

**POZZOLANICITY TEST FOR POZZOLANIC CEMENT**

**BY**

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## DECLARATION

I certify to the best of my knowledge that this dissertation does not contain any material previously published or written by another person except where due references are made in the text.

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**AFFECTIONALLY DEDICATED TO MY PARENTS**

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## LIST OF ABBREVIATIONS

GCCL	- Galle Cement Company Limited
LQL	- Lanka Quarries Limited
PCCL	- Puttalam Cement Company Limited
LSF	- Lime Saturation Factor
SHL	- shale
Q.C	-quarry clay
A.C	- achchimale clay
ASTM	-American standard test method
BS	- British standard
CaO	- Lime
SiO <sub>2</sub>	- Silica
Al <sub>2</sub> O <sub>3</sub>	- Alumina
Fe <sub>2</sub> O <sub>3</sub>	- Iron oxide
MgO	- Magnesia
SO <sub>3</sub>	- Sulphur Trioxide
CA	- Calcium Silicate
C <sub>3</sub> S	- Tricalcium silicate
C <sub>2</sub> S	- Dicalcium Silicate
C <sub>3</sub> A	- Tricalcium aluminate
C <sub>4</sub> AF	- Tetracalcium Aluminoferrite
SR	- Silica ratio

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## **CHAPTER 1**

### **1. INTRODUCTION AND OBJECTIVES**

#### **1.1. INTRODUCTION**

##### **1.1.1 BACKGROUND**

Pozzolanas are usually defined as materials which, though not cementitious in themselves, contain constituents which will combine with lime at ordinary temperatures in the presence of water to form stable insoluble compounds possessing cementing properties. The pozzolanas can be divided into two groups, natural and artificial.

The natural pozzolanas are for the most part materials of volcanic origin, but include certain diatomaceous earths. The artificial pozzolanas are mainly obtained by the heat treatment of natural materials such as clays and shales and certain silicious rocks, and pulverised fly ash.

Ancient Greeks and Romans firstly used Pozzolanas in combination with lime. Structures built more than 2500 years ago by using natural pozzolanas has still remained in very good condition, for example, the roman aqueducts and water storage tank in Greece. Today, Portland cement and pozzolan blends containing either natural or artificial pozzolan have been used extensively all over the world.

Burnt-clay pozzolanas are produced by burning suitable clays or shales at a temperature which varies from 600 °C to over 900 °C, depending of the nature of the clay and the condition of burning. In Portland pozzolan mixtures the amount of Portland cement replaced by pozzolan depends on physical and chemical properties. (Lea. E.M. et al., The Chemistry of cement and concrete, Third edition)

Holcim (Lanka) Ltd still does not produce pozzolanic cement. There are so many advantages in producing pozzolanic cement. It causes to reduce the cost of production and increase the profit of the company by reducing the energy consumption. Throughout my research, test the pozzolanicity mixing various percentages of shale, quarry clay and achchimale clay in to Portland cement in different burning temperatures.

## **1.1.2. COMPANY PROFILE**

### **1.1.2.1. HISTORY OF HOLCIM**

The Puttalam cement production plant was built in 1968 as a government corporation and was later doubled its production capacity in 1971. Then the company was sold as a private enterprise to a foreign investor in 1994. Other shareholders intervened and then the plant was sold to “Holder bank” demonstrating Government corporation culture in a democratic socialist country. Private owners looked only for short-term profits and as a result workforce was demoralized. In August 1996, the Swiss Holder bank”group acquired a majority shareholding of Puttalam Cement Company Limited and assumed management control. Subsequently, nearly Rs. 2 billion was invested at the Puttalam Cement plant upgrading production facilities and improving environmental performance.

In early 1999 Ruhunu Cement Company Limited was acquired and combined with Puttalam Cement Company Limited to form the PCCL group. Further additions of the group name with the commissioning of GCCL and LQL.

The PCCL group now operates as a single entity supplying cement to every part of Sri Lanka. Whilst the new aggregates operation services the Colombo and Galle markets and points in-between.

The “PCCL group” currently employs over 1200 people and has an acute awareness of our social responsibility, thus highlighting our continued industry stewardship.

The brand name of the cement is “Holcim” and the trade mark is



Holcim is the only premium cement available in Sri Lanka that is made from local raw materials. It is assured by both local and international quality standards (ISO 9002 certified).

### **1.1.2.2. HOLCIM IN THE WORLD**

Holcim is one of the world's leading suppliers of cement, as well as aggregates (gravel and sand), concrete and construction-related services. From its origins in Switzerland, the Group has grown into a global player with strong market presence in over 70 countries on all continents. The Group employs more than 48,000 people.

### **1.1.2.3. HOLCIM IN SRI LANKA: [HOLCIM (LANKA) LTD]**

Holcim (Lanka) Ltd, formerly Puttalam Cement Company Limited is a part of the global Holcim group and is the predominant and preferred supplier of cement in Sri Lanka. Holcim (Lanka) Ltd is the leader in cement industry of Sri Lanka and plays an important part in the economic development of our country.

Holcim (Lanka) Ltd has one fully integrated cement plant in Palavi, Puttalam, one grinding unit in Galle and one packing unit in Kurunegala located in different areas of the country. At present, 945 employees are working in Holcim (Lanka) Ltd. Approximately; they generate annual revenues of 60 million USD.

Holcim (Lanka) Ltd manufactures about 1.0 million metric tones of cement annually. The Holcim Cement range, being the market leader, meets customer requirements in cements, with the sub-brands

- Holcim Supiri
- Holcim Pedereru
- Holcim Seegra
- Extra Durable Cement.

These products are made at Palavi and Galle plants, which are ISO 9000 quality system certified. Holcim (Lanka) Ltd.

## **1.2. OBJECTIVES**

### **1.2.1. PRIMARY OBJECTIVE**

The Pozzolanic cement manufacturing process is a cost effective method than Portland cement. Now there is a trend to produce the Pozzolanic Cement in worldwide. Holcim Lanka (pvt) Ltd is also trying to produce that cement. Therefor, in my research, determined the Pozzolanicity of Pozzolanic cement using shale, quarry clay and achchimale clay.

### **1.2.2. SPECIFIC OBJECTIVES**

- Determination the compressive strength of the cement
- Determination the Fineness of the cement

## **CHAPTER 2**

### **2. REVIEW OF LITERATURE**

#### **2.1. CEMENT**

##### **2.1.1. SCINTIFIC DEFINITION OF CEMENT**

Cement is a hydraulic binder. That is a finely ground inorganic material which, when mixed with water, forms a paste which sets and hardens by means of hydration reactions and after hardening it retains its strength and stability even under water. ( Lea. E.M.et al., The Chemistry of cement and concrete, Third edition, page 417)

##### **2.1.2. PORTLAND CEMENT**

John Smeaton, in 1776, first recognized that lime prepared by heating limestone containing clay gave satisfactory mortars. William aspdin, a mason by profession, belonging to leads in England, is accredited with the invention of Portland cement. Guided by the idea that clay must be heated to produce a material similar to volcanic ash, he ground a mixture of calcined limestone and clay with water to make slurry and heated the partly dried slurry in lime kilns. The product was crushed and finely ground to a powder, on mixing with water, set to give a hard, stone like mass. Its colour and appearance resembled that of stones quarried from the isle of Portland on the English coast, and so aspdin named his product Portland cement. (R.Gopalan.et al., Engineering Chemistry, page 10.2)

##### **2.1.3. POZZOLANIC CEMENT**

This is the most ancient among the types of manufactured cements. It was first made from volcanic ash from mount visuvius situated around a place called pozzuoli in Italy. Volcanic ash consisting of silicates of calcium, iron and aluminium, when mixed with lime and heated produces pozzolanic cement.

## 2.2. CEMENT INDUSTRY

### 2.2.1. MANUFACTURING PROCESS

The major raw materials required for the manufacture of Portland cement are,

- Calcareous materials (limestone, chalk, marble)
- Argillaceous materials (silicate containing materials such as clay)
- Gypsum

The fusion of the above materials, taken in proper proportions, produces the Portland cement. The actual manufacturing involves the following operations. (R.Gopalan. et al., Engineering Chemistry, page 10.3-10.4)

- Mixing
- Burning
- Grinding
- Packing

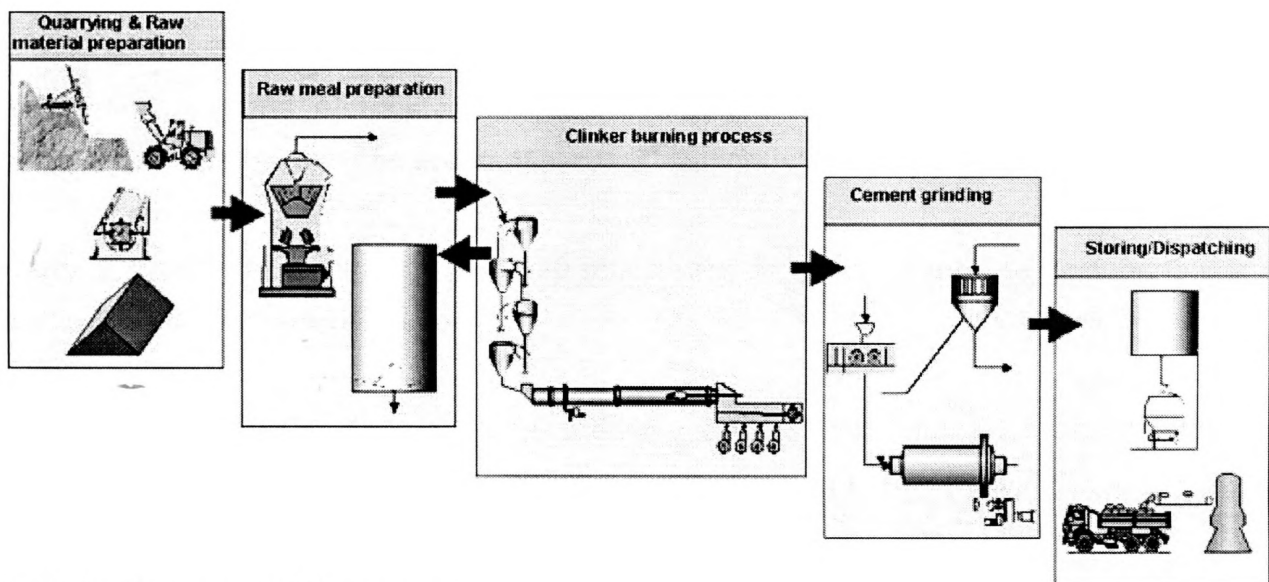


Fig.2.1. Process of the cement

The process of the cement preparation shown in the Fig.2.1, describe in following text.

## 2.2.2. MIXING

### 2.2.2.1. QUARRYING



Fig 2.2. Profile of a Quarry

Quarrying is the vital part and the root of any cement industry. Quarrying describes the surface mining of rock where as 'open pit' mining describes the surface mining of minerals. In this quarry open pit mining is done. Quarrying is the breaking of the rock in safe and economical way and transporting the result to the plant for further process.

Quarrying involves planning, purchasing/leasing of suitable equipment, drilling, blasting, loading and transport of rock in quantities sufficient to permit continuous operation of the cement plant. All this must be accomplished as efficiently and safely as possible to maximize the return on investment.

Quarry is situated at Aruwakalu about 40 miles away from the factory. Limestone thus quarried then transported by railway wagons.

At the quarry up to 25m depth earth has red salty sand, which should be evacuated before reach the limestone bed. This is the high cost operation, which has indirect advantages. Limestone bed has sandy limestone yellowish- whitish up to 5.6 m which is the low quality limestone.

After that the most wanted high quality limestone layer of abut 22 m depth is present but it varies from place to place.



#### **2.2.2.2. GEOLOGICAL FEATURES OF THE LIMESTONE BEDS**

There are many rock types existing in the Earth's crust and all are due to geological process. At our quarry high grade limestone of reefal origin and low quality limestone rich in siliciclastic material.

#### **2.2.2.3. DRILLING**

Bench drilling is a term for designating the method where surface holes are drilled for blasting towards a free surface. Larger hole diameter means that longer holes can be accurately drilled. The position of the drill hole is important if successful breaking to occur. Quarry operations usually use an intermediate diameter drill hole of between 64-65 mm diameters.

##### **2.2.2.3.1. DRILLING PARAMETERS**

Determining the size and number of blast holes as well as the drill required to drill them is based on many different parameters like Capital, nature of the rock, required fragmentation size distribution, monthly tonnage required, vibration and air blast.

#### **2.2.2.4. BLASTING**

It is an intermediate step in the process of quarrying. Choosing the correct explosives is one of the most important decisions influencing the design and operation of quarrying. The objective of a blast is suitably fragmentation of the rock. It is also noted that it cannot be repeated if once incorrectly blasted. So it is obvious that the planning is very important here. The greater the effort put into the planning and preparation the better the results will be. The selection of the correct and economically viable explosives is also very important. (Lea, F.M. et al., The Chemistry of cement and concrete, Third edition, pages 430-434)

### **2.2.2.5. TRANSPORTATION OF RAW MATERIALS TO THE FACTORY**

Having blasted the material then the high and low qualities mixed roughly according to the target and transported to the factory by the railway wagons. In one trip about 25 wagons each having 22 tons of material is transported. Normally five trips are made for a day. Then the transported raw materials are unloaded using the tippler, where they are sent to the crusher afterwards.

### **2.2.2.6. PROCESS OF THE RAW MILL**

The raw mill feed meets the hot waste gas (8000-12000 m<sup>3</sup> / hr) at the mill inlet. The grinded material is lifted by the gas stream to a certain height where the bag house filter is fixed. Coarse particle which is separated by the bag house filter is then re-introduced to the mill while the fine particle which is in the required size range is sent to twin cyclone where the air and the material are separated. The material separated in such a way is directly directed to kiln feed pump. The dust collected in the bag house filter is also sent with the material to the kiln feed pump. The fluidized material at the kiln feed pump is directed to blending silos. The minimum air speed inside the mill should be 6 m/s. A pipe is used to maintain this speed. During the dry season the amount of hot gas required to dry the feed is less as the material contains less moisture. The lack volume of the swept air is recycled by a pipe. Also another pipe is used to balance the line. ( Lea,. F.M.et al., The Chemistry of cement and concrete, Third edition, pages 436-438)

#### **2.2.2.6.1. PROCESS MONITORING AT THE RAW MILL**

Every hour the temperature of the following are measured.

- Raw mill outlet bearing temperature
- Counter shaft bearing temperature
- Inlet bearing temperatures

There are two limits for these temperatures called the 'Alarming temperature' and the 'Tripping temperature'. If there is any abnormality in these temperatures immediate action should be taken to control it to avoid jamming of the mill.

Here the kiln feed final preparation is the main operation. Mainly the Lime Saturation Factor (LSF) of  $99 \pm 2$ , Silica ratio of  $2.4 \pm 0.2$  is reached by purposely adding the ground material.

### **2.2.3. BURNING**

The dry powder or wet slurry, depending on the procedure used in the first step, is then burnt in a rotary kiln. The rotary kiln used for cement manufacture is a long steel cylinder, the inside surface of which is lined with fire brick refractory. The length of the kiln commonly ranges from 30 to 160 metres and the diameter from 2 to 4 metres. The rotary kiln is slightly inclined downwards towards the exit end. It is mounted on rollers and can be rotated at desired speeds, say one revolution per minute. The charge is introduced from the upper end of the kiln. As the kiln is rotated, the charge moves down. It is heated from the lower end using solid, liquid or gaseous fuel. A huge flame is driven from this end and here temperature is about 1500 C. The charge which is fed from upper cooler end gradually travels down and meets the hot gases moving in the opposite direction.

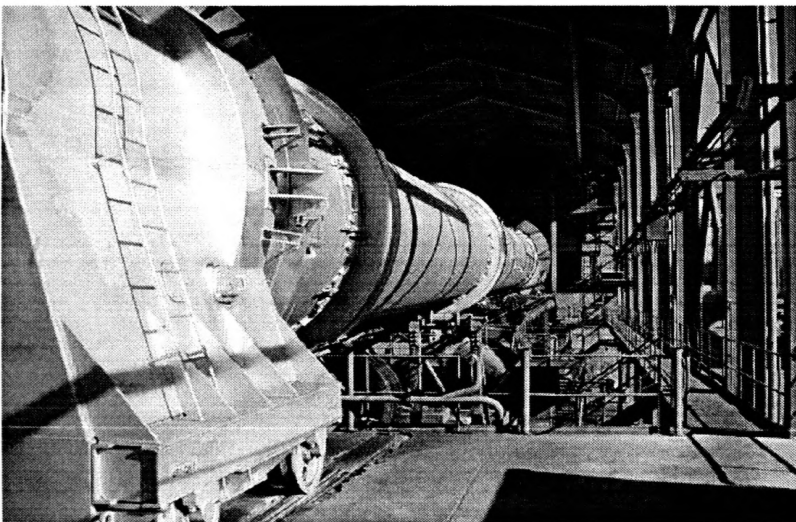


Fig. 2.3. The cement Kiln

The upper one –fourth of the length of the kiln is called drying zone. The temperature in this zone is moderate and the moisture is driven out here and materials get heated. The middle portion of the kiln is known as calcining zone and the temperature here about 900 °C. Carbon dioxide is expelled from limestone in this heating zone. The bottom portion of the kiln is the hottest where the temperature range is 1400-1500 °C. This is called the burning zone and the mixture melts and forms little rounded pasty masses about the size of peas which are called clinkers. The clinker falls out from the lower end of the kiln in to clinker coolers where it cooled by the atmospheric air which passes over it. The air thus becomes hot and is used for blowing the lower end. (R. Gopalan et al., Engineering Chemistry, page 10.4)

Table 2.1 Reaction taking place in the cement kiln (R.Gopalan.et.al.,Engineering Chemistry,page 10)

TEMPERATURE(°C)	REACTION	COMMENTS
>100	Evaporation of water	
>500	Removal of combined water from clay	
>800	Decomposition of CaCO <sub>3</sub> ,MgCO <sub>3</sub> .Formation of CA, C <sub>2</sub> F and C <sub>2</sub> S begins	
800 -900	Cl <sub>2</sub> A <sub>7</sub> Forms	Decomposes to C <sub>3</sub> A at higher temperatures
900 -1100	Decomposition of CaCO <sub>3</sub> completed.	
1100 -1200	C <sub>2</sub> S maximum. C <sub>3</sub> A and C <sub>4</sub> AF form	
1250 -1280	Formation of liquid phase	C <sub>3</sub> A, C <sub>4</sub> AF, A, F dissolve and recrystallise on cooling
1250 -1450	C <sub>3</sub> S, Free-lime content decreases	

#### **2.2.4. GRINDING**

The cooled clinker is discharge and then passed on to tube mills where it is ground to the requisite fineness according to the class of product. Finely ground clinker sets very rapidly by absorption of moisture from the atmosphere; a retarding agent like gypsym is added to the extent of about 3.5% by weight to control the process. The mixture of clinker and gypsym powder is known as cement.

#### **2.2.5. PACKING**

Cement is transferred from the storage silos to the packing plant for bag and bulk loading. Bagging is done by high speed in line and rotary packing machines. Filled bags are automatically weighed, sealed and loaded onto trucks by conveyor belt. The total combined rated capacity of all Inducement's packing machines is 5,000 tons per hour. Bulk cement is loaded into special tank trucks for delivery to customers' portable silos at construction sites or is trucked to Inducement's port facilities.

#### **2.2.6. QUALITY CONTROL**

The production process at each of plant is monitored by the particular plant's individual and centralized control room where computerized equipment is used to monitor the entire process from raw materials at storage to grinding of cement at the finish mill.

Cement quality checks are carried out continuously. To ensure the production of consistently high quality cement, a modern system of automatic samplers, automatic X-ray analyzers and process computers perform on-line control on the proportioning of raw materials to maintain consistent chemical composition of the cement produced.(Mehta, P.K., "Pozzolanic and Cementitious By-Product in concrete-Another Look", Silica Fume, Slag and Natural Pozzolan in Concrete Proc. 3rd International Conference, 1989, Trondheim, Norway.)

## 2.3. CHEMISTRY OF PORTLAND CEMENT

The major constituents of cement are lime, silica, alumina, iron oxide, magnesium oxide, sulphur trioxide, sodium oxide, and potassium oxide.

Table 2.2 Composition range of portland cement

(R. Gopalan et. al., Engineering Chemistry, page 10.7)

OXIDES	FORMULA	PER CENT LIMIT
Lime	CaO	60 - 67
Silica	SiO <sub>2</sub>	17 - 25
Alumina	Al <sub>2</sub> O <sub>3</sub>	3 - 8
Iron oxide	Fe <sub>2</sub> O <sub>3</sub>	0.5 - 0.6
Magnesia	MgO	0.1 - 0.4
Sulphur trioxide	SO <sub>3</sub>	1 - 2
Soda and potash	Na <sub>2</sub> O + K <sub>2</sub> O	0.5 - 1.3

Although Portland cement is being used for a very long time. Its detailed chemistry is complicated and not yet fully understood. The important constituents and the properties of cement in terms of its setting and hardening properties will be briefly discussed below.

### 2.3.1. SETTING AND HARDENING

When cement is mixed with water, various hydration and hydrolysis reactions of the compound take place. The compounds thus formed are responsible for setting, so solidification and gradual hardening. The process of solidification consists of three steps.

- Initial setting
- Final setting
- hardening

### 2.3.2. INITIAL AND FINAL SETTING

When Portland cement is mixed with about half its weight of water, plastic cement pastes results. This paste gradually loses plasticity and finally becomes stiff and hard. In this setting process, two stages, namely Initial setting and Final setting, are recognized-based on the ability of a weighted needle-which penetrates the mass under prescribed set of conditions. The initial set takes place when the needle does not penetrate the mass at all. The times taken for these two steps are respectively known as initial setting and final setting time.

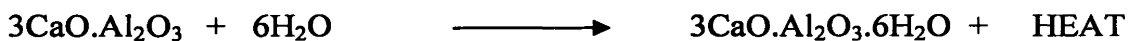
The setting time depends on the composition of cement, temperature and quantity of water used in gauging. For ordinary cements initial setting time should not be less than 45 minutes to allow sufficient time for mixing, transportation and placing between the aggregates. The final setting time should not be more than 10 hours.

### 2.3.3. CHEMICAL REACTION FOR SETTING

#### 2.3.3.1. INITIAL SETTING

The Constituents of cement are anhydrous and, on mixing with water, a number of complex hydration and hydrolysis reactions take place, as given below

Tricalcium aluminate hydrates very rapidly and it is a high exothermic reaction.



The presence of gypsum in the cement helps to retard the speed of the initial set as it reacts with tricalcium aluminate forming calcium sulphoaluminate which does not show any tendency to rapid hydration.



### 2.3.3.2. FINAL SETTING

The reaction of water with tricalcium aluminate is important, as the product of the reaction has a marked effect on the early strength and hardness of the cement. The tricalcium silicate combines with water more slowly and is jellified within a few hours as per reaction shown below.



### 2.3.3.3. HARDENING

Hardening is a relatively slow process. After the final set, the solid begins to gain strength and it is this increase in strength which is referred to as hardening. Hardening or development of strength depends upon the chemical combination of cement and water and will thus continue with great speed at first and go on reducing gradually.

Dicalcium silicate also reacts with water similar to  $3\text{CaO}.\text{SiO}_2$ . But the rate of this reaction is slower and thus is responsible for the progressive increase of strength and hardness.



Because of the much slower rate of reaction, it proceeds for a considerable period of time

.(R.Gopalan et al., Engineering Chemistry, page 10.8-10.10)

## 2.4. MANUFACTURING OF POZZONANIC CEMENT

It is made by grinding some solidification regular with Portland cement clinker and mineral additives (pozzolna) defined at their standardized ratio. It is a product that gains its strength by freezing after it is mixed with water.

Modern pozzolanic cements are a mix of natural or industrial pozzolanas and Portland cement.



Portland cement + pozzolana  $\longrightarrow$  pozzolanic cement

In order to use water, the high alkalinity of pozzolana makes it especially resistant to common forms of corrosion of sulfates. Once fully hardened, the Portland cement – pozzolana blend may be stronger than Portland cement, due to its lower porosity, which also makes it more resistant to water absorption.

## 2.5. POZZOLANAS

Pozzolana is a siliceous and aluminous material which reacts with calcium hydroxide in the presence of water to form compounds possessing cementitious properties at room temperature and that have the ability to set under water. It transformed the possibilities for making concrete structures, although it took the Romans some time to discover its full potential. Typically it was mixed two-to-one with lime just prior to mixing with water. The Roman port at Cosa was built of Pozzolana that was poured underwater, apparently using a long tube to carefully lay it up without allowing sea water to mix with it. The three piers are still visible today, with the underwater portions in generally excellent condition after 2100 years.

pozzolanic ash, is a fine, sandy volcanic ash, originally discovered and dug in Italy at Pozzuoli in the region around Vesuvius, but later at a number of other sites. Vitruvius speaks of four types of pozzolana: black, white, and grey, red. It is found in all the volcanic areas of Italy, such as Naples, in various colours: black, white, grey and red

Pozzolans are used in lime pozzolana mortars, in blended pozzolanic cements, and as direct additions to a concrete mix. In the traditional use of pozzolanas in lime mortars the material was used in a relatively coarse state, as indeed it still is for example, in Italy, where the specification for natural pozzolanas used in building mortars only requires that the maximum size of grains shall not exceed 5 mm. the coarser particles act as more or less inert aggregate and only the finer material, which grades down to a fine dust, as an additive pozzolana. With the development of the use of pozzolanas with Portland cement the practice has become common of grinding them to cement fineness. Thus the Italian pozzolanic cements, the German trass cements, and the pozzolanic cements used in the U.S.A. are underground mixtures of pozzolana and Portland cement clinker. (Mehta, P.K., "Pozzolanic and Cementitious By-Product in concrete-Another

Look”, Silica Fume, Slag and Natural Pozzolan in Concrete Proc. 3rd International Conference, 1989, Trondheim, Norway.)

Lime- pozzolana mortars are used as building mortar in many parts of Europe and enjoy a good reputation. The original reason for the use of pozzolanic cements was the improved durability, combined with some economy, obtained in concrete in marine, hydraulic, and under ground structures. The reduction in heat evolution later provided another reason for their use in large mass concrete works and then, with the discovery of alkali – aggregate reaction, it was found that the addition of pozzolanas was often a useful means of preventing expansion arising from this cause. Finally pozzolanic cements have been developed as general constructional cements. Thus, in Italy, cements with 30-40 per cent pozzolana content are widely used in the same mix proportions as Portland cement for concrete in general building construction, for dams, and for reinforced concrete in sea water. A fairly high- strength Portland cement is used to help offset the loss in strength Portland cement is used to help offset the loss in strength at early ages.

Pozzolanas are usually defined as materials which, though not cementitious in them selves, contain constituents which will combine with lime at ordinary temperatures in the presence of water to form stable insoluble compounds possessing cementing properties. The pozzolanas can be divided into two groups, natural and artificial.

### **2.5.1. NATURAL POZZOLANA**

Pozzolanas of volcanic origin consist of glassy incoherent materials or compacted tuffs arising from the deposition of volcanic dust and ash. they may occur as dues for example the Rnenish trass , in a consolidated rock like form underlying material deposited subsequently, or in a more fragmentary and unconsolidated state like some of Italian pozzolanas. The Italian pozzolanas occur in the neighbourhood of Vesuvius and around Naples as incoherent trachytic tuffs, and around thẽ Rome, where they are present in a more compact form through still soft and easily worked. They are obtained from open pits and quarries, some of which have been use in centuries.

The volcanic pozzolanas are composed of a mixture of a mixture of silicates and contain both glass and crystalline particles. The volcanic ash and dust from which they were formed has

undergone rapid cooling, and in some cases has subsequently suffered considerable chemical alternation leading to the formation of zeolitic compounds. The zeolitization of vitreous volcanic ash has been reproduced in the laboratory by hydrothermal treatment at 200-400 °C in an alkaline medium. Volcanic ashes, such as those of Vesuvius, which contain a high content of crystalline minerals are much more stable and show only low pozzolanic activity. The zeolites found include herchellite, phillipsite, chabazite, and analcite. These are compounds of type  $(R_2Ca).Al_2O_3.4SiO_2.XH_2O$  differing in their contents of calcium and alkali cations. Analcite approximate to  $Na_2.Al_2O_3 .4SiO_2.2H_2O$ .

Three theories have been advanced to explain the origin of the activity of the pozzolanas.

1. the zeolitic materials is regarded as an alternation product of the original minerals, produced by prolonged exposure, after deposition, to waters containing carbon dioxide and to superheated steam.
2. pozzolanas originate from materials carried by volcanic eruption from geological strata of essentially clay like composition ; materials that during eruption have undergone heating sufficient to produce complete dehydration and chemical alternation.
3. The vitreous portion of pozzolanas is essentially a very porous areogel of high internal surface area.

### **2.5.2. ARTIFICIAL POZZOLANAS**

Burnt clays have in the past been used in mortar and concrete mainly when Portland cement were not available, or when a saving in cost has resulted. The materials used were, however, produced under crude and uncontrolled conditions of burning and, as it as since been shown that a degree of care in burning similar to that required in the manufacture of Portland cement is required to produce the best products, it seems probable that only relatively inferior materials were obtained. The investigations of Feret in France, of the building research station in Great Britain, and various American workers, have shown that excellent burnt clay pozzolanas can be obtained. Such a material was, for instance, used in the bonneville dam in the U.S.A. and in oil well cement manufactured in Great Britain. Modern work in India has also been directed to the production of more controlled materials.

Burnt clay pozzolanas are produced by burning suitable clays or shales at a temperature which varies from  $600^{\circ}\text{C}$  to over  $900^{\circ}\text{C}$ , depending in the nature of the clay and the conditions of burning; the product is ground to cement fineness. Certain spent oil shales which have undergone burning subsequent to distillation also yield, when selected with due care, a pozzolana of similar type and quality.

Raw clays consist essentially of a group of hydrated aluminium silicates, though alumina may be replaced to varying extents by bases such as  $\text{MgO}$ ,  $\text{Na}_2\text{O}$  and  $\text{CaO}$ . The water contents can also vary considerably. Kaolinite,  $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot \text{H}_2\text{O}$ , is a well-defined mineral and the kaolinite group also includes other minerals of the same composition such as nacrite, dickite and metahalloysite. The motmorrillonite group covers a variety of minerals with  $\text{Al}_2\text{O}_3 : \text{SiO}_2$  ratios varying from 1:3 to 1:6 and their isomorphous solid solutions. They can be typified by ideal formulae such as motmorrillonite  $\text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2$ , nontronite  $\text{Fe}_2\text{O}_3 \cdot 3\text{SiO}_2$ .

All clays have a considerable content, some 10-15 per cent, of combined water. In the kaolinite type clays this is not lost on heating until at temperature of  $500^{\circ}\text{C}$  or above is reached but the motmorrillonite and illite types commence to lose water at rather lower temperature. ( Lea, F.M. et al., The Chemistry of cement and concrete, Third edition, pages 418-420)

## **2.6. TYPES OF CLAY**

### **2.6.1. SHALE**

Shale (also called mudstone) is a fine-grained sedimentary rock whose original constituents were clay minerals or mud. It is characterized by thin lamina breaking with an irregular curving fracture, often splintery and usually parallel to the often-indistinguishable bedding plane. This property is called fissile. Non-fissile rocks of similar composition but made of particles smaller than 1/16 mm are described as mudstones. Rocks with similar particle sizes but with less clay and therefore grittier are silt stones.

In Pozzolanic cement, Natural Pozzolans can be easily ground in to very fine powder and can quickly react with lime and occurring Pozzolanic reaction. Various percentages of shale in various temperatures use to check the pozzolanicity instead of Pozzolans.

### **2.6.2. ARUWAKKALU QUARRY CLAY**

The plants quarry is established at the Aruwakkaru area, which has a weathering igneous rock type. The limestone is found under the 15 meters from the overburden layer, under that layer can be seen another silica rich clay layer and under that layer has another carbonate rich limestone layer. These layers cannot be taken out very purely; because of there are some clay pockets can be seen along these layers. In this quarry, clay layer is richer from the silica, which has a form of Quartz.

### **2.6.3. ACHCHIMALE CLAY**

Achchimale clay is a siliceous and aluminous material which reacts with calcium hydroxide in the presence of water to form compounds possessing cementitious properties at room temperature and that have the ability to set under water. It is taken from achchimale area and called achchimale clay.

## **2.7. CHEMISTRY OF POZZOLANIC CEMENT**

Pozzolanic cements are mixes of Portland cement and pozzolan which is dispersed in water and kept under certain conditions eventually produce solutions unsaturated with calcium hydroxide.

Cement and pozzolan show different reaction processes and react at different ages. As is known, the pozzolanic reaction becomes apparent after 7-15 days since mixing occurred.

Production of pozzolanic cements is made by two ways. Clinker and pozzolan are ground separately and mixing with after grinding and clinker is ground with pozzolan at the same time. Although more homogeneous cement is obtained by the first method, particle size of Portland cement and pozzolan differ from each other and are not well controlled. On the other hand, in the second method, problem of homogeneous mixture arises whereas the better particle size distribution is obtained.

The reaction of pozzolanas with calcium hydroxide under moist conditions called pozzolanic reaction. The lime pozzolan reaction can occur in Portland pozzolan cement when the lime is produced by the hydration of  $\beta$  C<sub>2</sub>S and C<sub>3</sub>S minerals in the cement. This reaction begins in the first few hours of the hydration, but the effect of the pozzolanic reaction take place more slowly. The mechanism of the lime pozzolan reaction in Portland pozzolan cement is not understood completely. This simple explanation of reaction can be given as follows.



(Pozzolan + hydrated lime)(Mehta, P.K., "Pozzolanic and Cementitious By-Product in concrete- Another Look", Silica Fume, Slag and Natural Pozzolan in Concrete Proc. 3rd International Conference, 1989, Trondheim, Norway.)

## **2.8. INTERNATIONAL STANDARDS AND REGULATIONS**

### **2.8.1. BRITISH STANDARD (BS) FOR POZZOLANIC CEMENT**

- The calcium compounds present in lime, and calculated as CaO, shall not be less than 93 per cent by weight of ignite sample.
- The loss on ignition, after correction for the carbon dioxide content, shall be between 21 and 25 per cent of weight.
- The expansion in the Le Chatelier soundness test laid down in the standard shall not exceed 2mm.

- The residue on the BS 85 mesh sieve shall not exceed 1 per cent and after passing this sieve not more than 2 per cent shall remain on the BS 170 sieve

### **2.8.2. ASTM STANDARD FOR POZZOLANIC CEMENT**

- The ASTM specification requires for pulverised ash strength of 800 lb/in<sup>2</sup> at 7 days in the 'pozzolanic activity index with lime' test and one of 85 percent of the control at 28 days in the 'pozzolanic activity index with Portland cement' test.
- There is also requirement that the compressive strength of a 1:0.25:2.5 portland cement: pozzolana : graded sand mortar shall not be less than that of a 1: 2.75 portland cement : graded sand mortar using the same cement at 7 or 28 days.
- The proportion of pozzolana is required to be between 15 and 45 per cent by weight of cement and pozzolana.

## CHAPTER 3

### 3. METHADODOLOGY

#### 3.1. METHADODOLOGY:

##### 3.1.1. STANDARDIZATION OF THE EDTA SOLUTIONS

1.00 g of calcium carbonate ( $M_1$ ) was weighted and placed it in a 400 ml beaker with approximately 100 ml of water. The beaker was covered with a watch glass and it was carefully introduced approximately 10ml of 0.1 M hydrochloric acid. It was stirred with a glass rod and was ensured that dissolution is completed; then the beaker was brought to boil in order to expel the dissolved carbon dioxide. Was cooled to room temperature and transferred to volumetric flask. The beaker and watch glass were washed carefully with water and was added the washing to the solution and was made up to 1000 ml of water.

The 50 ml of calcium solution was pipetted into a beaker. It was diluted with water to a suitable volume. The pH of this solution was adjusted by using a pH meter to 12.5 with the sodium hydroxide solution. The end point was determined using following method.

##### Visual determination of the end point (alternative method)

About 0.1 g of the calcon indicator was added, without weighing. It was stirred and titrated with 0.03 mol/l EDTA solution until the colour changes to from pink to blue. The volume  $V_1$  was used to calculate the standardization factor  $F_1$  of the EDTA solution from following formula.

$$F_1 = (M_1 * 16652) / V_1$$

$M_1$ -Mass of Calcium Carbonate

$V_1$ - Volume of EDTA



### **3.1.2. STANDARDIZATION OF THE SOLUTION OF HYDROCHLORIC ACID**

0.2g of sodium carbonate was weighed and it was added to the 250 ml conical flask. Solution was dissolved in 50 ml to 75 ml of water. Five drops of methyl orange indicator was added to the solution and titrated with the 0.1 mol/l dilute hydrochloric acid until the colour changes from yellow to orange.

The factor  $F_2$  of the solution of Hydrochloric acid was calculated by using below formulae.

$$F_2 = (M_2 * 18870) / V_2$$

$M_2$ -Mass of Sodium Carbonate

$V_2$  - volume of HCl

### **3.1.3. STORAGE AND FILTRATION**

100 ml of freshly boiled water was pipette into polyethylene container. It was sealed and placed in the uniform temperature enclosure until equilibrium is reached (about 1 h). The container was removed from the uniform temperature enclosure. 20g of cement was poured to be tested in to polyethylene container by using the wide stem funnel. The container was immediately sealed hermetically.

The container was shaken vigorously for about 20 s to avoid formation of cement lumps. A horizontal rotary motion was used to prevent, any part of the sample or liquid being thrown up and remaining separated from the rest of the solution.

The container was replaced in the uniform temperature enclosure, to make sure that its base is horizontal so that deposited layer of cement has a uniform thickness. All operations were performed outside the uniform temperature enclosure as quickly as possible. After a period of 8 days or 15 days in the uniform temperature enclosure, the container was removed and filtered the solution immediately under the vacuum through the Buchner funnel in to the vacuum flask by

using dry double filter paper in less than 30 s. The vacuum flask was sealed immediately and let the filtrate cool to room temperature.

### **3.1.4 DETERMINATION OF HYDROXYL ION CONCENTRATION**

The vacuum flask was shaken to homogenize the filtrate. 50ml of the solution was pipetted into the 250ml beaker. Five drops of methyl orange indicator was added and determined the total alkalinity with the 0.1 mol/l dilute hydrochloric acid. The titration end point corresponds to the colour change from yellow to orange. The titrated solution (A) was kept for the determination of calcium oxide concentration.

The Hydroxyl ion concentration was calculated using following formula

$$[\text{OH}^-] = 2 * V_3 * F_2$$

$V_3$ -Volume of Hydrochloric acid

$F_2$ -Factor of Hydrochloric acid solution

### **3.1.5. DETERMINATION OF THE CALCIUM OXIDE CONCENTRATION**

The pH of the titrated solution (A) remaining after completing determination of hydroxyl ion concentration was adjusted to 12.5 with sodium hydroxide solution, using the pH meter. Solution was titrated with 0.03 EDTA solutions to determine the end point.

The calcium oxide concentration was calculated in mill moles per litre, from the formula

$$[\text{CaO}] = 0.6 * V_4 * F_1$$

$V_4$ - Volume of EDTA solution

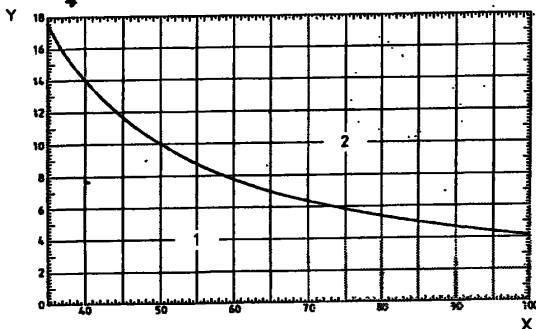
$F_1$ - Factor of EDTA solution

### 3.1.6 ASSESSMENT OF POZZOLANICITY

The graph was plotted against the concentrations of hydroxyl ion to calcium ion (expressed as calcium oxide) in the solution. As a point on a figure which shows the saturation concentration of calcium ion (expressed as calcium oxide) as the function of the hydroxyl ion concentration at 40 °C. the curve in figure may be expressed mathematically over the range 45 to 90 mmol/l [OH] by  $[CaO] = 350 / ([OH]-15)$  where the calcium ion and hydroxyl ion concentrations are given in mill moles per litre.

The cement satisfies the test for pozzolanicity when the point plotted is bellowing the curve of calcium ion saturation concentration shown on below figure.

EN 196-8:2005 (E)



1 – Pozzolanicity passes

2 – Pozzolanicity fail

X – Hydroxyl ion concentration

Y- Calcium ion concentration

Figure 3.1 Standard Pozzolanicity curve

## CHAPTER 4

### 4. RESULTS AND DISCUSSION

#### 4.1 POZZOLANICITY OF CEMENT SAMPLES

##### 4.1.1. SHALE

Under 700 °C and 800 °C the concentrations of hydroxyl ion and calcium oxide values (mmol/l) mentioned in following table.

Table 4.10 Pozzolanicity of shale samples at 700°C and 800 °C

SAMPLE	OH	CaO	POZZOLANICITY
700 SHL 10%	49.04	24.31	FAIL
700 SHL 20%	36.68	13.51	PASS
700 SHL 35%	25.78	8.58	PASS
800 SHL 10%	48.83	24.56	FAIL
800 SHL 20%	37.72	14.22	PASS
800 SHL 35%	41.50	15.75	FAIL

These hydroxyl and calcium oxide concentrations values plotted in a standard curve to test the pozzolanicity. The cement satisfies the test for pozzolanicity when the point plotted is bellow the curve of calcium ion saturation concentration shown on below figure

- 1 – Pozzolanicity pass
- 2 – Pozzolanicity fail
- X – Hydroxyl ion concentration (mmol/l)
- Y – Calcium ion concentration (mmol/l)

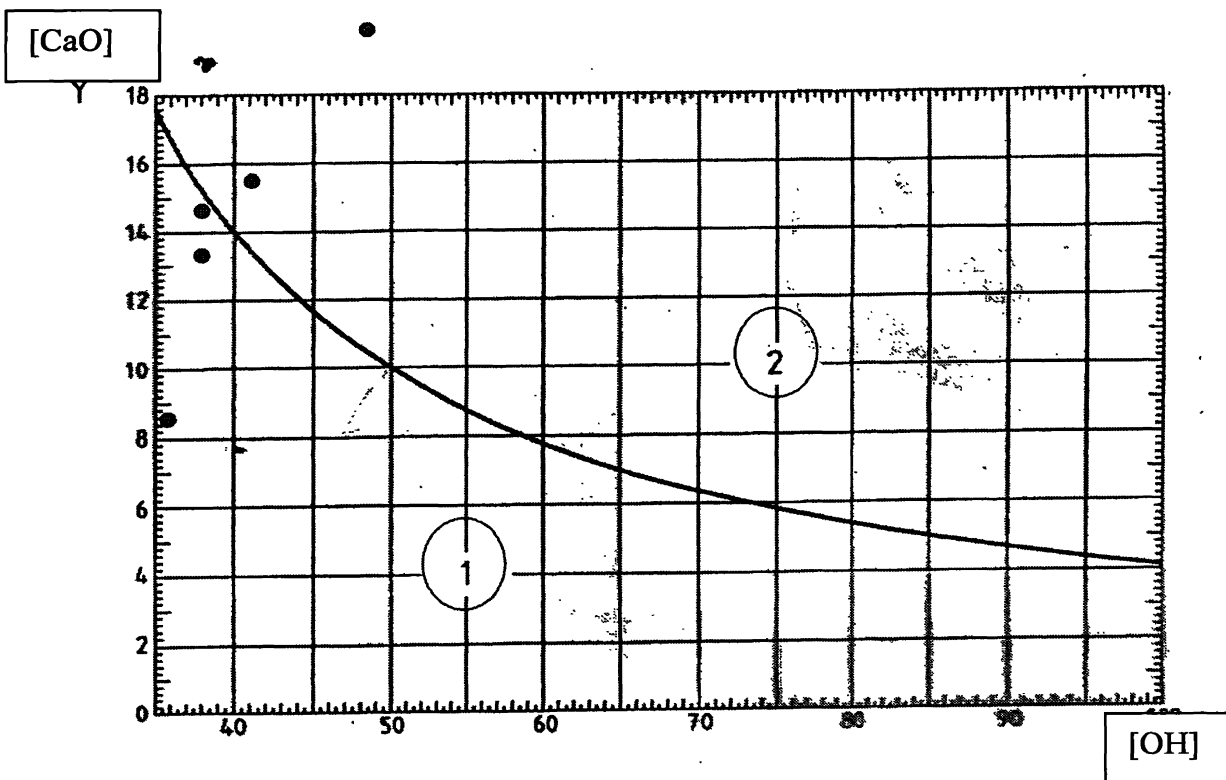


Figure 4.1 Standard Pozzolanicity curve for shale

According to the above graph, it is presented that Pozzolanicity gives under 700 °C Shale 20%, 700 °C Shale 35% and 800 °C Shale 20%. 700 °C shale 10%, 800 °C shale 10% and 35% samples don't show the pozzolanicity, which is shown in the upper part of the curve in this figure. But 800 °C shale 35% sample is very close to show pozzolanicity. 700 °C shale 10% sample shows the highest hydroxyl ion concentration and 800 °C shale 10% shows the highest CaO concentration. According to present results, 700 °C Shale 20%, 700 °C Shale 35% and 800 °C Shale 20% samples can be used to produce pozzolanic cement.

### 4.1.2 QUARRY CLAY

Under 700 °C and 800 °C the concentrations of hydroxyl ion and calcium oxide values (mmol/l) mentioned in following table.

Table 4.2 Pozzolanicity of quarry clay samples at 700°C and 800 °C

SAMPLE	OH <sup>-</sup>	CaO	POZZOLANICITY
700 Q.C 10%	42.56	25.47	FAIL
700 Q.C 20%	31.85	19.04	FAIL
700 Q.C 35%	28.29	21.74	FAIL
800 Q.C 10%	47.73	26.43	FAIL
800 Q.C 20%	42.96	27.62	FAIL
800 Q.C 35%	46.11	24.09	FAIL

These hydroxyl and calcium oxide concentrations values were plotted in a standard curve to test the pozzolanicity. The cement satisfies the test for pozzolanicity when the point plotted is bellow the curve of calcium ion saturation concentration shown on below figure

EN 196-5:2005 (E)

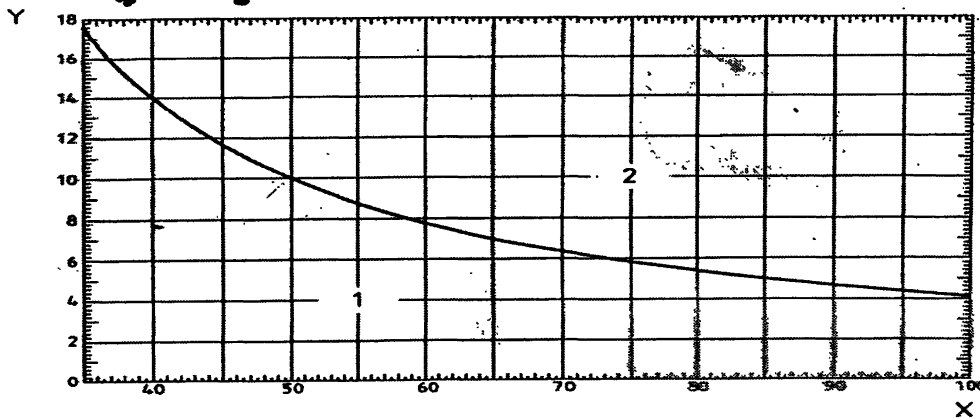


Fig 4.2. Standard Pozzolanicity curve for quarry clay

- 1 – Pozzolanicity pass
- 2 – Pozzolanicity fail
- X – Hydroxyl ion concentration (mmol/l)
- Y – Calcium ion concentration (mmol/l)

According to above figure all samples don't show the pozzolanicity. Under 700 °C and 800 °C all plotted values are shown above part of the standard curve. 800 QC 10% sample shows the highest hydroxyl concentration and calcium oxide concentration. According to results it's difficult to use any sample to use pozzolanic cement.

### 4.1.3. ACHCHIMALE CLAY

Under 700 °C and 800 °C the concentrations of hydroxyl ion and calcium oxide values (mmol/l) mentioned in following table

Table 4.3 Pozzolanicity of achchimale clay samples at 700°C and 800 °C

SAMPLE	OH <sup>-</sup>	CaO	POZZOLANICITY
700 A.C 10%	50.56	24.32	FAIL
700 A.C 20%	49.04	20.92	FAIL
700 A.C 35%	39.82	15.75	FAIL
800 A.C 10%	42.57	26.36	FAIL
800 A.C 20%	38.64	21.22	FAIL
800 A.C 35%	35.59	18.43	FAIL

These hydroxyl and calcium oxide concentrations values plotted in a standard curve to test the pozzolanicity. The cement satisfies the test for pozzolanicity when the point plotted is bellow the curve of calcium ion saturation concentration shown on below figure

- 1 – Pozzolanicity pass
- 2 – Pozzolanicity fail
- X – Hydroxyl ion concentration (mmol/l)
- Y – Calcium ion concentration (mmol/l)

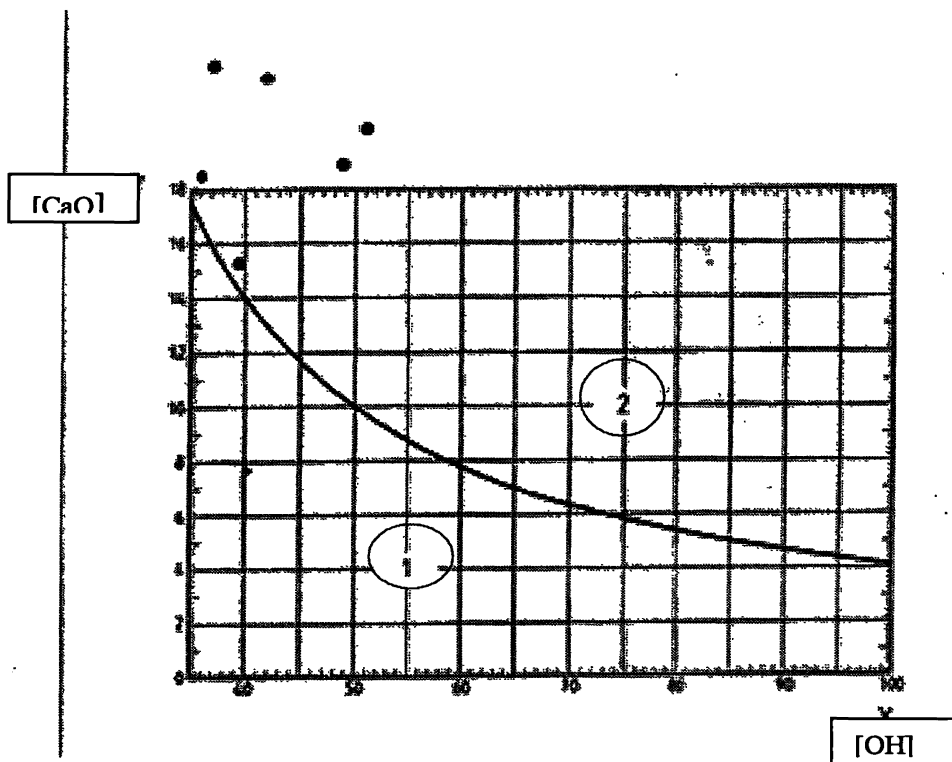


Fig 4.3. Standard Pozzolanicity curve for achchimale clay

According to above figure all samples don't show the pozzolanicity. Under 700 °C and 800 °C all plotted values are shown above part of the standard curve. 700 QC 10% sample shows the highest hydroxyl concentration and 800 AC 10% sample shows the highest calcium oxide concentration. 700 AC 35% sample are just closed to the standard curve. so it is closed to show pozzolanicity. But According to results it's difficult to use any sample to use pozzolanic cement.



## 4.2 COMPRESSIVE STRENGTH VARIATION OF CEMENT SAMPLES

The replacement amount has an important role for this strength development and of pozzolanic cement at specified ages. Therefore, relationship between pozzolan content and strength development were taken in to consideration and represented in figure1, 2, and 3.

### 4.2.1 SHALE

In my experiment determined the 1D, 2D, 7D and 28 D compressive strength values of cement samples using compressive strength testing machine. These values plotted with pozzolan content and study the compressive strength variation.

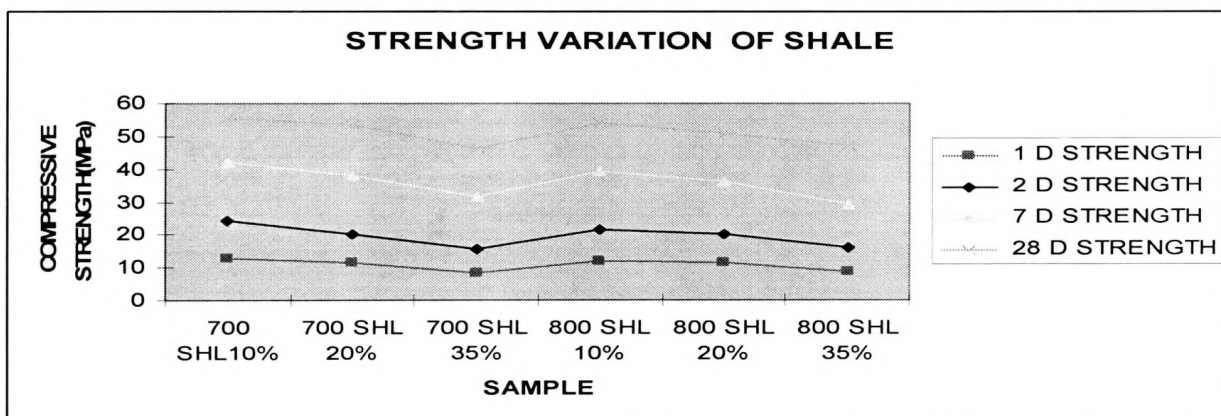


Fig 4.4. Compressive strength variation of shale

According to this figure shows compressive strength increases with the time. When pozzolan content increases compressive strength drops down. Because of hardening reaction the compressive strength increases in cement samples. Hardening reaction occur with dicalcium silicate. According to table 2.1 can see  $C_2S$  forms above  $1000^{\circ}C$ . when adding pozzolan content, the amount of Portland clinker decreases. Then amount of  $C_2S$  decreases. That is the reason for the decrease the early strength of the cement samples.

The highest compressive strength value gives under 700 shale 10%.the lowest 28 D compressive strength value gives 700 shale 35% sample. But all these compressive strength value gives higher than the values which are needed for the international standards and regulations.

#### 4.2.2 QUARRY CLAY

Through my experiment determine the 1D, 2D, 7D and 28 D compressive strength values of quarry clay cement samples using compressive strength testing machine. These values plotted with pozzolan content and study the compressive strength variation.

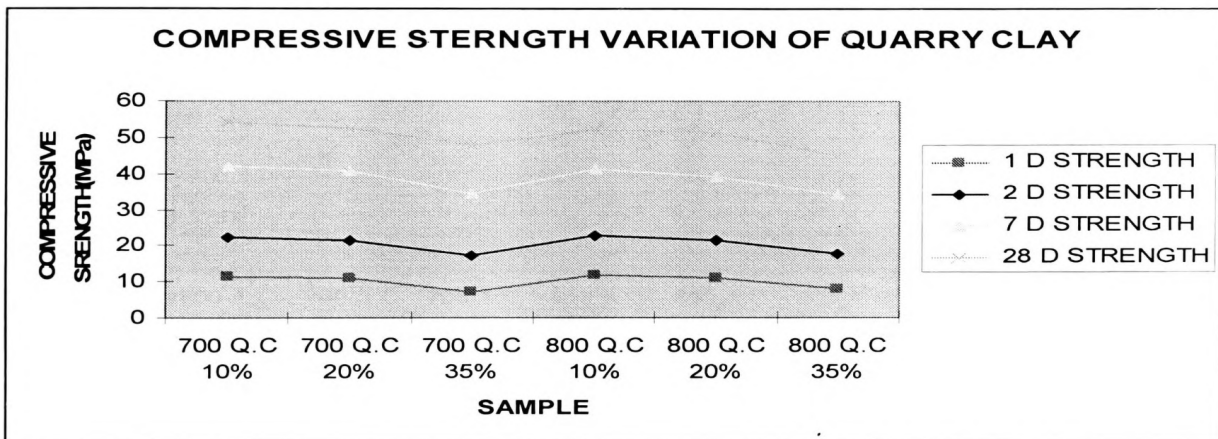


Fig 4.5. Compressive strength variation of quarry clay

700 QC 10% sample gives the highest 28D compressive strength value. But this quarry clay samples dont give pozzolanicity.

According to these figures shows early strength drop down with pozzolan content. When pozzolan content increases, compressive strength decreases. Percent decrease in strength was not proportional with pozzolan content of cement. In the pozzolanic cements show law earlier strength values. After that strength increases. But these law earlier strength values are higher than cement standards and regulations.

### 4.2.3 ACHCHIMALE CLAY

700 AC 10% sample gives the highest compressive strength value. The same pattern also represented in achchimale clay like quarry clay and shale. Because of amount of dicalciumsilicate decreases the early strength drops down. In pozzolanic cement generally consider about the later strength. So when adding pozzolana in to Portland cement clinker, early strength drops down. But because of pozzolanicity later strength increase than the Portland cement.

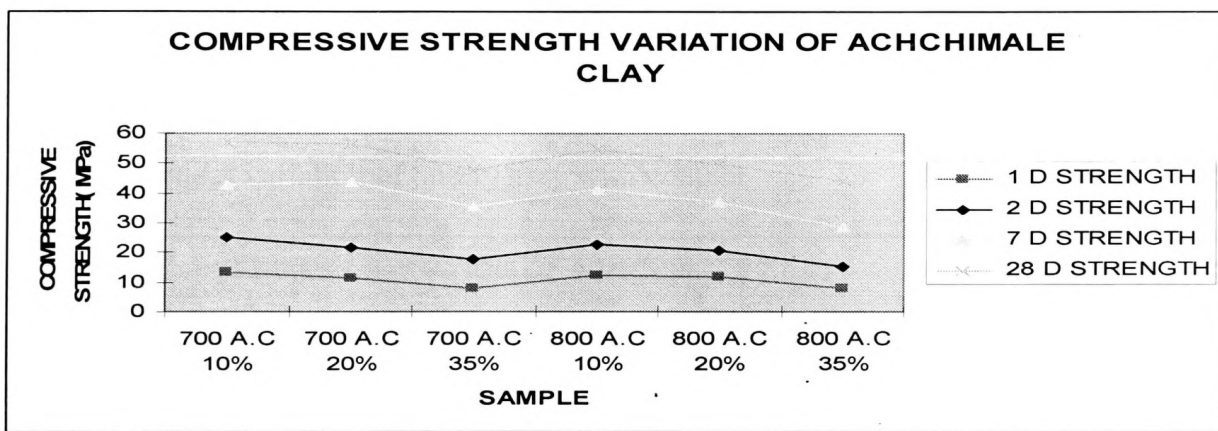
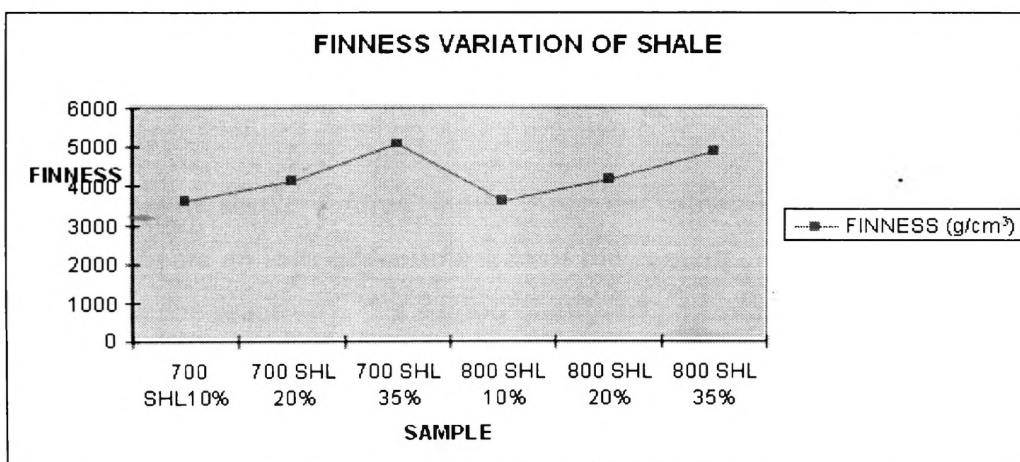


Fig 4.6. Compressive strength variation of achchimale clay

### 4.3. FINENESS VARIATION OF CEMENT SAMPLE

The fineness values of cement samples were given in table 4.7, 4.8; 4.9



#### 4.7. Fineness variation of shale with content

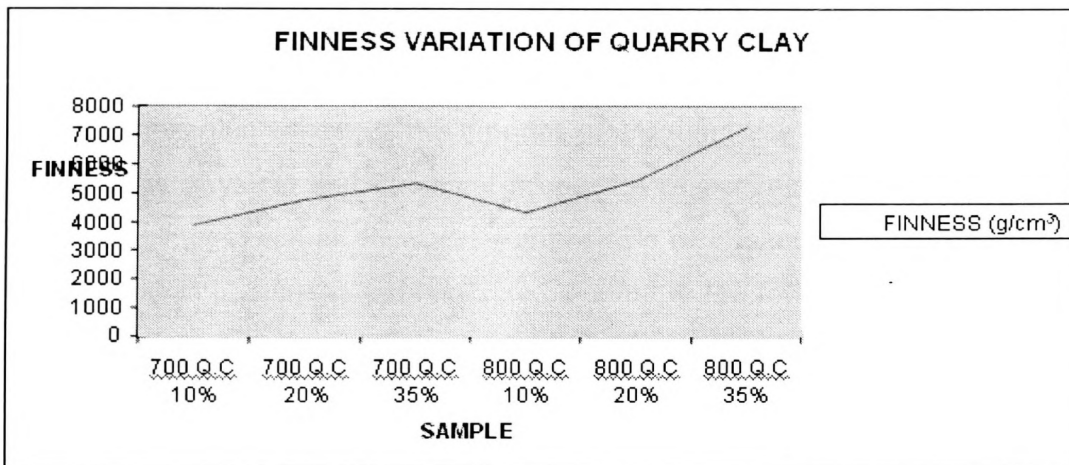


Fig 4.8. Fineness variation of quarry clay with content

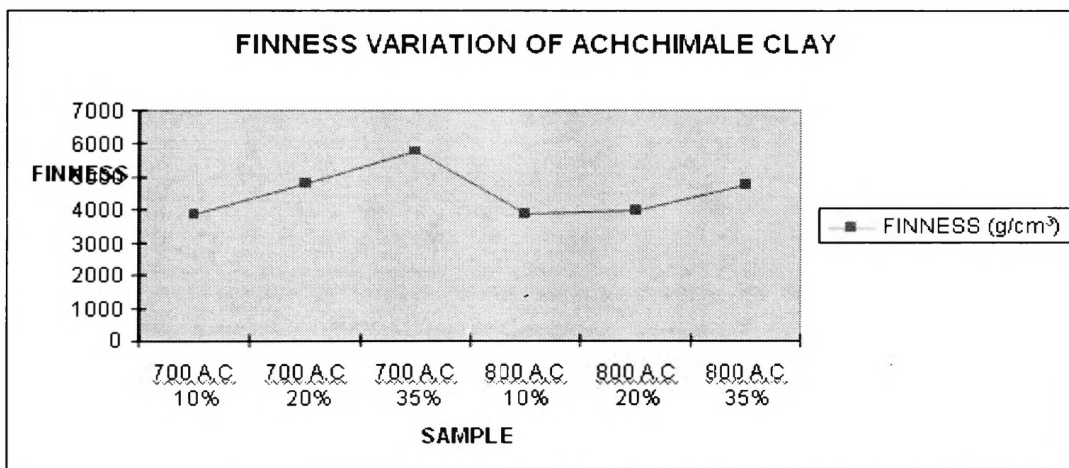


Fig 4.9. Fineness variation of achchimale clay with content

In this my experiment, determined the fineness values of shale, quarry clay and achchimale clay. These fineness values graphed with pozzolan content.

According to above figures shows fineness values increase with pozzolan content. Fineness values indicate an idea of particle size of the cement. When particle size becomes small it causes to increase the reactivity. It's a good quality of pozzolanic cements.

Throughout all cement samples, the highest fineness value included in 700 QC 35% sample. 700 SHL 35% shows the maximum fineness value in shale samples and 700 AC 35% sample

represent the maximum value in achchimala clay samples. according to above figures of sh1, qc, ac all figures shows the same pattern. The fineness values increase with pozzolan content. Fineness value of cement can be increased by using pozzolanas.

portland pozzolan mixtures the amount of Portland cement replaced by pozzolan depends not only on the physical and chemical properties of pozzolan but also on the characteristic of the Portland cement such as chemical composition of clinker and fineness. Vast amount of research on pozzolanas in cement and concrete have led to the following general conclusions related with the properties of cementitious systems containing pozzolans:

## **CHAPTER 5**

### **5. CONCLUSIONS AND RECOMMENDATIONS**

#### **5.1. CONCLUSIONS**

- Under 700°C, the shale at 20%, 35% gives pozzolanicity. Under 800°C, the shale with 20% cement sample gives pozzolanicity. But in manufacturing process burning is too expensive process. According to my results 700 °C is suitable temperature for adding pozzolana. So adding pozzolana is the profit.because of this reason 700 SHL 35% very suitable for produce pozzolanic cement.
- Early strength drops down with pozolan content. When pozzolan content increases, compressive strength decreases. Percent decrease in strength was not proportional with pozzolan content of cement.
- Fineness values increase with increasing pozzolan content.

#### **5.2. RECOMMENDATIONS**

- Cooling effect should consider for this experiment.
- For the full understand of the effect of temperature on the pozzolanicity, should extend the temperature range 500<sup>0</sup>C – 1000<sup>0</sup>C

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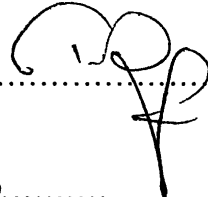
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