

Characterization of Historical Mortars

A Thesis

submitted to

Indian Institute of Science Education and Research Pune in partial fulfillment of the requirements for the BS-MS Dual Degree Programme

By

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May, 2021

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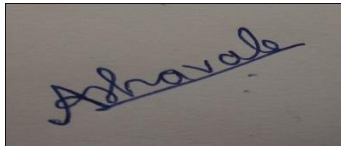
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Certificate

This is to certify that this dissertation entitled “Characterization of Historical Mortars” towards the partial fulfillment of the BS-MS dual degree programme at Indian Institute of Science Education and Research, Pune represents the study and work carried out by Athang Dhavale at the Undergraduate Laboratory, IISER Pune under the supervision of Dr Pushkar Sohoni and mentorship of Dr Abhijit Biswas during the academic year 2020-2021.



Dr Pushkar Sohoni



Athang Dhavale

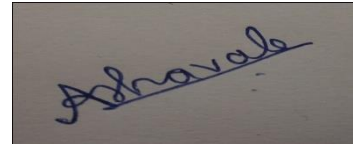
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Declaration

I hereby declare that the matter embodied in the report titled “Characterization of Historical Mortars” is the result of the work carried out by me at the Undergraduate Laboratory; IISER Pune under the supervision of Dr Sohoni and the mentorship of Dr Biswas and the same has not been submitted elsewhere for any other degree.



Athang Dhavale

Date: 15 May 2021



Dr. Pushkar Sohoni

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Acknowledgement

I have been working under the supervision of Dr Pushkar Sohoni for over two years. He has always been supportive regarding my work and I am grateful to him for supporting me ably during this project. I would also like to give a special thanks to my mentor Dr Abhijit Biswas for his invaluable help and guidance. Also I would like to express my sincere thanks to Mr-Rahul Maity and Dr Vaidyanathan for helping me out with my XRD tests. Similarly I would also like to thank Dr Gyana Ranjan Tripathy and the ECS department; IISER Pune for providing results for my XRF tests. I would also like to thank Mr.Aditya Malkar for providing samples along with their inventories and my guide Dr Siddhesh Kamat.

Lastly I would like to thank my family and friends for their continuous support in the present hard times. Their motivation has greatly helped me in finishing my project.

Abstract:

Historical monuments are the cultural heritage of any nation. They serve as points of remembrance and can facilitate tourism. In this project, there is an attempt to characterize mortar samples obtained from different historical monuments in the Indian state of Maharashtra. Their chemical composition has been analyzed and tabulated. This study can serve as a reference material for any future research in this field and the data obtained could serve as a benchmark for any monument restoration work if it takes place in these selected areas. This study also tried to determine if any particular pattern or trend is observable in the chemical parameters calculated for these samples.

Chapter 1: Introduction to Historical Mortars

Mortar usage began with mud and clay-based mortars as they were low-cost and easy to prepare; and helped to contain the natural materials used by the ancient civilizations. For instance, in Egypt this gradually progressed to mortars made with gypsum where the natural material was heated on small fires. Eventually, in the course of history, mortars found usage as lining materials in cisterns, wells, aqueducts, shafts and duct-drains, as supporting materials for pavements and mosaics and as plasters on external and internal walls [1]. They were also used as supporting materials for frescoes. Historical mortars were usually composite. They comprised of:

A) Binder (hydraulic or an aerial binding material or a mixture of these binding materials): In mortar, a binder is a material which keeps the other components together through forces of cohesion or adhesion. Some of the most common binders are calcium carbonate (lime), gypsum, cement (natural or artificial) and some types of clay.

B) Aggregates (crystalline or amorphous): These are substances which provide stability, volume and a resistance to wear or erosion. This includes various types of sands, crushed rock, gravel, slag, etc.

C) Additives (Passive or active): They are used to increase the performance of mortars. Properties like water resistance, shock absorption, bond strength can be improved through the use of additives. Examples of additives used in the mortars: a) lime, b) silica fume c) polypropylene fibers.

Under this project, samples were collected from historical monuments such as forts and palaces within the Maharashtra state and there was an attempt to figure out if any definitive pattern is observed with respect to aspects such as historical period, then geo-political conditions, nature of monument etc. A total of 18 samples were collected from 9 locations. The samples could be crudely differentiated based on 3 aspects:

1) Location

2) Nature of monument

3) Type of source structure.

Note that some of the samples belong to the same location but are extracted from different source structures. For instance, within a fort there are various constructions such as water tanks, fortification walls, watchtowers, etc. Is the mortar used for all these constructions the same? Or is there is a variation? And if it is, to what extent is it so?

The sources from which the samples were extracted are discussed in the next chapter.

Chapter 2: Sample Collection

2.1 Ausa Fort

Location co-ordinates: 18.25°N 76.5°E.

History and info: This fort was significant in the conflicts between the Deccan Sultanates in the post -Bahamani period. Later it was captured by Malik Ambar in 1014. It is almost square in shape and has a moat or khandak (ditch) around, approximately (120 ft) in width which is now nearly dry. It also has a glance, a retaining wall, a double rampart and a covered way fortified further with massive bastions, which are mostly semi-circular and is mounted with a huge cannon.





These 2 samples are collected from underground water tank inside the core area which is known as '*Pani Mahal*'. Water tank is accessed through narrow stairs. Flat roof of the tank is supported with colonnade and piers connected with shoulder arches. Structure is built in stone masonry using lime mortar and the ceiling is also plastered with lime mortar. Samples were taken from the soffit of the arch which was undisturbed though partly eroded, from which the core portion was augered with the help of a pointed tool. Fresh samples were collected in the plastic zip pouch with an air tight packing. A total of 2 samples were collected from this source with one of them having external plaster as its primary component.



A third sample was collected from the structure above the bastion near entrance, which was the Watch-tower, built in traditional lakhori bricks and lime surkhi mortar. Portion near the soffit of vault and joint of the masonry was augered and the sample was thus obtained. Both the selected locations were undisturbed and free from moisture contact.

Total: 3 samples.

2.2 Kharda Fort

Location co-ordinates: 18.633439°N 75.475365°E.

History and other info: This fort was built by Sardar Nimbalkar in 1745. On March 11, 1795, the Marathas defeated the Nizam at this place. The wall and the entrance of this fort are still in a good condition. Also there is a barawa and a mosque inside the fort. There is also a memorial temple dedicated to Pant Nanasaheb Nimbalkar.

Sample was collected from the structure in the core area which is vaulted. Stone and lime mortar was used in building the stable. Extraction point was at the inner side on the soffit. The sample was removed using the helm of a pointed tool and collected in plastic zip air tight pouch.

KHARDA FORT:



Total number of samples: 1

2.3 Malegaon Fort

Location co-ordinates: 20.545805°N 74.530301°E.

History and other info: In 1740, this fort was constructed by Naro Shankar Raje Bahadur who was a general of the Maratha Empire. It is presently in a fine condition. The entrance doors are lofty and the height of the inner wall is around sixty feet. There are a couple of cannons at the fort entrance and a hexagonal shaped well is inside the fort.

Sample was collected from the damaged merlons above bastion constructed in stone and lime surkhi mortar. Fresh and unexposed mortar was removed with the help of a pointed tool and stored in a plastic zip pouch.

MALEGAON FORT:



Total number of samples: 1

2.4 Galna Fort

Location co-ordinates: 20.7733387°N 74.5333314°E

History and other info: It was a major fort during medieval times in the south Khandesh region of Maharashtra. The fort bastion and entrance are still in a good condition. It was an important fort on the Burhanpur-Surat trade route.

GALNA FORT



Sample 1 was collected from the damaged wall of Arcade structure by removing top masonry and the fresh exposed mortar was collected in a plastic zip pouch.



Sample 2 was collected from the structure which is partially underground and covered with a brick vault and a dome built in traditional lokhori brick and lime mortar. Structure is a typical '*Hamamkhana*' (bath). Sample was collected from the core brick joint of the damaged corner of the roof.

Total number of samples: 2

2.5 Dharur Fort

Location co-ordinates: 18.819032°N 76.104719°E.

History and other info: Dharur Fort was built on the plain on Palghat hills and it had a natural protection as it was surrounded by valleys from three sides and had a trench built to protect it from the front. Until one enters it the valleys are not seen and thus it appears as a complete land fort. It is referred in the donation given by the Rashtrakuta king, Govina III as Dharur.

DHARUR FORT

Area Location of Sample 1

SAMPLE-1 (Sample of internal wall Plaster of Ruins of Masjid)





SAMPLE-2 (Mortar from Masonry of Merlons on Bastion)



Total number of samples: 2

2.6 Naldurga Fort

Location co-ordinates: 17.815685°N 76.284953°E.

History and other info: It was named after the King Nalraja who had it built in the medieval era .The walls in the inner section of the fort are ruined and there is a wide road leading up to the centre. A significant structure which connects the fort and the Ranmandala is a dam constructed across the Bori river. There are quite a few bastions present within this fort namely; Sangram Buruj, Sangam Buruj, Nagar Buruj and Upli Buruj which is the highest point in the fort.

Naldurga Fort:



Sample 1 was collected from the Ruin structure in the core area, which was just an arch gateway type structure built in stone and lime mortar. Fresh core sample was collected with the help of tools and stored in a plastic zip pouch.



Sample 2 was collected from the west-side inner rampart wall at the damaged portion of the bastion. Stone and lime mortar was used in masonry. Fresh core sample was collected in a plastic zip pouch.



Sample 3 was collected from the damaged portion at the inner gateway of the fort. Fresh core sample was collected with the help of an auguring tool. Masonry was constructed in stone and lime mortar.



Total number of samples: 3

2.7 Holkarwada Chandwad

Location co-ordinates: 20.330556 °N 74.245278°E.

It was built by Queen Ahilyabai Holkar in the eighteenth century. It has a famous network of subways to various key locations like Goddess Renuka Temple and a well near Vinchur which was built by the Queen as a safe passage from enemies. There is also a huge old warehouse which was used to store grains. The palace has wood carved pillars and a decorative façade.



Sample 1 was collected from the Door Jamb of 'Mazghar' in the rear courtyard. Core sample from the joint of Brick masonry was collected with the help of tools and stored in a plastic zip pouch. This plaster was dismantled during the repair work done by the Department under a restoration project.



Sample 2 was collected from the joint of stone masonry at plinth of the courtyard. Core fresh lime mortar was collected from the damaged surface with the help of tools and stored in a plastic zip pouch.

Total number of samples: 2

2.8 Devargaon Wada:

Location co-ordinates: 20.038256 °N, 74.115608 °E

This residential mansion belonged to Narayanbhau Shinde, who was the *dewan* of Baroda in 1868, during the Rule of Maharaja Khanderao Gaikwad (1847-70). It is located in Nashik district of Maharashtra.

A single sample was extracted from this location.



Total number of samples: 1.

1.9 Khoja Naik Fort; Nastanpur

Location co-ordinates: 20.319722 °N 74.796944 °E

This fort was built in post-medieval age. This is a land fort. The King who built this fort had vast amounts of wealth and was targeted by the British for it. However they failed to capture him and he escaped via an underground tunnel. There are quite a few urban legends regarding this place.

NASTANPUR FORT:



Sample 1 was collected from the bastion Wall of entrance which was damaged and in a dilapidated condition. Masonry was mass lime concrete with a brick aggregate. Fresh sample was collected by removing the upper layer with the help of tools and collected in a plastic zip pouch.



Sample 2 was collected from the damaged fencing wall just outside the fort entrance. The wall was built in a traditional lakhori brick and lime mortar. Fresh core sample was collected in a plastic zip pouch.



Total number of samples: 2

1.10 Chakan Fort

Location co-ordinates: 18.757722 °N 74.862500 °E

The name of this fort is 'Sangram Durga'. It is a hill fort which is famous for the defense put by the Maratha commander Firangoji Narsala against an overwhelming force of the Mughals during Shaista Khan's invasion of Pune.



Sample was collected from niche of the structure, which was built from brick and lime mortar.



Total number of samples: 1

Before moving on to the experimental section, it is better if each of the samples is assigned a code and certain selected features of every location tabulated. The table presented below will be useful as a reference.

Table 2.1: Summary of the Sample Data

Sample code with Name	Building type and location name	Extraction source of the sample and mortar type	Additional notes
S1: Kharda Stable	Land Fort; Kharda	Stable: It is a Vault structure used as a Shelter for horses. Type: Stone and Lime Mortar.	Building age is around 250 years. The sample was extracted using a hammer and a chisel.
S4: Devorgaon Wada	-NA-	Pillar of fencing gate. Type: Stone and lime Mortar.	Building age is around 200 years. The sample was extracted through the use of drill core.
S2: Galna Water Tank S6: Galna Wall	Hill Fort; Galna	i) Underground storage structure. ii) Vault/Arch of the structure. Type: Brick and Lime Mortar.	Building age is around 450-500 years. Samples were extracted using a hammer and a chisel.
S3: Malegaon Fort	Land Fort; Malegaon	Bastion and merlons Type: Stone and Lime Mortar.	Building age is around 250 years. The sample was extracted using a hammer and a chisel.
S5: Ausa Fort Watch Tower S12: Ausa Fort Water Tank	Hill Fort; Ausa	i) Watch-tower: A high structure to	Building age is around 600 years.

PP S17:Ausa Fort Water Tank		create an elevated observation point. ii) Underground water tank. Type: Stone and Lime Mortar.	The samples were extracted using a hammer and a chisel.
S7: Nasthanpur II S18: Nasthanpur Nandgaon I	Land Fort; Nasthanpur	i) The damaged fencing wall just outside of the fort entrance. ii) The bastion Wall of the entrance. Type: Stone, Brick, Lime and Mud Mortar.	Building age is around 200 years. The Samples were extracted using a hammer and a chisel as well as a drill core.
S8: Dharur Bastion S9: Dharur Ruins Plaster	Land Fort; Dharur	i) Bastion. ii) Ruined inner wall of the structure. Type: Stone, Brick and Lime Mortar.	Building age is around 400 years. The Samples were extracted using a hammer and a chisel.
S10: Chakan Fort Burj	Hill Fort; Chakan	A bastion towards the east side of the Gateway. Type: Stone, Brick, Lime and Mud Mortar.	Building age is around 500-600 years. The sample was extracted through the use of drill core.
S11:Naldurga Plaster Ruins S14: Naldurga Entrance Gate	Land Fort; Naldurga	i) Inner Rampart Wall. ii) Entrance Gate.	Building age is around 600 years. The Samples were

S16:Naldurga Fort Wall		iii) Fortification Wall. Type: Stone and Lime Mortar.	extracted using a hammer and chisel.
S13:Holkar Wada Brick Wall S15:Holkar Wada Stone Wall	-NA-	i) Door in the rear courtyard. ii) Plinth of the courtyard. Type: Stone, Brick, Lime and Mud Mortar.	Building age is around 200 years. The samples were extracted through the use of a drill core.

Chapter 3: Laboratory Investigations

In this section the different strategies deployed to carry out the experiments are discussed. This section will comprise of the experimental setup used, list of the chemicals along with the preparation of reagents and the description of the analytical methods which were followed.

3.1 Chemicals used:

Potassium chromate, Hydrochloric acid, Sulphuric acid, Nitric acid, Oxalic acid, Sodium hydroxide, Ethyl alcohol, Acetone, Ammonium chloride, Liquor Ammonia, Calcium carbonate, Sodium carbonate, Silver nitrate, Potassium nitrate, Eriochrome Black T (EBT), Phenolphthalein, Methyl Red, Methyl Orange, Solochrome Dark Blue, Sodium chloride, Magnesium chloride, Potassium sulphate, Barium chloride, Sodium acetate, Glacial acetic acid, Disodium salt of Ethylenediamine tetraacetic acid(di-Na EDTA), Ethylenediamine tetraacetic acid (EDTA).All chemicals were purchased from *Sigma-Aldrich* and used as such except Calcium carbonate, Sodium chloride and Sodium carbonate.

3.2 Equipment/Apparatus:

Burette Stand, Burettes (50 ml and 25 ml), Pipette (10 ml), Auto-pipette(s) (Range 10 μ L-1000 μ L), Conical Flasks, Erlenmeyer Flasks, Storage Bottles(250 ml and 500 ml),Spatulas, Stirring Rod, Magnetic beads, Round-bottom Flasks, Measuring cylinders (25 ml and 50 ml), Hot-plate, Beakers (25 ml,50 ml,100 ml and 250 ml), Glass funnels, Heating Mantles, Filtration Assembly, Falcon tubes (15 ml).Filter paper(Whatman no.1 and Whatman no.41),Stoppers, Colour-coded pH papers, Vacuum desiccator, Plastic tubes, Grinder, Pestel, Sieves.

3.3 Preparation of Reagents:

a) Potassium chromate Indicator solution: 12.5 g of Potassium chromate was dissolved in 150 ml of Milli Q water. Silver nitrate was added to this solution until the formation of a red precipitate. This was kept standing for 12 hours, thereafter filtered using Whatman

No.1 filter paper and then the volume was made up to 250 ml.

b) Standard Silver nitrate solution (0.0141 M): 0.598 g of Silver nitrate was weighed and added to a small amount of Milli Q water and then diluted to 250 ml. This solution was stored in an amber bottle away from the sunlight.

c) Standard Sodium chloride solution (0.0141 M): Sodium chloride salt was taken in a Petri dish and dried in a vacuum oven at 130 °C for six hours. Thereafter; 0.206 g of Sodium chloride was weighed accurately and added to a small amount of Milli Q water and then further diluted to 250 ml. The rest of the salt was then stored in a vacuum desiccator to prevent any contact with air.

d) Buffer solution A for Sulphate test: 15 g Magnesium chloride, 0.5 g Potassium nitrate, 2.5 g Sodium acetate and 10 ml glacial acetic acid were dissolved together in 250 ml of Milli Q water and the volume was made up to 500 ml.

e) Stock Sulphate Solution (1000 ppm): 0.91 g of Potassium sulphate was dissolved in 500 ml of Milli Q water.

f) Phenolphthalein Indicator solution: 0.5 g of Phenolphthalein powder was dissolved in 50 ml of 95 % ethyl alcohol and then the volume was made up to 100 mL using Milli Q water.

g) Sodium Carbonate solution (0.05 N): Sodium carbonate was taken in a Petri dish and dried in a vacuum oven at 130 °C for six hours. Thereafter; 0.625 g of Sodium carbonate was accurately weighed and dissolved in 250 ml of Milli Q water.

h) Standard Sulphuric acid solution (0.1 N): 0.7ml of concentrated Sulphuric acid was measured accurately and then diluted to 250 ml using Milli Q water.

i) Methyl Orange Indicator solution: 0.05 g of Methyl Orange powder was dissolved in Milli Q water and the volume was made up to 100 ml.

j) Methyl Red Indicator solution: 0.2 g of methyl red powder was dissolved in 100 mL solution with ethyl alcohol: Milli Q water ratio 3:2.

k) Solochrome Dark Blue Indicator: 0.2 g of finely powdered dye was dispersed in 100 g Sodium Chloride.

l) Standard EDTA solution (0.01M): 1.862 g of disodium salt of EDTA was dissolved in Milli Q water and the volume was made up to 500 ml.

m) Standard Calcium solution: This solution requires Calcium carbonate which prior to

its use was dried in a vacuum oven at 130 °C for six hours. Thereafter; 0.5 g of Calcium carbonate was taken in an Erlenmeyer flask and concentrated Hydrochloric acid was added until it completely dissolved. Then, 100 ml of Milli Q water was added and the flask was heated on a hot plate for few minutes to expel carbon dioxide gas. After this, the flask was allowed to cool and few drops of Methyl Red indicator solution were added to it. After this the pH of the solution was adjusted by measured additions of 1:1 HCl and Liquor Ammonia so as to achieve an orange-coloured solution.

n) Sodium hydroxide solution (2 N): 20 g of Sodium hydroxide was dissolved in 250 ml of Milli Q water.

o) Ammonia-Ammonium Buffer solution for Total-Hardness Test: 16.9 g of Ammonium chloride was dissolved in 143 ml concentrated Ammonia solution. Simultaneously; 1.179 g EDTA and 0.644g Magnesium chloride were dissolved in 50 ml of Milli Q water. The latter solution was added to the Ammonia-Ammonium Chloride solution and the total amount was diluted to 250 ml.

p) Eriochrome Black T indicator: 0.5 g of finely powdered chemical was dispersed in 100 g of Sodium chloride.

3.4 Sample Preparation:

The collected samples were grinded with the help of a stainless steel mortar and pestle and sieved through a 150 µm sieve. Thus each sample was converted to an aggregate of extremely fine particles. All analyses were performed using these samples as the core material. They were divided into two parts: One for conductivity, pH and alkalinity analyses and the other one for Chloride, Sulphate, Calcium and Magnesium analyses.

(A) Sample Preparation for conductivity, pH and alkalinity measurement:

1 g of grinded mortar sample was accurately weighed and added to 100 ml of Milli Q water. This suspension was collected in an Erlenmeyer flask. A magnetic bead was put inside this flask. This conical flask was placed on a Magnetic Stirrer and the sample was stirred at a speed of about 750 rpm for about 3 hours. After this, the sample was filtered

using Whatman No. 1 filter paper and the filtrate was stored. The undissolved impurities were discarded and this procedure was repeated for all the samples. Conductivity, pH and alkalinity measurements were carried out using the filtrates of the samples.

(B) Sample Preparation for Radical Analysis (Cl^- , Ca^{2+} , Mg^{2+} , SO_4^{2-}):

1 g of grinded mortar sample was accurately weighed and added to a RB Flask. 6 ml of concentrated nitric acid and 94 ml of Milli Q water was added to it. This flask was placed in a heating mantle within a laboratory fume hood and heated till the volume was reduced below 20 ml; Milli Q water was then added to make up the volume to 20 ml and filtered using a filtration assembly connected to a vacuum pump. In this case, Whatman No.41 filter paper was used. The filtrates were collected and stored in Falcon tubes. Chloride, Sulphate, Calcium and Magnesium tests were carried out using the filtrates of the samples.

Rationale behind these protocols: In Method A) the parameters; conductivity, pH and alkalinity would have been affected by the addition of H^+ ions from the acid; thus amounting to erroneous results. In Method B), it is imperative that the constituent mineral components get separated from each other so as to obtain definitive results through conduction of radical tests. The process followed here is called Acid Digestion. In this process, the components of the sample break down and dissolve in the acid. In the initial stages of sampling, both hydrochloric and nitric acid were used in equal ratio for digestion but hydrochloric acid was found to affect the chloride radical test, so only nitric acid was used in the subsequent sample preparations. Besides this, the addition of concentrated nitric had two key advantages. Firstly, the pH of the sample was significantly reduced. This minimized relative adsorption and precipitation at container surface and also prevented microbial degradation. Secondly, nitric acid is a very strong oxidizing agent. Thus it facilitates the dissolution of minerals as well as metal ions in the aqueous medium.

3.5 Sample Analysis:

A) Conductivity Test:

Conductivity of the samples was measured using a **Eutech Con700 Conductivity meter**. All samples showed conductivity below 500 $\mu\text{S}/\text{cm}$. The samples and their corresponding conductivity values are listed down in the Table 4.2 under the Results Section.

B) pH Test:

The pH of the sample solutions were measured using a **Eutech ph1500 ph meter**. The nature of samples was found to be mildly basic with pH in the range of 7-9. The samples and their corresponding pH values are listed down in the Table 4.2 under the results section.

C) Alkalinity Test: As per the APHA standard method [2], alkalinity test was performed.

Procedure: Sulphuric acid was standardized against Sodium carbonate solution. 10 ml of sodium carbonate solution was taken; 40 ml water was added along with few drops of Phenolphthalein and titrated against Sulphuric acid until the pink colour disappeared. After this, 20 ml of filtrate was taken in a conical flask and the volume was made up to 50 ml, Sulphuric acid was taken in the burette. Few drops of Phenolphthalein indicator were added to the solution but there was no appearance of the pink colour. Thereafter, few drops of Methyl Orange indicator were added to the solution and titrated against Sulphuric acid. Burette reading was noted at the end point.

Composition used during testing: 20ml of sample filtrate dissolved in 50 ml of water.

Note that the Sample Blank for this test was Milli Q water. The observation tables are presented below:

Table 3.1a: Burette readings for the Standardization of Sulphuric Acid

Sample Name/Code	Sulphuric Acid burette reading (ml)	Final Burette Reading (ml)
Sodium carbonate solution	4.8	4.8

(10 ml)	4.8	
	4.8	

Table 3.1b: Burette readings for the alkalinity test

Sample Name/Code	Sulphuric Acid burette reading(Point B:Appearance of orange-pink colour) (ml)	Final Burette Reading (ml)
Blank	Instantaneous	Instantaneous
S1	0.4 0.4 0.4	0.4
S2	0.2 0.2 0.2	0.2
S3	0.3 0.3 0.3	0.3
S4	0.3 0.3 0.3	0.3
S5	0.3 0.3 0.3	0.3
S6	0.3 0.3 0.3	0.3
S7	0.3 0.3 0.3	0.3

S8	0.4 0.4 0.4	0.4
S9	0.4 0.4 0.4	0.4
S10	0.3 0.3 0.3	0.3
S11	0.5 0.5 0.5	0.5
S12	0.1 0.1 0.1	0.1
S13	0.3 0.3 0.3	0.3
S14	0.4 0.4 0.4	0.4
S15	0.2 0.2 0.2	0.2
S16	0.3 0.3 0.3	0.3
S17	0.2 0.2 0.2	0.2
S18	0.3	0.3

	0.3	
	0.3	

D) Chloride Test: As per the APHA standard method [3], chloride test was performed.

Procedure: Silver Nitrate solution was standardized against Sodium chloride solution. After this, 0.5 ml of sample digestion filtrate was taken in a conical flask with the help of auto-pipette and the volume was made up to be 50 ml, Silver Nitrate was taken in the burette. 1 ml of Potassium chromate indicator was added to the solution which resulted in a yellow colour. This solution was titrated against the Silver nitrate solution .A colour change from faint yellow to brick red was observed. The titrant volume was recorded at the point of colour change and the subsequently the burette readings were recorded for all the mortar samples.

Composition used during testing: 0.5 ml of sample filtrate dissolved in 50 ml of water.

The burette readings are presented in the tables below:

Table 3.2a: Burette readings for the Standardization of Silver nitrate solution

Sample Name/Code	Sulphuric Acid burette reading (ml)	Final Burette Reading (ml)
Sodium chloride solution (10 ml)	10.6	10.6
	10.6	
	10.6	

Table 3.2b: Burette readings for the chloride test

Sample Name/Code	Silver nitrate burette reading(Appearance of Brick red colour) (ml)	Final Burette Reading (ml)
Blank	0.2	0.2
	0.2	

	0.2	
S1	0.8 0.8 0.8	0.8
S2	0.9 0.9 0.9	0.9
S3	1.1 1.1 1.1	1.1
S4	0.6 0.6 0.6	0.6
S5	0.7 0.7 0.7	0.7
S6	1.0 1.0 1.0	1.0
S7	0.3 0.3 0.3	0.3
S8	0.8 0.8 0.8	0.8
S9	1.0 1.0 1.0	1.0
S10	1.1 1.1 1.1	1.1

S11	0.6 0.6 0.6	0.6
S12	0.9 0.9 0.9	0.9
S13	0.6 0.6 0.6	0.6
S14	0.7 0.7 0.7	0.7
S15	0.8 0.8 0.8	0.8
S16	0.9 0.9 0.9	0.9
S17	1.1 1.1 1.1	1.1
S18	1.0 1.0 1.0	1.0

E) Sulphate Test: As per the APHA protocol [4], sulphate test was performed with the use of a **HIMADZU UV-1900 UV-Vis Spectrophotometer**.

Procedure: 100 μ L of sample filtrate was taken in a falcon tube and the volume was made up to 10 ml, 2 ml of Buffer Solution A was added to this along with a pinch of Barium chloride. A white turbidity appeared. This was placed on a vortex for 1 min

under a constant speed

This test resulted in a failure, so only basic details and procedure are mentioned here.

Presumed reasons for failure:

- 1) This test requires a UV Spectrophotometer which is a highly sensitive instrument. Also this test only detects sulphate ions in a relatively narrow range of concentration.
- 2) Due to the pandemic, appropriate amount of time could not be given for multiple repetitions.

F) Calcium Test: As per the APHA protocol [5], Calcium test was performed.

Procedure: EDTA solution was standardized against Standard Calcium solution. 10 ml of Standard Calcium solution was taken; 40 ml water was added along with 1 ml of Ammonium-ammonia buffer. A pinch of EBT was added to this solution and it was titrated against EDTA solution to a blue colour end-point. After this, 100 µL of sample filtrate was taken in a conical flask with the help of auto-pipette and the volume was made up to be 50 ml. To this, 1 ml of Sodium hydroxide solution was added. A magnetic stirrer was placed below the conical flask and a magnetic bead was put in it. Standard EDTA solution was taken in the burette. A pinch of Solochrome Dark Blue indicator was added to the sample solution. This solution was titrated against the Standard EDTA solution. A colour change from pink to blue was observed. The titrant volume was recorded at the point of colour change and the subsequently the burette readings were recorded for all the mortar samples. Note that the Calcium and Total (Calcium and Magnesium) tests were performed using the same standardized EDTA solution. So data listed in Table 3.3a is also usable for the Total Test for Calcium and Magnesium.

Composition used during testing: 0.1 ml of sample filtrate dissolved in 50 ml of water.

The observation tables are presented below:

Table 3.3a: Burette readings for the Standardization of EDTA solution

Sample Name/Code	EDTA burette reading (ml)	Final Burette Reading (ml)

Standard Calcium solution (10 ml)	10.9	10.9
	10.9	
	10.9	

Table 3.3b: Burette readings for the Calcium test

Sample Name/Code	EDTA burette reading(Appearance of Blue colour) (ml)	Final Burette Reading (ml)
Blank	0.3	0.3
	0.3	
	0.3	
S1	3.0	3.0
	3.0	
	3.0	
S2	3.6	3.6
	3.6	
	3.6	
S3	3.3	3.3
	3.3	
	3.3	
S4	3.0	3.0
	3.0	
	3.0	
S5	2.6	2.6
	2.6	
	2.6	
S6	3.0	3.0
	3.0	
	3.0	
S7	3.2	3.2

	3.2 3.2	
S8	3.5 3.5 3.5	3.5
S9	2.9 2.9 2.9	2.9
S10	3.5 3.5 3.5	3.5
S11	2.3 2.3 2.3	2.3
S12	3.0 3.0 3.0	3.0
S13	2.8 2.8 2.8	2.8
S14	2.3 2.3 2.3	2.3
S15	3.1 3.1 3.1	3.1
S16	2.5 2.5 2.5	2.5
S17	3.5 3.5	3.5

	3.5	
S18	2.1 2.1 2.1	2.1

Few key points/modifications:

- 1) After Sodium Hydroxide solution is added, drop a colour-coded pH paper to check whether the pH of the solution is above 9.
- 2) Solochrome Dark Blue indicator may not provide an effective visible colour change if added in small amounts. Adjust this amount through trial and error.

G) Total Test for Calcium and Magnesium: As per the APHA protocol [6], Total Hardness test was performed for determination of Magnesium content in the samples.

Procedure: EDTA solution was standardized against Standard Calcium solution. 10 ml of Standard Calcium solution was taken; 40 ml water was added along with 1 ml of Ammonium-ammonia buffer. A pinch of EBT was added to this solution and it was titrated against EDTA solution to a blue colour end-point. After this, 100 μ L of sample filtrate was taken in a conical flask with the help of auto-pipette and the volume was made up to be 50 ml. To this, 1ml of Ammonia-ammonium buffer solution was added. A magnetic stirrer was placed below the conical flask and a magnetic bead was put in it. Standard EDTA solution was taken in the burette. A pinch of Eriochrome Black T indicator was added to the sample solution. This solution was titrated against the Standard EDTA solution .A colour change from wine-red to blue was observed. The titrant volume was recorded at the point of colour change and the subsequently the burette readings were recorded for all the mortar samples.

Composition used during testing: 0.1 ml of sample filtrate dissolved in 50 ml of water.

The burette readings are presented in the table below:

Table 3.4: Burette readings for the Total Test for Calcium and Magnesium

Sample Name/Code	EDTA burette reading(Appearance of Blue colour) (ml)	Final Burette Reading (ml)
Blank	0.6 0.6 0.6	0.6
S1	3.8 3.8 3.8	3.8
S2	4.4 4.4 4.4	4.4
S3	4.2 4.2 4.2	4.2
S4	3.7 3.7 3.7	3.7
S5	3.3 3.3 3.3	3.3
S6	3.9 3.9 3.9	3.9
S7	4.3 4.3 4.3	4.3
S8	4.1 4.1 4.1	4.1
S9	3.8	3.8

	3.8 3.8	
S10	4.3 4.3 4.3	4.3
S11	3.0 3.0 3.0	3.0
S12	3.7 3.7 3.7	3.7
S13	3.8 3.8 3.8	3.8
S14	3.1 3.1 3.1	3.1
S15	3.9 3.9 3.9	3.9
S16	3.2 3.2 3.2	3.2
S17	4.2 4.2 4.2	4.2
S18	2.9 2.9 2.9	2.9

3.6 X-Ray Tests:

A) X-Ray Diffraction Test:

Powder diffraction technique is useful for the structural characterization of mortar samples. The instrument used for this test was a **Rigaku MiniFlex 600 XRD Machine**. For this test, about 200 mg of each grinded sample was collected in separate vials and then used directly without any additives. The data obtained from the XRD test was used to plot graphs with the help of Origin Software. Kindly refer Fig (4.1-4.18) for the same.

Instrument Limitation: The instrument was able to detect corresponding intensity values for 2Θ in the range of (10-90) degrees. Any compounds that have their peak values outside this range were not detected.

B) XRF Test:

XRF test is useful for the determination of constituent elements in a sample. The instrument which was used for this test was a **Bruker S8 TIGER XRF Machine**. This test required sample processing. The protocol is described below:

1) About 6 g of each finely grinded sample was collected in separate falcon tubes and deposited to the concerned laboratory. Initially, the samples were gradually heated to about 1000 °C for about 4 hours in a muffle furnace. Quartz crucibles were used as containers. Their weight before and after heating was measured which helped to determine the Loss of Ignition (Refer Table 4.1a and Table 4.1b)

2) After cooling, the samples were taken to another furnace. The samples were mixed with a flux material (Lithium meta-tetra borate) in the ratio of about 1: 17. They were then inserted in platinum crucibles. Here, the heating is quick and the temperature rises to about 1000 °C. As a result, a melt is formed which is then dropped in a mold leading to the formation of fusion beads.

3) These beads were then used in the XRF machine and based on the available standards; data regarding the relative proportion of different elemental components constituting the samples was obtained. Kindly refer (Table 4.1a and Table 4.1b) for the same.

Instrument Limitation: The sum with LOI can be in the range of 95-105. This is presumably due to over or under-quantification of certain constituents of the samples.

Chapter 4: Test data and Results

4.1 XRF test:

The XRF test results indicated that the primary components of the samples were calcium based compounds along with relatively lesser amounts of silica. Besides this, compounds of elements like Magnesium, Aluminum, Sodium, Phosphorous, Potassium, Vanadium, Titanium, Manganese, Nickel, Zinc, Barium, Chromium and Sulphur were found in trace amounts. The relative proportions of every component are presented in the two tables below:

Table 4.1a: XRF Data

Sample Code	Sum with LOI	LOI at 900°C	CaO (%)	SiO ₂ (%)	Al ₂ O ₃ (%)	Fe ₂ O ₃ (%)	MgO (%)	Na ₂ O (%)	K ₂ O (%)	SO ₃ (%)
S1	103	32.24	37.719	16.447	5.611	5.138	2.959	0.441	0.461	0.455
S2	106	38.02	45.308	11.024	3.905	2.930	2.305	0.269	0.322	0.321
S3	103	35.10	42.275	13.016	4.278	3.545	3.169	0.282	0.161	0.127
S4	97	35.15	38.398	12.727	4.133	2.978	2.211	0.218	0.225	0.152
S5	104	29.78	33.803	20.767	5.657	6.183	2.943	0.316	0.870	0.332
S6	104	33.86	37.946	16.422	5.426	4.161	3.772	0.446	0.358	0.145
S7	103	36.94	41.553	11.482	3.873	2.735	4.924	0.142	0.229	0.159
S8	103	36.54	43.815	11.199	4.280	3.892	1.413	0.054	0.199	0.173
S9	103	31.38	36.229	17.761	5.677	5.743	3.382	0.360	0.512	0.212
S10	104	37.63	43.716	11.769	3.779	2.649	2.124	0.201	0.348	0.225
S11	103	28.54	30.838	22.049	6.115	7.002	3.773	0.618	0.533	0.532
S12	105	36.90	38.179	15.872	4.542	3.788	1.826	0.547	0.851	0.299
S13	103	31.02	35.699	18.368	5.494	4.463	5.033	0.457	0.328	0.187
S14	103	29.95	31.082	19.887	6.920	6.431	5.346	0.258	0.385	0.506

S15	103	35.30	38.653	15.339	2.775	3.049	2.775	0.555	0.661	0.753
S16	104	29.55	32.363	20.965	2.551	7.512	2.551	0.496	0.325	0.217
S17	103	39.37	43.311	9.863	1.667	3.032	1.667	0.270	0.365	0.270
S18	103	30.40	28.278	22.442	5.734	5.625	5.734	0.401	0.362	0.276

Table 4.1b: XRF Data

Sample Code	P ₂ O ₅ (%)	V ₂ O ₅ (%)	NiO (%)	TiO ₂ (%)	Cr ₂ O ₃ (%)	Mn ₂ O ₃ (%)	ZnO (%)	ZrO ₂ (%)	BaO (%)
S1	0.159	0.0003	0.0233	0.934	0.0123	0.1028	0.0075	0.058	0.724
S2	0.183	0.0000	0.0215	0.518	0.0145	0.0630	0.0030	0.062	0.380
S3	0.095	0.0000	0.0210	0.577	0.0140	0.0620	0.0040	0.039	0.417
S4	0.130	0.0010	0.0200	0.531	0.0140	0.0620	0.0050	0.048	0.402
S5	0.318	0.0080	0.0240	1.418	0.0110	0.2340	0.0100	0.104	1.131
S6	0.231	0.0000	0.0210	0.689	0.0200	0.0620	0.0050	0.076	0.528
S7	0.278	0.0000	0.0210	0.354	0.0130	0.1480	0.0050	0.086	0.241
S8	0.238	0.0010	0.0260	0.589	0.0120	0.3440	0.0090	0.077	0.516
S9	0.215	0.0010	0.0220	0.975	0.0110	0.0915	0.0030	0.072	0.752
S10	0.313	0.0000	0.0223	0.457	0.0088	0.0848	0.0070	0.095	0.339
S11	0.206	0.0090	0.0210	1.377	0.0100	0.0700	0.0030	0.073	1.092
S12	0.352	0.0000	0.0230	0.719	0.0050	0.0910	0.0080	0.107	0.564
S13	0.171	0.0000	0.0230	0.682	0.0240	0.0620	0.0100	0.061	0.515
S14	0.198	0.0050	0.0220	1.213	0.0110	0.1200	0.0060	0.074	0.951
S15	0.521	0.0000	0.0200	0.495	0.0140	0.0620	0.0070	0.146	0.380
S16	0.191	0.0078	0.0223	1.447	0.0098	0.0760	0.0080	0.071	1.132
S17	0.165	0.0000	0.0220	0.611	0.0040	0.0980	0.0085	0.062	0.462
S18	0.443	0.0020	0.0210	0.771	0.0250	0.1460	0.0130	0.131	0.593

4.2 Conductivity test:

For the data obtained from the conductivity test kindly refer to Table 4.2.

4.3 pH Test:

The data obtained from the pH test is tabulated in Table 4.2.

Note: For the tests (4.4-4.7), the values obtained post-calculations are tabulated in Table 4.2.

4.4 Alkalinity test:

Calculations:

1) During Standardization, the normality of Sulphuric acid (N) was determined.

It is given by the formula: $N = 10 \times 0.05 / Y$

Here Y = Total volume of Sulphuric acid required for titration.

2) After this, Alkalinity can be determined by the formula:

Total Alkalinity as CaCO_3 (mg/L) = $\{(A+B) \times N \times 50000\} / \text{Volume of sample}$

Here A = Volume of Acid used for colour change after use of Phenolphthalein Indicator.

B = Volume of Acid used for colour change after use of Methyl Orange Indicator.

N = Normality of the Acid used.

4.5 Chloride test:

Calculations:

1) During Standardization, the normality of Silver nitrate solution (N) was determined.

It can be obtained by the formula: $NV = N_1V_1$

Here, N = Normality of Silver nitrate solution.

V = Volume of Silver nitrate solution recorded at end point.

N_1 = Normality of Sodium chloride solution.

V_1 = Volume of Sodium chloride solution taken in the conical flask.

2) After this, the Chloride content can be determined by the formula:

Cl^- (g/Kg) = $[(A-B) \times 35450 \times N] / V / 50$.

Here, A = Volume of titrant required for sample.

B = Volume of Blank required for sample.

N = Normality of Silver nitrate solution.

V = Volume of sample taken in ml.

4.6 Calcium test:

Calculations:

1) During Standardization, the calcium carbonate equivalent of 1 ml EDTA was determined.

It can be obtained using the formula:

CaCO₃ equivalent of 1 ml EDTA(Y) = 10/X where X = Required volume of EDTA.

2) After this, the Calcium content can be determined by the formula:

Calcium hardness as CaCO₃ (g/kg) = $\frac{[(A-B)*Y*1000]}{V}/50$.

Here, A = Volume of titrant required for sample.

B = Volume of titrant required for Blank.

V = Volume of Sample taken in ml.

Y = CaCO₃ equivalent of 1 ml EDTA.

4.7 Magnesium test:

Before determining the Magnesium content, total hardness of the sample was obtained.

Calculations:

1) During Standardization, the calcium carbonate equivalent of 1 ml EDTA was determined.

It can be obtained using the formula:

CaCO₃ equivalent of 1 ml EDTA(Y) = 10/X where X = Required volume of EDTA.

2) After this, the Total Hardness can be determined by the formula:

Total hardness as CaCO₃ (g/kg) = $\frac{[(A-B)*Y*1000]}{V}/50$.

Here, A = Volume of titrant required for sample.

B = Volume of titrant required for Blank.

V = Volume of Sample taken in ml.

Y = CaCO₃ equivalent of 1 ml EDTA.

3) The Hardness due to Magnesium is calculated using the formula:

Magnesium Hardness as CaCO₃ (g/kg) {Z} = (Total Hardness – Calcium Hardness)
(g/kg)

4) Amount of Magnesium (Mg²⁺) = 0.243*Z.

Table 4.2: Results of the laboratory wet tests

Sample Code	Conductivity (μS)	pH	Total Alkalinity as CaCO_3 (mg/L)	Chloride (g/kg)	Calcium hardness as CaCO_3 (g/kg)	Magnesium (g/kg)
S1	271.80	8.15	104	11.32	495.18	22.28
S2	76.40	7.68	52	13.20	605.22	22.28
S3	76.30	7.67	78	16.97	550.20	26.74
S4	82.10	8.14	78	7.54	495.18	17.83
S5	95.00	8.10	78	9.43	421.82	17.83
S6	74.20	8.10	78	15.09	495.18	26.74
S7	72.70	8.27	78	9.43	531.86	35.65
S8	79.00	8.27	104	11.32	586.88	13.37
S9	83.43	8.34	104	15.09	476.84	26.74
S10	106.60	8.27	78	16.97	586.88	22.28
S11	262.90	8.23	130	7.54	366.80	17.83
S12	342.50	7.73	26	13.20	495.18	17.83
S13	66.80	7.95	78	7.54	458.50	31.20
S14	85.70	8.02	52	9.43	366.80	22.28
S15	224.30	7.99	52	11.32	513.52	22.28
S16	113.80	7.93	78	13.20	403.48	17.83
S17	234.60	7.75	52	16.97	586.88	17.83
S18	106.60	8.02	78	15.09	330.12	22.28

4.8 XRD test:

Based on the data, XRD Graphs were plotted for all the samples and analysis of these peaks confirmed the presence of CaCO_3 , Calcite and Silicon Oxide.

Note: The hkl values along with their corresponding compounds are written on the graph itself.

The XRD plots of the samples are presented below:

a) Sample S1

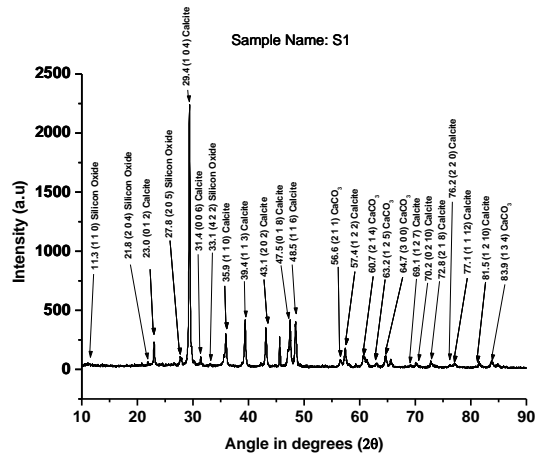


Fig 4.1: Powder-XRD plot of Sample S1

JCPDS Ref. Numbers: Silicon Oxide: 88-1155; Calcite: 47-1743; CaCO₃: 85-1108.

b) Sample S2

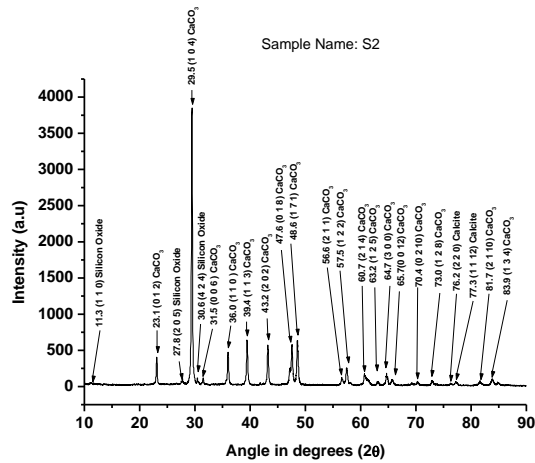


Fig 4.2: Powder-XRD plot of Sample S2

JCPDS Ref. Numbers: Silicon Oxide: 88-1155; CaCO₃: 85-1108.

c) Sample S3

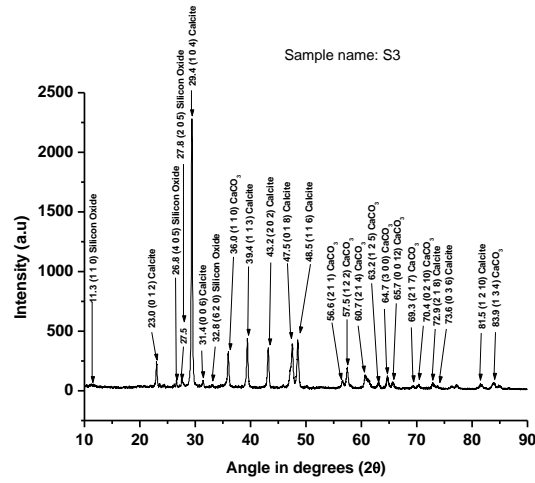


Fig 4.3: Powder-XRD plot of Sample S3

JCPDS Ref. Numbers: Silicon Oxide: 88-1155; Calcite: 47-1743; CaCO₃: 85-1108.

d) Sample S4

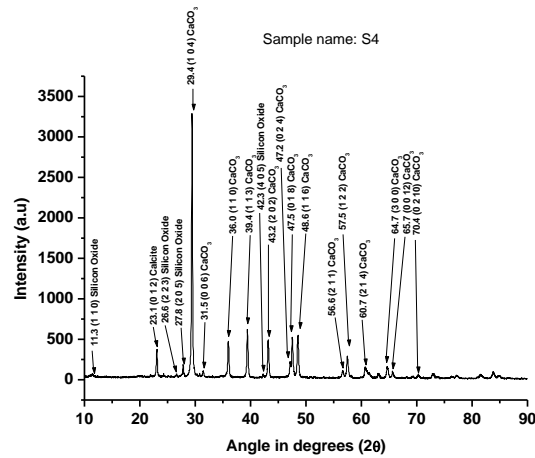


Fig 4.4: Powder-XRD plot of Sample S4

JCPDS Ref. Numbers: Silicon Oxide: 88-1155; CaCO₃: 85-1108.

e) Sample S5

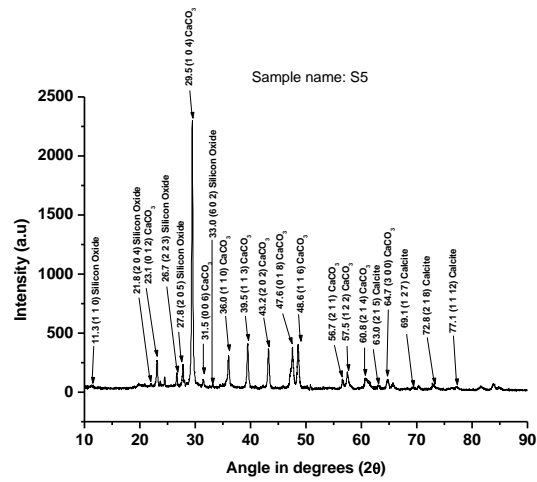


Fig 4.5: Powder-XRD plot of Sample S5

JCPDS Ref. Numbers: Silicon Oxide: 88-1155; Calcite: 47-1743.

f) Sample S6

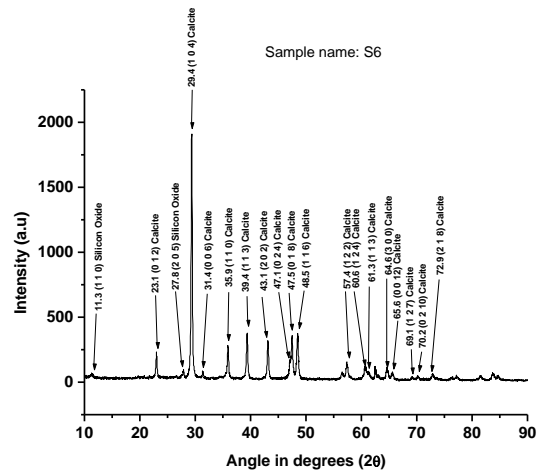


Fig 4.6: Powder-XRD plot of Sample S6

JCPDS Ref. Numbers: Silicon Oxide: 88-1155; Calcite: 47-1743.

g) Sample S7

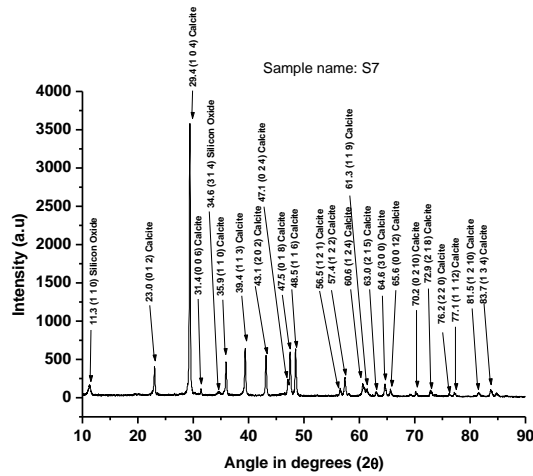


Fig 4.7: Powder-XRD plot of Sample S7

JCPDS Ref. Numbers: Silicon Oxide: 88-1155; Calcite: 47-1743.

h) Sample S8

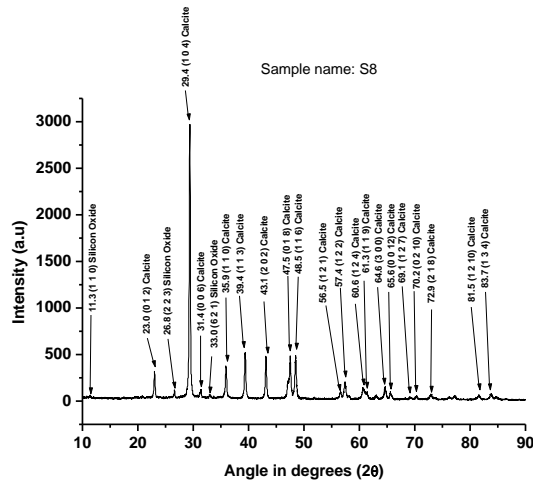


Fig 4.8: Powder-XRD plot of Sample S8

JCPDS Ref. Numbers: Silicon Oxide: 88-1155; Calcite: 47-1743.

i) Sample S9

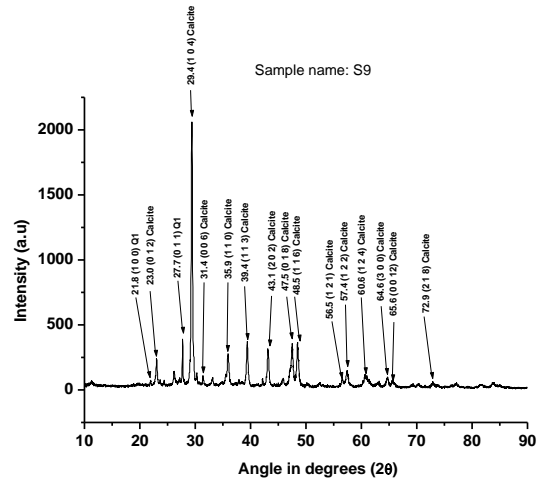


Fig 4.9: Powder-XRD plot of Sample S9

JCPDS Ref. Numbers: Q1 (Quartz low): 86-1565; Calcite: 47-1743.

j) Sample S10

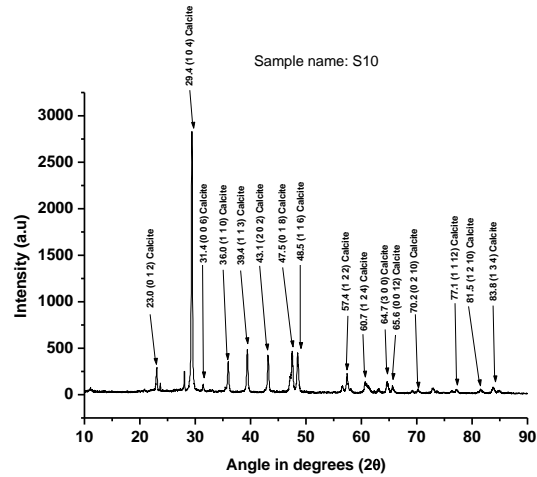


Fig 4.10: Powder-XRD plot of Sample S10

JCPDS Ref. Numbers: Calcite: 47-1743.

k) Sample S11

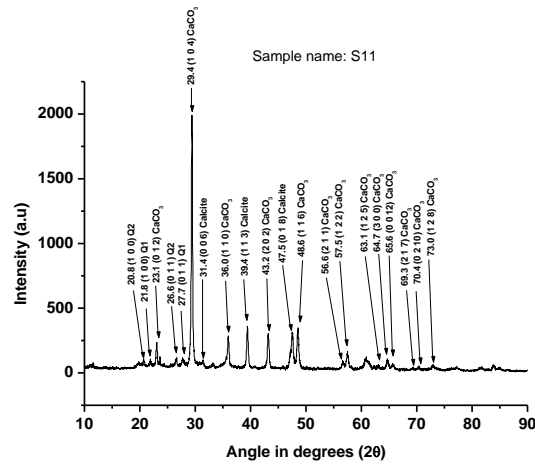


Fig 4.11: Powder-XRD plot of Sample S11

JCPDS Ref. Numbers: Calcite: 47-1743; Q1 (Quartz low): 86-1565; CaCO₃: 85-1108; Q2 (Quartz pha): 89-8934.

l) Sample S12

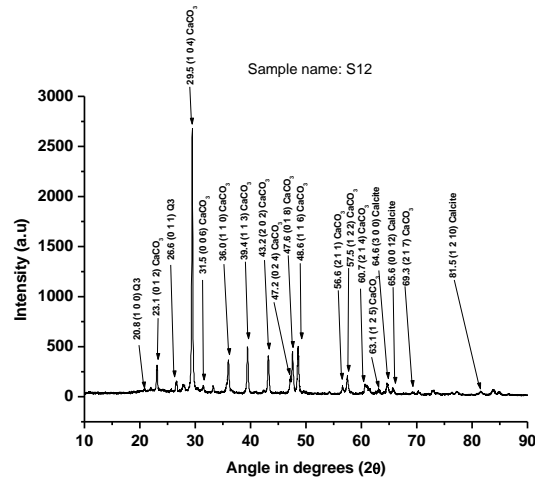


Fig 4.12: Powder-XRD plot of Sample S12

JCPDS Ref. Numbers: Calcite: 47-1743; Q3 (Quartz low): 87-2096; CaCO₃: 85-1108;

m) Sample S13

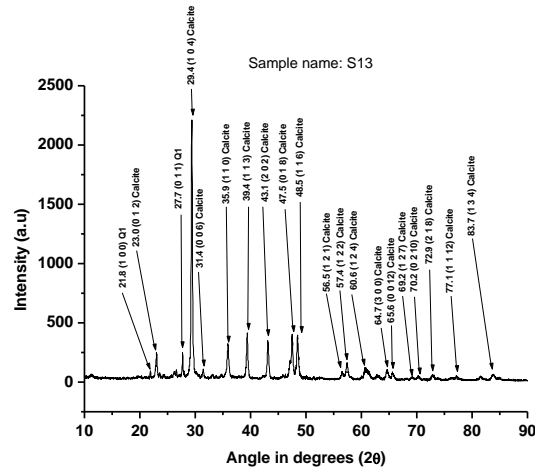


Fig 4.13: Powder-XRD plot of Sample S13

JCPDS Ref. Numbers: Calcite: 47-1743; Q1 (Quartz low): 86-1565.

n) Sample S14

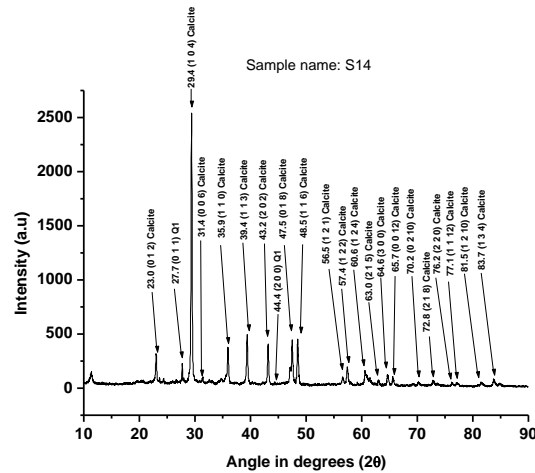


Fig 4.14: Powder-XRD plot of Sample S14

JCPDS Ref. Numbers: Calcite: 47-1743; Q1 (Quartz low): 86-1565.

o) Sample S15

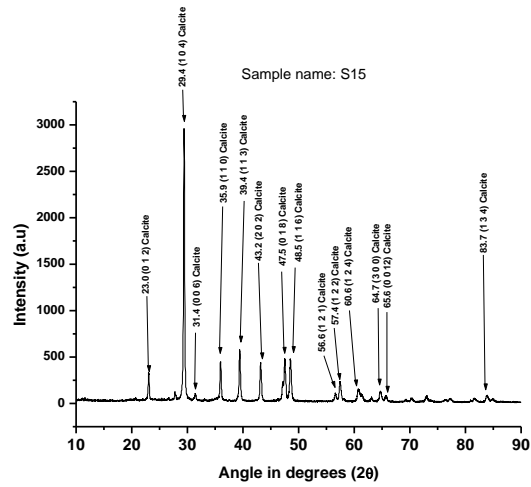


Fig 4.15: Powder-XRD plot of Sample S15

JCPDS Ref. Numbers: Calcite: 47-1743.

p) Sample S16

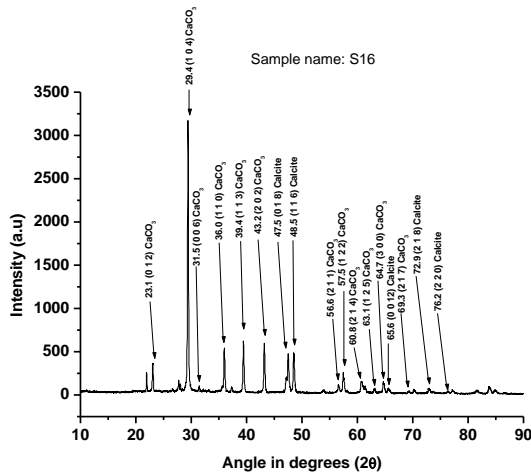


Fig 4.16: Powder-XRD plot of Sample S16

JCPDS Ref. Numbers: Calcite: 47-1743; CaCO_3 : 85-1108.

q) Sample S17

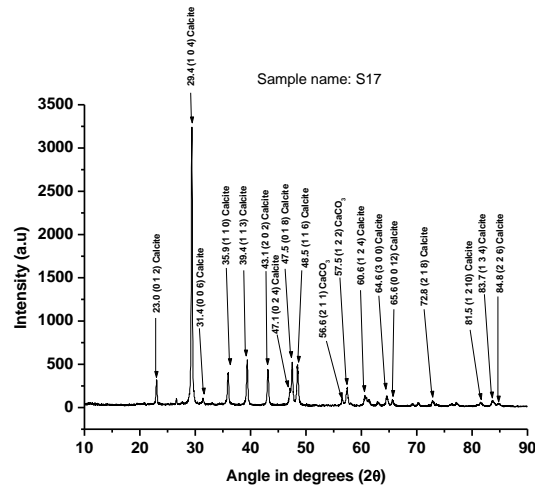


Fig 4.17: Powder-XRD plot of Sample S17

JCPDS Ref. Numbers: Calcite: 47-1743; CaCO₃: 85-1108.

r) Sample S18

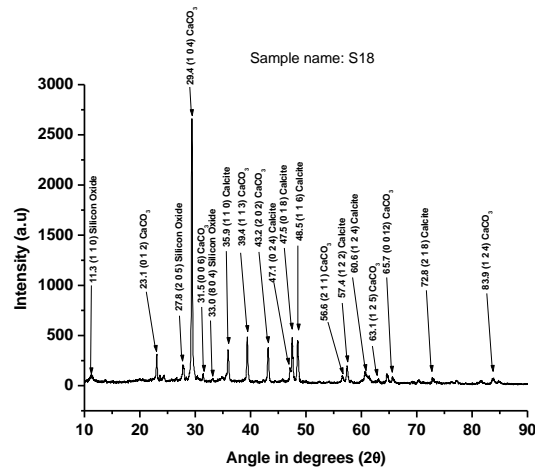


Fig 4.18: Powder-XRD plot of Sample S18

JCPDS Ref. Numbers: Silicon Oxide: 88-1155; Calcite: 47-1743; CaCO₃: 85-1108.

Chapter 5: Discussion and Conclusions

Kindly note that this research didn't cover the study of organic components in the mortar samples. Their relative proportion is definitely less but to what extent they would affect the physical and chemical properties cannot be commented based on the obtained results. Apart from that, the range and mean of different parameters concerning the mortar samples are presented in the table below:

Table 5.1: Parameters and their range values

Parameter	Approximate Range	Approximate Mean
Age of construction	200-600 years	-NA-
Conductivity	70-350 μ S	136 μ S
pH	7.6-8.3	8.0
Alkalinity	20-130 mg/L	77 mg/L
Chloride content	7.5-17 g/kg	12.3 g/kg
Calcium content	330-600 g/kg	487 g/kg
Magnesium content	10-35 g/kg	22.3 g/kg

From the table it can be seen that the pH values for almost all the samples were less than 8.3. Even in the alkalinity test, there was no change in colour due by addition of phenolphthalein indicator. Thus, we can infer that the basic nature is primarily due to carbonate and not due to hydroxyl ions. The XRF tests indicated the presence of Calcium oxide but the p-XRD analysis confirmed the presence of Calcite which is a naturally occurring mineral form of Calcium carbonate and Calcium carbonate. Also, it is known that at high temperatures Calcite and Calcium carbonate convert to Calcium oxide with the release of carbon dioxide. In the XRF test, the samples were heated to a temperature around 1000 °C. Thus our findings are consistent in regards to this. Silica is found to at around 15-25 % by relative proportion and Calcium carbonate/Calcite or a mixture of both being the primary component of the samples. Also it is observed that factors like location, nature of monument and period do not correspond to any

significant deviation in the material composition. It is found to be fairly consistent. Note that this study covered the samples extracted from historical monuments; primarily in the Maharashtra state. Based on these findings, if a restoration project is ever undertaken for monuments within this region; the results from this study may help in deciding the composition of the mortar to be used for restoration work. However depending upon the fragility of the monument it is recommended that a study of organic binders should be conducted before assigning the composition of the mortar.

References:

1) Antonia Moropoulou, Asterios Bakolas, Katerina Bisbikou, 'Investigation of the technology of historic mortars' in

Journal of Cultural Heritage, Volume 1, Issue 1 (2000), pp. 45-58.

2) APHA Standard Methods 2320 B; 22nd edition 2012.

3) APHA Standard Methods 4500-Cl⁻; 22nd edition 2012.

4) APHA Standard Methods 4500-SO₄²⁻; 22nd edition 2012.

5) APHA Standard Methods 3500-Ca B; 22nd edition 2012.

6) APHA Standard Methods 2340 C; 22nd edition 2012.