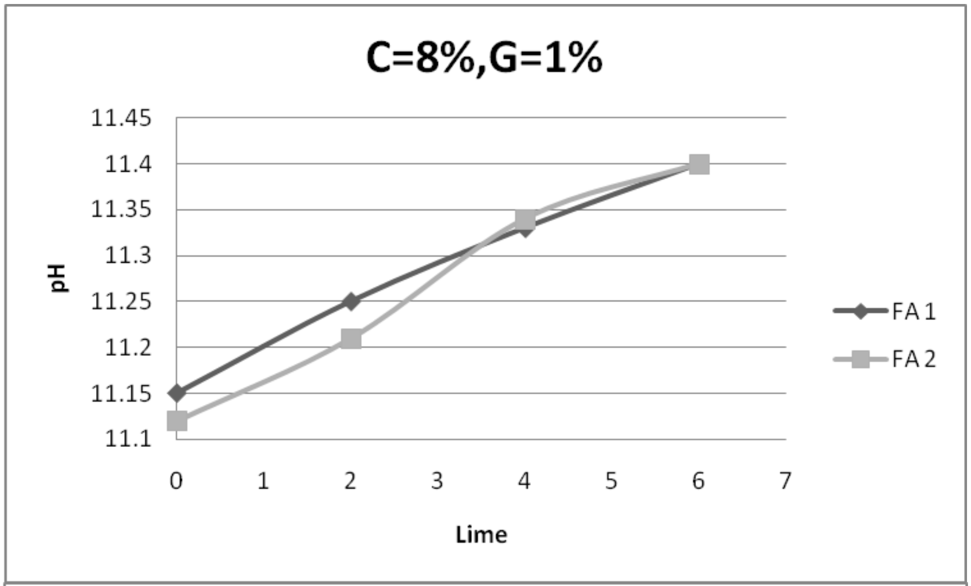
FA 1
FA 2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Lime
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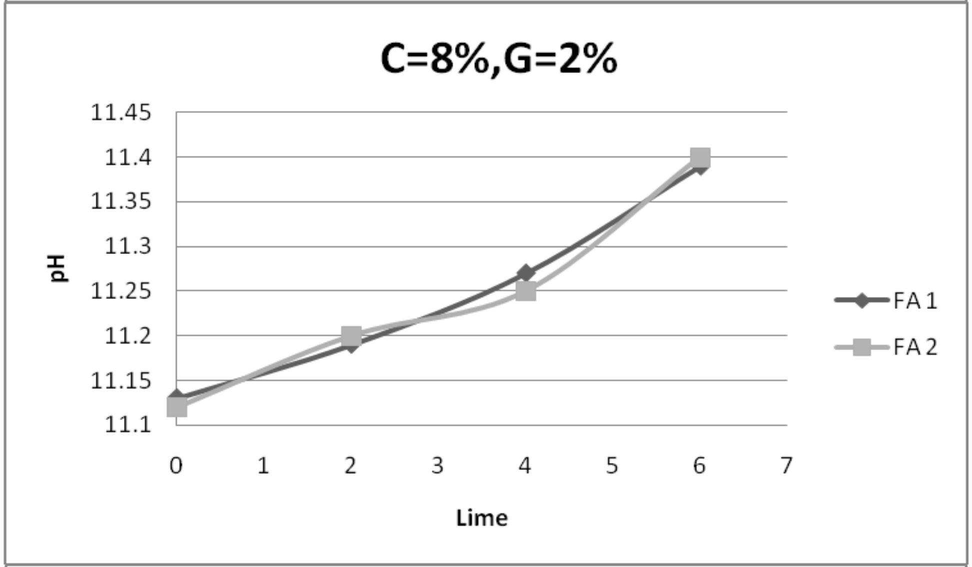
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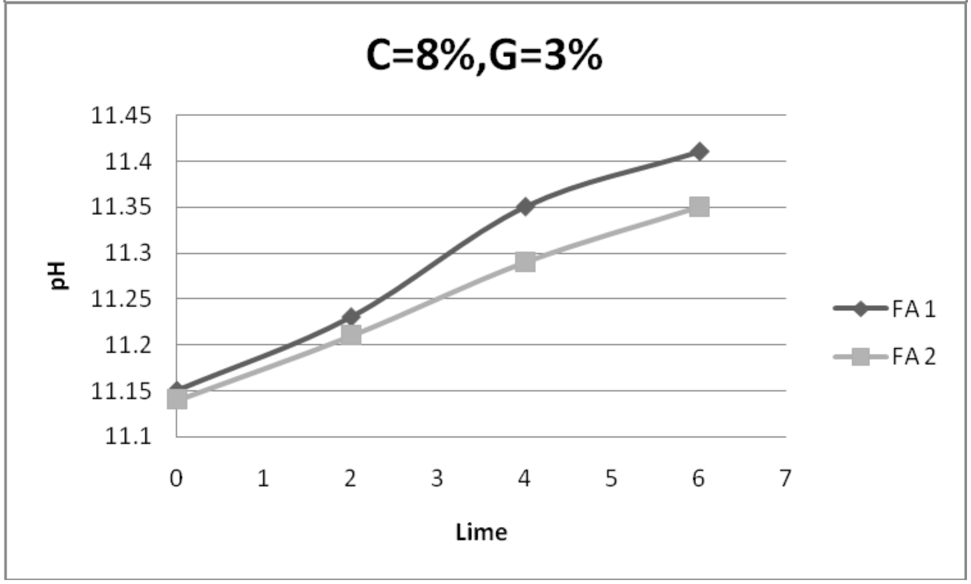
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FA 1
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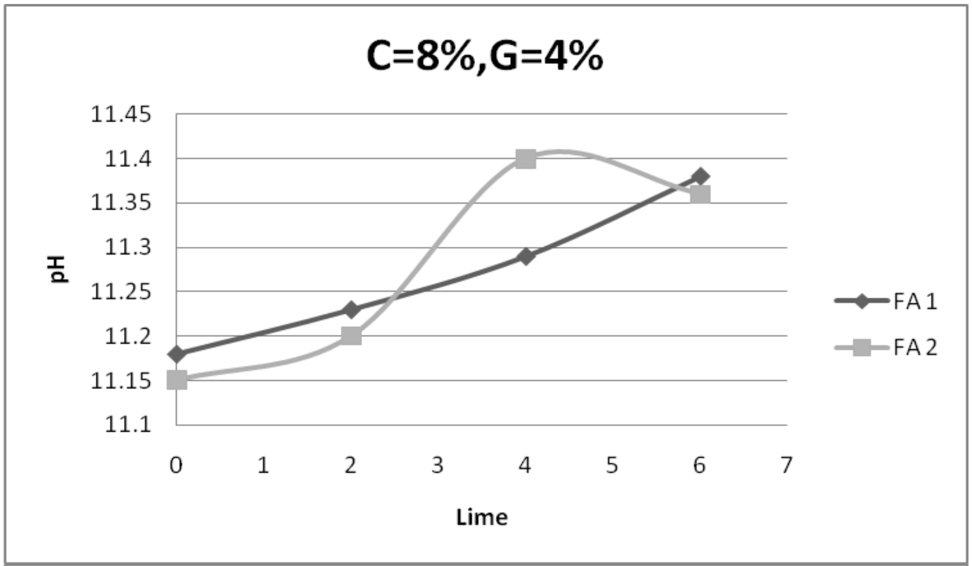
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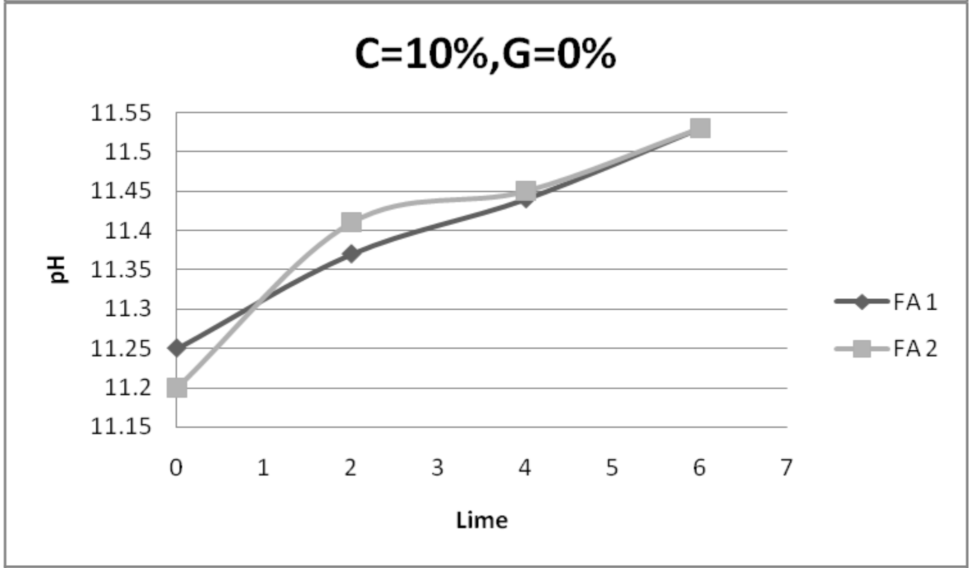
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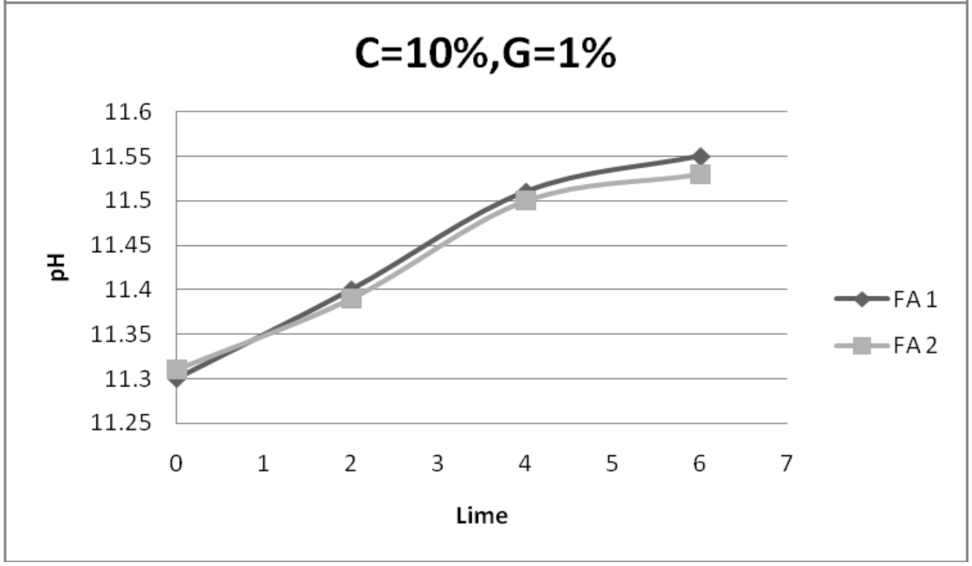
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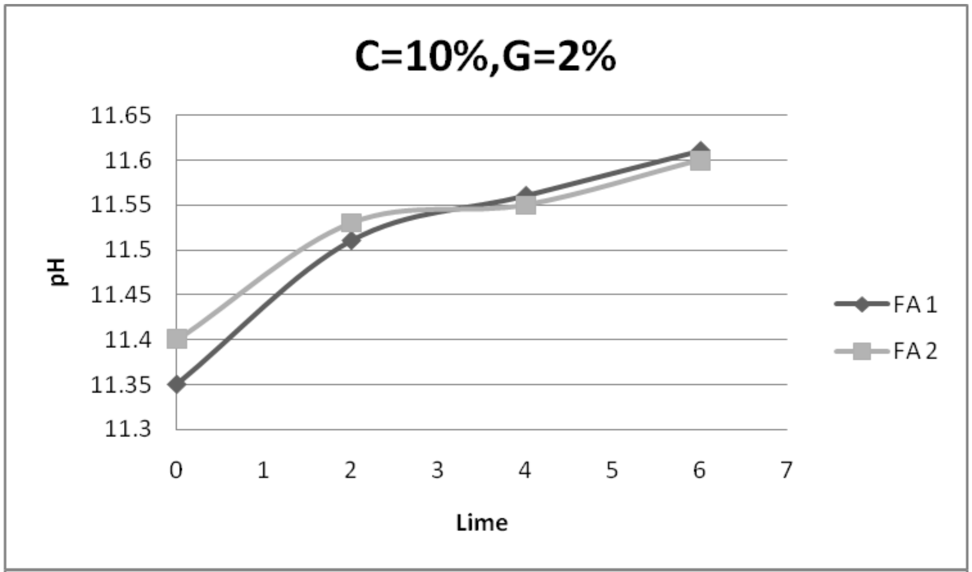
FA 2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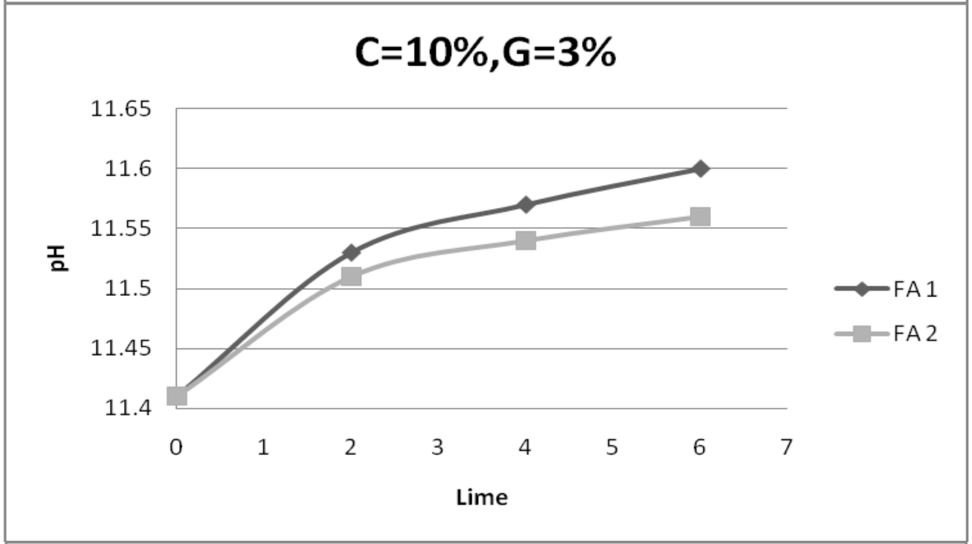
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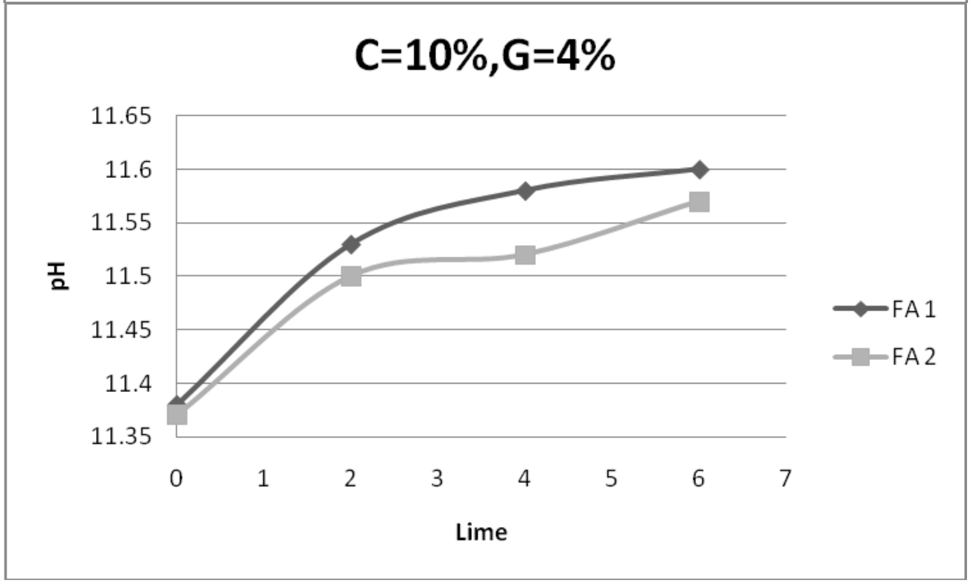
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6
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Lime
FA 1
FA 2

5.4 SEM – FLY ASH CHARACTERIZATION

5.4.1 RESULTS

In addition to the general physical characteristics and elemental composition of fly ash particles, the SEM data indicated intermixing of Fe and Al-Si mineral phases and the predominance of Ca non-silicate minerals .These results supported data obtained from pH studies and were consistent with XRD data.

Fig. 5.4.1.1 Magnified image of PTPS Fly ash

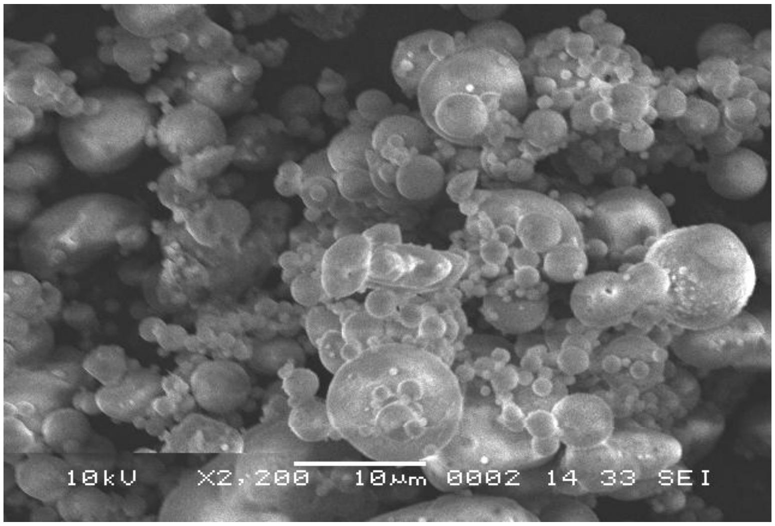
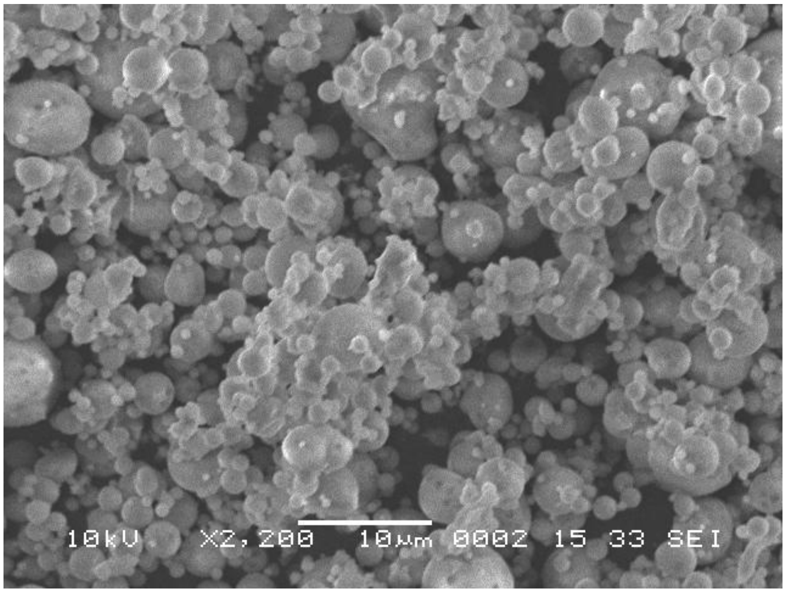


Fig.5.4.1.2 Magnifies Image of ETPS fly ash



5.4.2 MORPHOLOGY (PHYSICAL)

The morphology of a fly ash particle is controlled by combustion temperature and cooling rate. Hollow cenospheres and irregularly shaped unburned carbon particles tended to be in the upper end of the size distribution. Agglomerated particles and irregularly shaped amorphous particles are present due to inter-particle contact or rapid cooling.

5.4.3 ELEMENTAL COMPOSITION

As determined by SEM, the predominant elements in the Fly ash samples were silicon, aluminum, iron, calcium and oxygen in various compounds. Aluminum was primarily associated with silicon. Lesser amounts of the elements Potassium, magnesium, sodium, titanium, and sulfur were observed with the aluminum and silicon. Calcium was observed primarily with sulfur; it was not observed associated with silicon in any of the samples.

5.4.4 DISCUSSION

The chemical and physical properties of fly ash particles are a function of the mineral matter in the coal, the combustion conditions, and post-combustion cooling. During the combustion process, the heat causes the inorganic mineral to become fluid or volatile or to react with oxygen. During cooling, it may form crystalline solids, spherical amorphous particles or condense as coatings on particles.
The morphology and elemental data indicated that the fly ash samples were composed of amorphous alumino-silicate spheres and a lesser amount of iron-rich spheres.

The elemental spectra of the black spots suggesting that they are holes within the sphere. Many of the iron spheres consisted of iron oxide mixed with amorphous alumino-silicate.

In polished cross-section, it is seen that the iron oxide is not a surface phenomenon, but is an integral part of the solid particle.

The mixed iron/alumino-silicate content of the fly ash grains varies in intensity and texture. The iron oxide exhibits various textures both on the surface and interior of the particles.

The SEM observations were also consistent with X-ray diffraction (XRD) data on selected samples. The XRD data indicate that majority of the material in each of the samples analyzed was amorphous. The iron-rich phases identified by XRD are magnetite and hematite, which is consistent with previous studies of mixed iron oxide/alumino-silicate spheres. The XRD analysis also found clay and quartz at low levels.

The SEM data showed calcium associated with sulfur or phosphorus in distinct particles, not with the alumino-silicate particles in these fly ashes.

The XRD analysis identified the calcium compounds lime, anhydrite and gypsum. Volatile elements which enter the vapor phase during combustion are expected to be seen as distinct coatings on the surface of fly ash particles.

However, SEM examination of the fly ash samples showed no evidence of coatings on any of these fly ash particles.

5.4.5 CONCLUSIONS

The fly ash samples consisted mostly 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.

This mixing of phases is consistent throughout the internal structure of the fly ash particles, not just a surface phenomenon. The amount of mixing varies with each fly ash particle, and several internal and surface textures are identified.

The calcium-rich material was distinct in both elemental composition and texture from the amorphous alumino-silicate spheres. It is a non-silicate mineral like calcite, lime, gypsum or anhydrite.

In spite of the inherent variability of fly ash samples, the analysis indicated that the primary mineral/morphological structures are mostly common.

Quartz and alumino-silicates are found as crystals and as amorphous particles. Iron-rich particles typically exist as mixed iron oxide/alumino-silicate particles. Calcium is associated with sulfur or phosphorus, not with the alumino -silicates.

5.5 X-RAY DIFFRACTION

5.5.1 MORPHOLOGY AND MINERALOGICAL COMPOSITION

One of the most importance techniques for characterizing materials like fly ash is the X-ray diffraction (XRD). XRD can be used to identify the crystalline components provided a material contains crystal. The XRD pattern is like a crystal on which quantitive analysis can be done for the crystalline fraction of material. XRD patterns are much easier to interpret because of the development of the well designed software. Each sample is examined by scanning electron microscopy, in order to investigate the morphology of the particles. A small quantity of the samples is vaccum coated with a layer of carbon to ensure adequate conductivity. Scanning electron microscope micrograph is obtained by SEM-501 and images are shown. These images correspond to the particles of size 100, 50 and 20 um at resolution of 100,300 and 600 times.

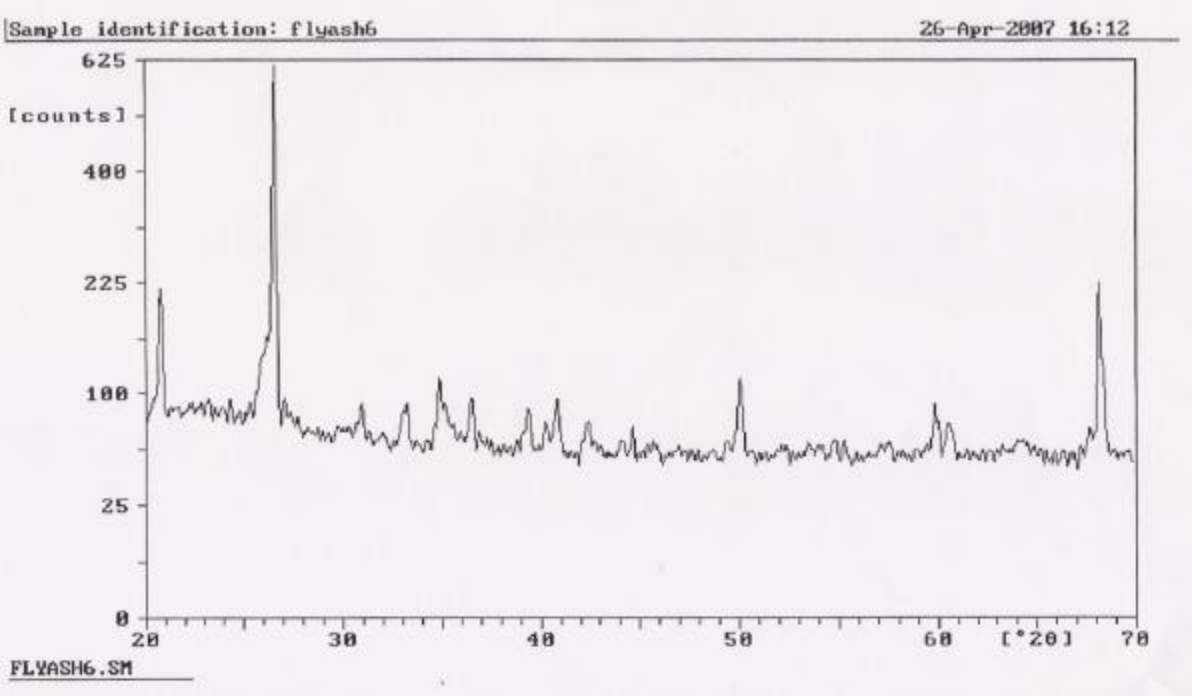


Fig 5.5.1.1 XRD Graph of PTPS fly ash

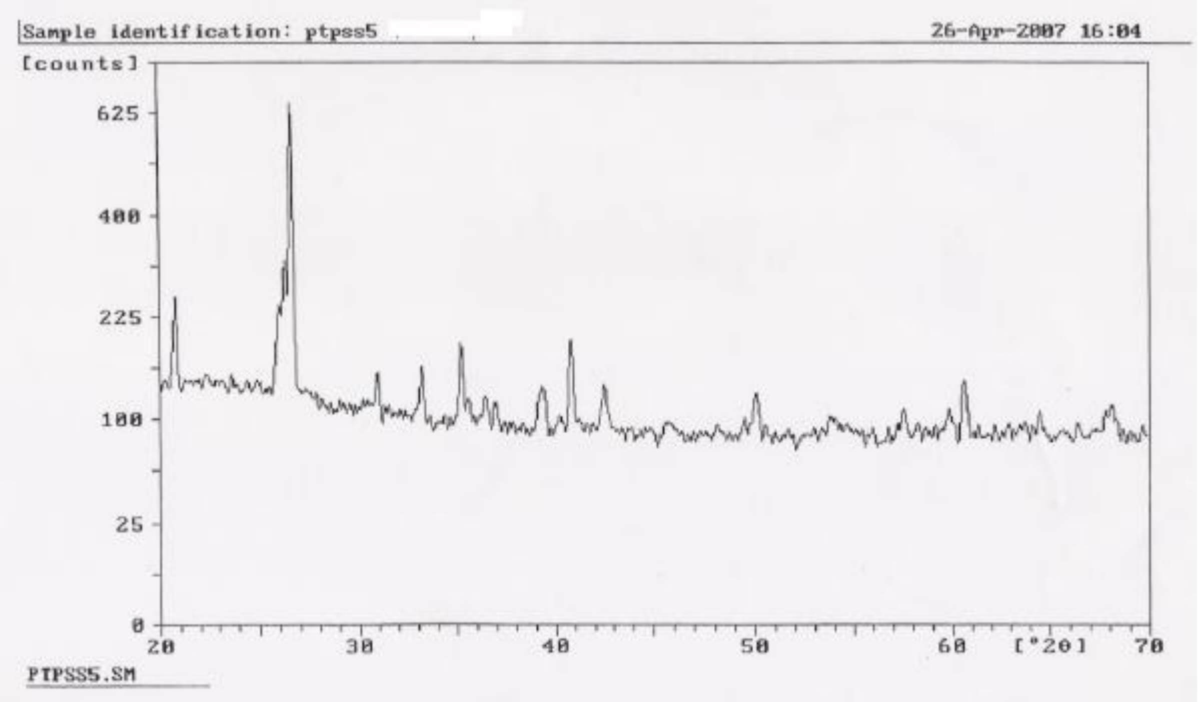


Fig 5.5.1.2 XRD Graph of ETPS fly ash

5.6 COMPRESSIVE STRENGTH

5.6.1 RESULTS AND DISCUSSION

Compressive strength data for the core specimen containing fly ash obtained from Patratu Thermal Power Plant fly ash and Ennore thermal power plant fly ash is given in Table.

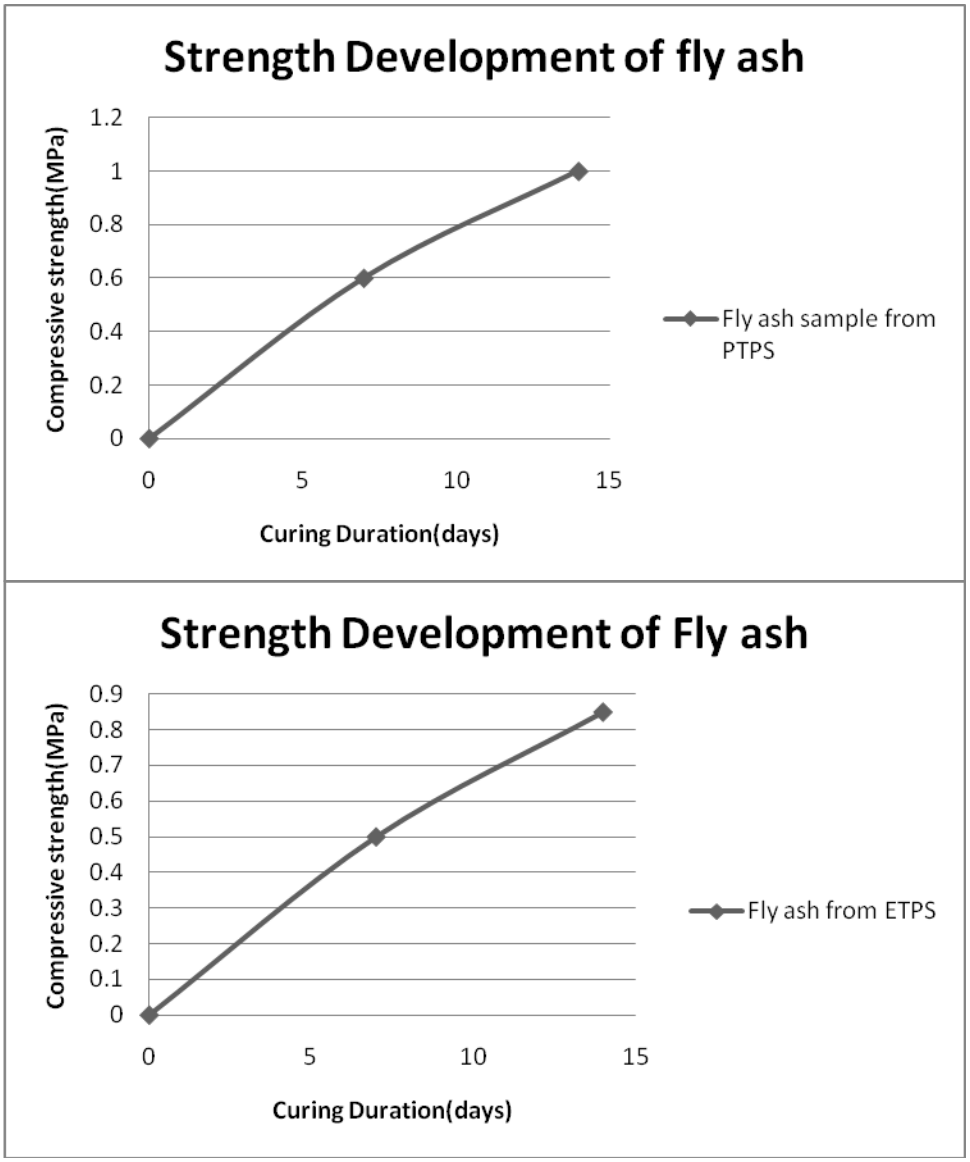
PARTICULARS	CURING DAYS	Compressive strength (MPa)	
		Fly Ash from PTPS	Fly ash from ETPS
SAMPLE NO.1	7	0.6	0.5
SAMPLE NO.2	14	1	0.85

The results compressive strength decreases when fly ash quantity was increased from certain proportions of the cement used by weight. The sample mix, containing fly ash with other mixing proportions showed compressive strengths of 0.6 MPa at the 7-day age and 1 MPa at the 14-day age of curing.

Thus, these results indicate that concrete containing low-calcium fly ash can be proportioned to meet the requirements of strength and workability for structural grade concretes. The desired strength can be achieved by adjusting the amount of fly ash, lime, gypsum and cement for the same water to cementitious ratio to achieve the desired strength and workability.

From the investigation of experiment it showed that at high fly ash replacement levels, the proportion of fly ash concrete strength to reference concrete strength increased substantially from 7 day to 14 days of curing.

Fig 5.6.1 Compressive strength versus age relationship



In case, additional curing time cannot be allowed; the mixture proportion can be adjusted to meet the job requirements in order to reach sufficient strength for stripping within a given length of time.

5.6.2 SUMMARY AND CONCLUSION

This study was directed toward evaluation of performance of sample specimen incorporating fly ash in addition to proportion of lime, gypsum and cement also. Sample mixes; contains 1 kg of fly ashes is proportioned to had cement replacement of 10 % cement, 6% of lime and 4% of gypsum by weight of fly ash sample. The water to cementitious materials ratio was maintained at approximately 0.3, and the desired workability of sample mixes was obtained.

In general, compressive strength increased with age, and decreased with increasing fly ash inclusions in the tested range of variables. However, the specimen containing fly ash in addition to lime, gypsum and cement is developed to gain maximum compressive strength with age. At an early 7-day age, sample specimen showed a high early compressive strength which is suitable for use in structural concrete.

Based upon data recorded, it can be concluded that specimen containing fly ash with appropriate proportion of certain additives can be proportioned to meet the strength and workability requirement for structural grade concretes.

The chemical, physical and mineralogical properties of fly ash had appreciable effects on performance of fly ash in concrete. Properties of cement influenced the performance of concrete. Therefore, it is necessary to determine the optimum mixture proportions for each cement, lime, gypsum and fly ash source before use.

Chapter 6

Conclusion

6. CONCLUSION

After conducting all the experiment related to strength development of fly ash based composite materials the following are the factors affecting Strength Gain of Lime-Cement-Gypsum-Fly ash composite material :

6.1 FLY ASH CHARACTERISTICS

- Fly ash type (classification, particle size distribution, etc.)
- Fly ash chemistry (pH, cation exchange capacity, etc.)
- Fly ash mineralogy (silica content, presence of gypsum, etc.)
- Moisture content
- Organics (type, amount, degree of decomposition, origin)

6.2 MIXING

- Mixing device
- Mixing time
- Types of stabilization agent/agents
- Proportions of stabilizer
- Time restraints during mixing and compacting

6.3 COMPACTING

- Packing pressure
- Packing tool / method
- Sample size (mold size)

6.4 CURING

- Curing time
- Curing temperature
- Curing humidity
- Freeze/thaw cycles
- Applied load

Strength test

- Sample extraction
- Type of test
- Loading rate
- Cyclic loading

6.5 GENERAL FINDINGS

- For 100% cement mixes, using the water/cement proportion ratio provides a simple and effective way to estimate the amount of cement required to attain a specified compressive strength.
- For a given lime-cement proportion, as dose rate increases, strength increases for sample specimen tested.
- Lime appears to have little impact on the fly ash, but the strength of the fly ash composite appears to be very sensitive to the addition of lime.

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