# Stabilization of Expansive Soil Using Wheat Straw Ash and Calcium

## **Carbide Residue Composite**



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# **Calcium Carbide Residue Composite**



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# DEDICATION

I dedicate this research to my beloved parents for their endless love, support,

encouragement and prayers.

Strive not to be a success, but rather to be of value.

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### ABSTRACT

Expansive soil is a problematic soil. It has properties of expansion, shrinkage, plasticity and low bearing capacity, which makes them inadequate for use in construction. Two industrial wastes, Calcium carbide residue (CCR) obtained from acetylene gas plants and Wheat straw ash (WSA) obtained from Oil mill boiler, are used as composite binder for stabilization of expansive soil. The influential factors studied were binder ratio and curing time. At first, expansive soil was stabilized with WSA, to find the optimum WSA content based on UCS results. It was found out to be 12.5% and was fixed as total binder content in this study. The (CCR:WSA) binder was added to soil in ratios of 0:100, 10:90, 25:75, 50:50 and 75:25, by replacing WSA with CCR in binder. Experimental investigations were done to study the compaction, plasticity, strength and deformation behavior of soil stabilized with composite binder (CCR:WSA). Microstructural analysis was also performed to study the mineralogical and morphological modifications happened during stabilization process. XRD, SEM and EDX tests were done for this purpose. It was observed that plasticity index, swelling pressure and swelling percent decreased after addition of CCR:WSA binder in expansive soil. Shear strength and unconfined compressive strength (UCS) increased after stabilization with composite binder. UCS of CCR:WSA stabilized soil increased by 35 times the original soil. SEM, EDX and XRD validate the findings of UCS and direct shear test. These tests have shown that the strength increase is caused by development of cementitious hydrates which are produced as result of reaction between Calcium hydroxide from CCR and (SiO2 + Al2O3) from WSA and soil. CCR:WSA ratio of 75:25 was recommended for obtaining the best results for stabilization of expansive soil. This study has proved that (CCR:WSA) composite binder is an effective, economical and environment friendly solution for stabilization of expansive soil.

**Keywords:** Soil Stabilization, Expansive soil, Calcium Carbide Residue, Wheat Straw Ash, Pozzolanic Reaction, Strength, Stress-strain response.

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# LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
USCS	Unified Soil Classification System
FAO	Food and Agriculture Organization
SDG	Sustainable Development Goals
CCR	Calcium Carbide Residue
WSA	Wheat Straw Ash
OPC	Ordinary Portland Cement
PSD	Particle Size Distribution
MDD	Maximum Dry Density
OMC	Optimum Moisture Content
UCS	Unconfined Compressive Strength
DDL	Double Diffuse Layer
PL	Plastic Limit
LL	Liquid Limit
PI	Plasticity Index
XRF	X-ray Fluorescence
XRD	X-ray Diffraction
SEM	Scanning Electron Microscopy
EDS	Energy-Dispersive X-ray Spectroscopy

## **CHAPTER 1: INTRODUCTION**

### 1.1 General

Sustainability has become an important factor in Construction industry. In construction sector, new ideas have emerged which are aimed at reducing the impact of construction on environment [1]. After industrial revolution, there has been rapid increase in living standards of society. This resulted in increase in production of waste and industrial byproducts [2]. It was estimated that nearly 8 billion people are responsible for 2.5 billion ton of waste in the current year. It was found that United States is generating 808 kg of waste per capita. In developing countries, most of waste is disposed of in open spaces, which create problems for human health and environment [3]. Anthropogenic activities have propagated a chaos in sustainability of earth [4].

Concrete is most widely used man made material on planet. Cement which is an integral component of concrete is responsible for 6% of global CO<sub>2</sub> release [5]. Construction industry is responsible for 23% of global CO<sub>2</sub> emission [6]. This CO<sub>2</sub> is causing climate change and global warming related issues. South Asia is the most impacted region by climate change [7]. Agriculture is one of most climate sensitive area of economy. Pakistan is an agricultural country. Much of country population depends directly or indirectly on agriculture for living. Global climate risk index places Pakistan at 7<sup>th</sup> position in list of most vulnerable nations due to its geographic and climatic characteristics [8].

Large quantity of agricultural waste is generated annually. These waste harm to environment and also reduce the precious land available for landfill applications. These wastes are used to generate electricity and as heating fuel for different industries. Some leftover agricultural wastes in field are burned in open, which causes release of harmful gases and particulate matter. It was estimated that open field burning of biomass accounted for 33.4% of total biomass burned in Asia [6]. Indian and China were main contributors to open field burning of biomass. It was found that open burning of leftover wheat straw caused the release of harmful gases in environment. Punjab and Sindh were most affected by this process [9]. The open burning of agricultural leftover waste creates the problem of smog and pollutes the air. This causes hazardous effect on human health.



Figure 1: Wheat Straw open field burning in village [10]

According to FAO(2017), global wheat production was 770 million tones. In the same year, the wheat output of Pakistan amounted to 27 million tons. It was estimated that for every kilogram of wheat produced, 1.5 kg of wheat straw waste is gained [11]. Wheat Ash had been used for stabilization of problematic soils by many researchers [11] - [12]. Wheat straw when burnt, is very rich in SiO<sub>2</sub>. It has good pozzolanic characteristics. It was reported that wheat straw ash (WSA) calcined at 600°C showed the optimal pozzolanic performance [13]. Rapid urbanization is causing an increase in industrial solid waste generation. These industrial residues should be used in a sustainable way to reduce impact of environment. Calcium Carbide Residue (CCR) is produced as a waste product of Acetylene gas facility. The annual output of CCR on global scale is 1423 kilo tons [14]. It possessed traits like commercial lime and was also non-hazardous. It has been reported that CCR exhibited outstanding mineralogical and physiochemical properties. Which makes it a cost effective and environmentally friendly alternative for improvement of soil [16].



Figure 2: Environmental Hazards Caused by CCR [15]

Expansive soil has swelling minerals. It consists of hydrophilic clay minerals, like smectite. It undergoes expansion when water is added and shrink when water is removed. It is very hard in dry state as compared to wet state. Its nature of expansion depends upon on initial water content, dry density, particle size and type of mineral. This soil exhibits a plastic and compressible character. These characteristics causes the development of cracks and differential settlement on structure built on expansive soil [3]. This study focuses on stabilization of expansive soil with WSA and CCR. Currently there is no research done to study combined effect of WSA and CCR on stabilization of problematic soil.

### 1.2 Problem Statement

Expansive soil is characterized by swelling, compressibility and low bearing capacity. Pakistan has huge reserves of expansive soil. CCR has been used with other agricultural waste ashes (rice husk ash, sugarcane straw ash & biomass ash) for stabilization of problematic soils. Due to significant amount of amorphous silica and pozzolanic properties, WSA has been used soil stabilization and as replacement of cement and sand in concrete.

Currently, research regarding application of CCR and WSA as composite binder, in soil stabilization, is insufficient and limited. This study aims to assess the viability of using CCR and WSA binder for soil stabilization. The successful soil stabilization will provide a sustainable, economic and safe solution for stabilization of expansive soil.

### 1.3 Research Objectives

This research aims to achieve the following objectives;

- I. Investigation of geotechnical characteristics of untreated expansive soil
- Investigation of compaction & plasticity behavior of soil treated with CCR-WSA.

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- III. Determination of strength & deformation properties of soil treated with CCR-WSA.
- IV. Microstructural characterization of expansive soil treated with CCR-WSA.

## 1.4 Scope of Research

This study will help mitigating problems related with expansive soil. It can be used in foundation construction over soft compressible soil, reducing pavement subgrade subsidence, slope stabilization, embankment construction and dam stability. It can be used for waste management of CCR and WSA. This study gives safe, sustainable and economic binder for stabilization of expansive soil. This study investigates the plasticity, compaction, strength and deformation characteristics of composite binder from CCR and WSA for expansive soil stabilization.

Pakistan is severely affected by climate change and global warming. Sustainable development goals have made great focus on environmental sustainability and human health. The SDG policy encourages the mitigation of waste by widespread waste reuse and recycling actions. Sustainable development goal (SDG-11) is directly related with solid waste collection and management. The aim of this study is to reuse the wastes products of CCR & WSA for improvement of expansive soil which will help in waste utilization and fulfilling the geotechnical applications altogether. Pakistan has an extensive cover of expansive soil. This research will provide sustainable and economic solution.

#### 1.5 Thesis Organization

This dissertation work is divided in five chapters. The summary of these chapters is given below:

• Chapter-1

This section provides an overview of the general background, problem statement, objectives of research and study's scope.

• Chapter-2

This chapter explained detailed literature review of materials and process of stabilization and previous studies conducted by researchers.

• Chapter-3

This chapter explains the research methodology to characterize the untreated soil, stabilization of soil with WSA and stabilization of soil composite binder CCR:WSA.

• Chapter-4

This chapter presents the results obtained by conducting tests mentioned in methodology and discussions on the results.

• Chapter-5

This section provides the conclusions drawn from the research findings and also presents the recommendations derived from this research.

### **CHAPTER 2: LITERATURE REVIEW**

### 2.1 General

Expansive soil is kind of clay soil which is rich in hydrophilic minerals like montmorillonite and illite. It undergoes expansion when water is added and shrink when water is removed. It is very hard in dry state as compared to wet state. Expansive soil is characterized by plasticity, compressibility, swelling, cracking and low bearing capacity. These properties exert pressure on the structure built on expansive soil [3].

Volumetric alterations due to swelling and shrinkage in soils are very problematic for structures constructed on these soils. These characteristics are specially damaging to lightly loaded structures like underground pipes, buildings, residential homes, sewerage lines, subsurface water tanks & reservoirs, roads, railway lines, irrigation channels etc. Expansive soils volumetric expansion and contraction is influenced by many factors but most important of them are clay content, water availability, mineralogical composition, soil fabric [16]. To alleviate the problems related to expansive soils, it essential to understand the characteristics of clayey soils.

## 2.2 Clayey Soil

#### 2.2.1 Clay Minerals

Chemical weathering of rock results in the permanent change in its mineral structure. These changes occur by action of water,  $CO_2$  and  $O_2$ . This process leads to formation of new minerals of different properties from parent rock. Chemical weathering results in production of crystalline particles of size less than 2 microns. These are termed as clay minerals [17].

There are two basic units in clay composition.

- 1. Silica Sheet
- 2. Alumina Sheet

Silica sheet contains silica-oxygen tetrahedron. Alumina sheet consists of aluminum-hydroxyl octahedron. These units exists in sheet structure. The charges are not balanced in these units that creates imbalances in charges. The charges on these units are negative in most of cases. So these units combine to form to stabilize these charges to attain a stable configuration and form sheet structures. These units do not exist as single units. The silica sheet is formed by mutual sharing oxygen present in silica-oxygen tetrahedron units. While in the case of alumina sheet, it is hydroxyl in between the two units. In this way, units combine and form a long sheet.



Figure 3: (a) Unit Structures; (b) Type of sheets in Clay [17]

The silica sheet is not stable and contains charges. These charges are negative in nature. While alumina sheet is stable in terms of charge and is neutral in that sense. Silicon and aluminum are in the center of their units. These are sometimes replaced by the some other cations. These replacing cations are of almost similar in size to ones they are replacing. These don't change the crystal structure. This phenomenon of cation replacement is called isomorphous substitution. These cations are mostly have valency smaller than the cations being replaced. In this way, more negative charge is present in the product [18].

The sheets of silica and alumina with one another in different combinations. This creates a layer structure. These layers then combine to form minerals, from which clays are formed. Different clay mineral are formed by different combinations of these layers and with different types of bonds in between them.



Figure 4: Clay Mineral Formation Flow Chart [19]

### 2.2.2 Kaolinite

Kaolinite is formed by stacking of layers. Each layer of Kaolinite is made of 1 silica and 1 alumina sheet. These basic sheets are bonded to each other by hydrogen bonds. As kaolinite contains 1 silica sheet and 1 alumina sheet so it is also termed as 1:1 clay mineral [17].

Hydrogen bonds are very stable type of bonds. These make the kaolinite crystal remain stable during hydration environment. Which makes kaolinite to exist in stability and stack in layers in much ease. Kaolinite minerals have very large number layers in their structures. These minerals contains layer in between 70 & 100. Kaolinite formation is dependent upon the presence of its constituents. Usually, it is present in areas when precipitation is high. The metals like, iron and cations are drained from the soil. Aluminum rich areas favors the formation of Kaolinite. This is because Kaolinite is 1:1 minerals. Isomorphous substitution in Kaolinite is very low. It is estimated that it happens in silica sheet with silicon being substituted by aluminum and reducing the positive charge. The chance of this happening is 1 in 400.

- Kaolinite particles are characterized by their huge structure of repeated layers. These are the general characteristics:
  - Thick
  - Large
  - Stiff
- > Hydraulic conductivity of kaolinite is more than montmorillonite and illite.
- Compressibility of kaolinite is lesser than montmorillonite and illite.
- Swelling and shrinkage of kaolinite is less than illite and montmorillonite. It depends upon the plasticity of soil. The soil having more plastic nature, will ultimately be with more swelling. That soil will be having more shrinkage.

#### 2.2.3 Illite

Illite is made up of 1 alumina sheet in between 2 silica sheet. It is 2:1 mineral. The bonds in between the kalinite layers is that of potassium bond. Isomorphous substitution occurs in silica sheet of illite with silicon being replaced with aluminum with lower valence. Illite is clay mineral whose presence is affected by the its constituents. Thus its cation exchange capacity is more than kaolinite [20].

- Illite structure consists of plate like films. Illite are shaped like kaolinite in some aspects like layering and also like montmorillonite like thin [19]. The particles are characterized by:
  - Terraced/ Layered
  - Thin
  - Flaky

> Hydraulic conductivity of illite is more than montmorillonite and less than kaolinite.

- Compressibility of is illite lesser than montmorillonite and more than kaolinite.
- Swelling and shrinkage of illite is more than kaolinite and less than montmorillonite. It depends upon the plasticity of soil. The soil having more plastic nature, will ultimately be with more swelling. That soil will be having more shrinkage.

### 2.2.4 Montmorillonite

Montmorillonite is the similar in structure to illite as its also contains two silica sheets with alumina sheet in between them. It is 2:1 mineral. Isomorphous substitution occurs in both sheets with silica sheets silicon being replaced with aluminum. In alumina sheet, aluminum is replaced by Magnesium and iron. So cation exchange capacity of montmorillonite is quite high. The layer of montmorillonite are attached by water and other cations. Thus bond is not strong and is susceptible to water and other exchangeable cations.

These minerals are formed in environments with more silicon than aluminum. Drainage of area should be poor. Its precipitation level should be low. Mostly found in arid area of world with poor drainage at site. The areas with more magnesium and iron are more in favor of smectite minerals. It is not very common to find clay soil with single type of mineral. These minerals occur in different combinations. Most of time, smectite minerals occur and with illite in mix with them [19].

- Montmorillonite is composed of particles which are flaky type. They are characterized by:
  - Thin
  - Platy/Filmy
  - Small particles
- Hydraulic conductivity of montmorillonite is less than both kaolinite and illite.
- Compressibility of montmorillonite is more than both kaolinite and illite.
- Swelling and shrinkage of montmorillonite is more than illite and kaolinite. It depends upon the plasticity of soil. The soil having more plastic nature, will ultimately be with more swelling. That soil will be having more shrinkage.

Due to isomorphous substitution, there is always net negative charges on the clay particles. Which causes the cations present in pore spaces to be attracted to clay's negatively charged space to enter and get attached to it. So there are water molecules and other cations like Calcium, magnesium, Sodium ions etc. These cations are not held strongly but are loosely held. These cations because their similar charge repel each other while being attracted to clays negatively charged surface. This layer of negatively charged particles with cations held close to it in dispersed condition is called Double

layer. It has been found that with increasing distance from the particle surface, the attraction forces are reducing and repulsive forces are increasing [18].



Figure 5: Diffuse Double Layer at Particle Surface [18]

### 2.2.5 Clay Fabric

There are repulsive and attravtive forces acting between the clay particles. The net effect of these forces will decide the structure of clay particles. Attractive forces are due to inter particle forces like Van Der Waal forces which act between two particles. Which are not charged and essentially neutral. Also, when cations of more valency are attracted to clay particles, attraction forces are increased. It also happens in the condition of more cations present in the space between double thicknesses. If repulsive forces dominate the soil particles, the structure of particles will be like dispersed. But if the attraction forces dominate and orientations of particles will be termed as flocculated structure. Soils get deposited from the water that is acting as agent to carry minerals from one place to another. These minerals get deposited from the water and when many minerals get deposited, they form suspensions. Clay suspensions is more

important to understand rather than that of sand and silt ones. Following are the associations developed between particles in clay suspensions [19].

## Dispersed

There is no connection between particles whatsoever.



**Figure 6:** Particle Alignment Modes in Clay (a) Dispersed & Deflocculated b) Aggregate and Deflocculated c)EF Flocculated and Dispersed d) EE flocculated and dispersed e) EF flocculated and Aggregated f) EE flocculated and Aggregated g) EF & EE flocculated and Aggregated [19]

#### Aggregated

Particles in clay suspensions are connected face to face.

Flocculated

Aggregates are held in edge to edge alignment. It can also be edge to face one.

Deflocculated

There is no connection between the aggregates.

The dispersed structure are often thick in nature. The flocculated clay particles make card house type structures that are very big in volume. But they are compressed to a more compact structure.

## 2.3 Soil Stabilization

Soil stabilization is technique which deals with improvement of engineering characteristics of problematic soil. This process is useful for soils which are problematic and pose hazard with working and constructing structures on them. The primary focus of this is to make problematic soil (like expansive soils, collapsible soils etc), more stable, more serviceable and stronger. There are many ways in which this can be achieved. There are two major types of soil stabilization:

- 1. Chemical Stabilization
- 2. Mechanical Soil Stabilization

### 2.3.1 Mechanical Stabilization

Mechanical stabilization is technique which deals with improvement of engineering characteristics of problematic soil by applying mechanical energy for changing its gradation [21]. There are many methods for mechanically stabilizing the soil, which are following:

• Compaction

- Pre-Wetting
- Stone Columns
- Densification
- Deep Soil Mixing
- Preloading
- Soil Replacement

## 2.3.1.1 Advantages of Mechanical Stabilization

- This technique is not complex and can applied with ease [22].
- This technique can be performed by unskilled labor.
- Less problematic soil can be stabilized with ease.
- This technique is environment friendly in the sense that it don't emit hazardous substance.
- Can be simply authenticated.

## 2.3.1.2 Disadvantages of Mechanical Stabilization

- More fuel is required to run this process
- Quality assurance is required more often
- Expansive cannot stabilized easily
- Works best with chemical stabilization

## 2.3.2 Chemical Stabilization

Chemical stabilization is technique which deals with improvement of engineering characteristics of problematic soil by adding in it the additives or stabilizers. Chemical stabilization is most favored by engineers which are working on site, due to reasons that it is very cost effective and performance that more reliable [23]. There are many stabilizers which are used for chemical soil stabilization. The soil stabilizers are shown in Figure 7.



Figure 7: Soil Stabilizers Categorized [22]

## 2.3.2.1 Advantages of Chemical Stabilization

- It can be validated with simple tests
- Wastes from different sectors can be used for chemical stabilization
- It can be applied to any soil
- It is very economical
- Factors affecting performance is reaction after mixing.

## 2.3.2.2 Disadvantages of Chemical Stabilization

- This technique can be hazard for environment
- It can be dangerous for labor at work
- Its working depend on many environmental factors
- Different materials in soil can destroy its performance
- This technique can be costly due to site conditions

### 2.3.3 Stabilization with Lime

Lime is most favored stabilizer for expansive soil stabilization. In this era, where highways and other modes of transportation ware being built extensively to cater the needs of population and economy. This stabilizer has proved its effectiveness by reducing cost for stabilization and improved engineering characteristics [24]. The major constituents of expansive soil are aluminate and silicate. The mechanism by which lime reacts with these constituents of expansive soil can be explained by these processes:

# 2.3.3.1 Hydration

When water is added to lime, it changes the lime to hydraulic one.

 $CaO(Lime) + H_2O(Water) \rightarrow Ca(OH)_2$  (Hydraulic Lime)+ Heat

Calcium hydroxide in the presence of water does not remain stable and breaks down to Calcium ion and hydroxyl ion. These ions will react with soil elements and modification process will start.

#### 2.3.3.2 Cation exchange

Calcium ions has positive charge on it. While clay particle have negative charges due to isomorphous substitution, which will cause the  $Ca^{+2}$  to be attracted to particle surface. This will cause replacement of already present monovalent cation to be replaced. In this way, divalent cations will increase in the pore solution. This will cause the diffuse double layer thickness to be reduced [25].

### 2.3.3.3 Flocculation and Agglomeration

After cation exchange, there will be reduction in repulsion between clay particles, which will reduce the distance between the clay surfaces. This will create a feasible environment for clay particles to make aggregations with each other. Flocculation will happen inside the soil structure. This will modify the soil structure, make it more serviceable.

### 2.3.3.4 Pozzolanic Reaction

The OH<sup>-</sup> which was released due to dissolution of Calcium hydroxide, will take part in soil chemistry. The alkalinity of soil will be increased. These processes will make the soil to leach alumina and silica from soil. These alumina and silica will react with Calcium present in the soil and make pozzolanic products. Calcium silicate hydrate will be formed along with calcium aluminate hydrate [26]. These reactions will provide soil with strength and load bearing against external agents.

#### 2.3.3.5 <u>Carbonation</u>

Calcium present in soil matrix can react with CO<sub>2</sub> present in atmosphere. This will form the Calcite products which secondary products from the calcium present in the system [27]. As this will consume the calcium present in the system and will stop the pozzolanic reaction with silicate and aluminate. Calcite will increase the strength but this is very much low as compare to pozzolanic products.

The first two phases are included in modification phase [16]. The pozzolanic reaction happens in Solidification. These phases change the soil properties altogether and enhance its engineering character.

> Lime reacts more proactively with smectite minerals

Soil has liking for CaO, and it will absorb lime and cation exchange process will start. After that process it can react with soil constituents and form pozzolanic products.



Figure 8: Mechanism of Lime Stabilization [16]

- During curing period, the pozzolanic reaction keep on taking place and forms products like Gismondine and tobermorite etc. These products will bind the soil particles and strengthen its structure [28].
- 2.3.4 Stabilization with Cement

The most common stabilizer for problematic soil stabilization in whole world is ordinary Portland cement (OPC). It is very useful for soil stabilization because of following reasons:

- ➢ Easily available
- ➢ UCS is higher

Quick improvement

It has four main constituents namely,

- $1. \ C_2S$
- 2. C<sub>3</sub>S
- 3. C<sub>3</sub>A
- 4. C<sub>4</sub>AF

Where,

C stands for CaO; S stands for SiO<sub>2</sub>; A stands for  $Al_2O_3$  and F stands for  $F_2O_3$ .

The mechanism of cement stabilization of soil is very similar to CaO stabilization.



Figure 9: Mechanism of Cement Stabilization [16]

# 2.3.4.1 Hydration

When water is added to Cement, it changes the dicalcium silicate and tricalcium silicate to release calcium ions in clay structure. These calcium ions react with soil constituents and form products of hydration.

 $2C_3S + 6H_2O \rightarrow Ca(OH)_2$  (Hydraulic Lime) + CSH (Calcium silicate hydrate)  $2C_2S + 4H_2O \rightarrow Ca(OH)_2$ (Hydraulic Lime) + CSH(Calcium silicate hydrate)

Calcium hydroxide formed in above reactions, in the presence of water does not remain stable and breaks down to Calcium ion and hydroxyl ion. These ions will react with soil elements and modification process will start [16].

#### 2.3.4.2 Cation exchange

Calcium ions has positive charge on it. While clay particle have negative charges due to isomorphous substitution, which will cause the  $Ca^{+2}$  to be attracted to particle surface. This will cause replacement of already present monovalent cation to be replaced. In this way, divalent cations will increase in the pore solution. This will cause the diffuse double layer thickness to be reduced [25].

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After cation exchange, there will be reduction in repulsion between clay particles, which will reduce the distance between the clay surfaces. This will create a feasible environment for clay particles to make aggregations with each other. Flocculation will happen inside the soil structure. This will modify the soil structure, make it more serviceable [16].

# 2.3.4.4 Pozzolanic Reaction

The OH<sup>-</sup> which was released due to dissolution of Calcium hydroxide, will take part in soil chemistry. This will boost the alkalinity in the soil. These processes will make the soil to leach alumina and silica from soil. These alumina and silica will react with Calcium present in the soil and make pozzolanic products. Calcium silicate hydrate will be formed along with calcium aluminate hydrate [26]. These reactions will provide soil with strength and load bearing against external agents.

Cement stabilization works best with following types of soils:

- Sand with fine
- Clays of low and medium plasticity

### 2.4 **Previous Literature**

It was reported in the past that Calcium carbide residue and Fly ash (FA) was used in improvement of properties of high plastic soil. CCR contains approximately 70.78% CaO. It is found that Calcium carbide residue has modified the engineering characteristics of clay soil which contains large amounts of natural pozzolanic material. FA increased the densification and pozzolanic reaction capacity [29]. It is possessing traits like commercial lime and is also non-hazardous. It has been reported that CCR exhibited outstanding mineralogical and physiochemical properties. Which makes it a cost effective and environmentally friendly alternative for improvement of soil [16].

Calcium carbide residue was used in improvement of properties of White Kaolinite and bentonite (Green). CCR contains 90.1% CaO. For this purpose, UCS, consolidation, FESEM, XRF, PSA and N<sub>2</sub>-BET were performed on soils treated with CCR. CCR was added in amount of (0%, 3%, 9%, 12%, 15%) of dry mass of soil. It was found that optimal strength of bentonite soil occurred at 9% CCR and optimal for Kaolinite occurred at 12% CCR content. There was increase of 6.8 times in strength of bentonite soil. While in kaolinite soil it was 5.8 times that of original soil, after 90 days of curing [30]. Denser soil fabric was obtained as result of stabilization. This concluded that CCR is a cost effective solution and environmentally viable stabilizer for clay type soils.

Rammed earth was stabilized by using CCR and FA. Binders were added in different ratios (CCR:FA = 40:60 & CCR:FA = 60:40). And binder was added in amounts of (3%, 6%, 9%, 12%, 15%). UCS values of (CCR:FA = 40:60) binder were maximum at 12% and that for (CCR:FA = 60:40) binders are maximum at 15% at all curing conditions (Siddiqua and Barreto 2018). While SEM images showed reduced void spaces in stabilized soil matrix, resulting in increased strength.

Rice husk ash (RHA) and CCR was used to stabilize the high plastic fine grained soil. A fixed amount of additive at 10% was used during the testing of Atterberg limits, UCS and SEM. It was found that plasticity limits improved greatly with use composite binder as compared to untreated soil and CCR treated soil. UCS values increased to 18 and 1.5 times for untreated soil and CCR treated soil respectively for binder (CCR:RHA= 60:40) [31].

It was also reported that the impact of wheat husk ash on the engineering properties of fine sand. WHA is added in amounts of (0, 3, 5, 7, 9)% of dry soil. Major tests performed were Compaction, UCS, CBR. It was concluded that OMC is maximum at 7% WHA content. UCS and CBR are giving maximum values at 7% of WHA [32].

WSA is normally used for cattle feed, bedding, pulp & paper, nanomaterials, fertilizers and mud house construction. In developing countries, left over wheat straw is burnt causing environmental and health problems. It is also a problem to tackle the ash left after burning [33]. It was found that investigation of WSA and bentonite on self-compacting system. WSA formulation is found to be more effective in making product with free lime and reducing the void spaces in soil matrix. Which makes treated soil more resilient to water absorption and acid attacks.

It was reported that global production of wheat is above 770 million tons in 2020. And that 1 ton of wheat gives 1300-1400 kg of wheat straw. Thus, crop residue when burnt in biomass plants and mills gives enormous amount of agricultural waste ashes. Open dumping these ashes creates environmental and health related issues. It causes groundwater contamination and reduced land available for further dumping [33].

Expansive soil was stabilized by using binder of wheat husk ash and sugar cane straw ash. The treated soil was tested for Atterberg limits, compaction, UCS and CBR. It was found that Atterberg limits improved as binder content increases up to 9%[34]. OMC, UCS & CBR values are maximum at 7% binder amount. It was concluded that this binder can be used to treat expansive soil.

### 2.5 Summary

All of this literature conclude that CCR has been used with other agricultural waste ashes (rice husk ash, bagasse ash and biomass ash) for stabilization of problematic soils. Due to significant amount of amorphous silica and pozzolanic properties, WSA has been used soil stabilization and as replacement of cement and sand in concrete. Currently, research regarding application of CCR and WSA as composite binder, in soil stabilization, is insufficient or nonexistent. This study aims to assess the viability of using CCR and WSA binder for soil stabilization.

# **CHAPTER 3: METHODOLOGY**

#### 3.1 General

This study is conducted to improve the properties of the expansive soil using wheat straw ash (WSA) and calcium carbide residue (CCR) composite. This chapter discusses the materials and methodology implemented to accomplish primary aim of this study. At first, material collection is discussed. Secondly, experimental scheme is explained, which consists of three phases. In first phase, untreated soil is characterized. In second phase, untreated soil is stabilized with WSA to find the optimum content. In third phase, soil is stabilized using WSA and CCR composite binder.

All the experiments performed were according to respective ASTM standards. All the geotechnical testing were performed in Geotechnical laboratory of NICE, NUST. Microstructural characterization by XRF, XRD, SEM and EDX were also performed to understand the morphological and mineralogical changes occurring during the stabilization process. The detail methodology is explained in the following sections.

### 3.2 Methodology

Methodology of this study comprises of four phases. The first phase is related with materials collection stand remaining three phases are related with experimentation. Following are the four phases:

- 1. Materials Collection
- 2. Characterization of Untreated Soil
- 3. Soil stabilization with WSA
- 4. Soil Stabilization with (CCR:WSA) composite binder



Figure 10: Research Methodology Adopted

### 3.3 Phase-1: Material Collection

This phase is related with collection of materials used in this study, which are untreated soil (Nandipur), CCR and WSA. All the materials were collected in bulk.

# 3.3.1 Soil

The untreated soil used in this research was obtained from Nandipur, Gujranwala. It was taken from a depth of 3ft from NSL. The soil was put in the polyethylene bags. It was transferred to laboratory. Initially, the soil was left in open for air drying. This was done for 3 days. Then to remove the remaining moisture it was dried in an electric oven at  $100 \pm 5^{\circ}$ C in an electric oven for 1 day. After that, it was taken out from oven and pulverized so that all of soil passes through No. 16 (1.18mm).

### 3.3.2 Calcium Carbide Residue (CCR)

CCR was taken from acetylene production facility in Wah Cantt. It was disposed of as byproduct/waste. It was then dumped in open disposal site at back of production facility. It was collected in polyethylene bags and transported to Geotechnical Lab at NICE, NUST. Initially, the CCR was left in open for air drying. This was done for 3 days. Then to remove the remaining moisture it was dried in an electric oven at  $100 \pm 5^{\circ}$ C in an electric oven for 1 day. It was ground to pass Sieve No. 40 (425µm). The color exhibited by CCR was white and it had specific gravity of 2.32 g/cm<sup>3</sup>. CCR waste in backyard of Acetylene Gas production facility is shown in Figure 13.



Figure 11: Untreated Soil Site Location

### 3.3.3 Wheat Straw Ash (WSA)

WSA was collected from Punjab Oil Mills Islamabad. Wheat straw was used as heating fuel in the facility. After burning the straw, ash was disposed of as waste. This ash was collected in polyethylene bags and transported to Geotechnical Lab at NICE, NUST. Initially, the WSA was left in open for air drying. This was done for 3 days. Then to remove the remaining moisture it was dried in an electric oven at  $100 \pm 5^{\circ}$ C in an electric oven for 1 day. It was ground to pass Sieve No. 40 (425µm).



Figure 12: Pulverized Untreated Soil from Nandipur



Figure 13: CCR waste piled up at Acetylene Plant in WAH Cantt



Figure 14: Wheat Straw Bales Stacked in Oil Mill

## 3.4 Phase-2: Untreated soil Characterization

Second phase is related with determination of properties of untreated soil. Engineering and geotechnical properties were determined for untreated soil, collected from Nandipur. Major testing performed were grain size analysis, plasticity, compaction, strength & deformation analysis and microstructural analysis.

- 1. Sieve Analysis
- 2. Hydrometer Analysis
- 3. Atterberg Limits
- 4. Specific Gravity
- 5. Compaction
- 6. Swell Percent
- 7. Swell Pressure
- 8. UCS
- 9. DST
- 10. XRF
- 11. XRD

## 3.4.1 Sieve Analysis

Sieve analysis test was performed for grain size analysis of untreated soil. The standard followed was ASTM D422, during the execution of test. Initially, a 300g soil sample was collected and subsequently pulverized. The soil sample was first dried in electric oven before the conducting test on it. The pulverized soil was then subjected to a washing process using a sieve labeled as #200. This was done to determine the weights of the soil passing through sieve #200 and the soil retained on sieve #200.

## 3.4.2 Hydrometer Analysis

Hydrometer analysis was performed to classify soil passing through sieve#200. The test was conducted following the guidelines mentioned in ASTM D422.



Figure 15: Hydrometer Test in the Laboratory

# 3.4.3 Atterberg Limits

Liquid limit, plastic limit and plasticity index test was conducted on the soil by using Casagrande apparatus. These parameters were determined to understand the plasticity behavior of untreated soil. The test was conducted following the guidelines mentioned in ASTM D4318. The soil sample was first dried in electric oven before the testing procedure.



Figure 16: Liquid Limit Test by Casagrande Apparatus

# 3.4.4 Specific Gravity

This test was performed according to ASTM D854. Soil used in this test was passing through sieve#4. This test was done to determine the specific gravity values for untreated soil collected from Nandipur. The following images show the proceeding of specific gravity test.



Figure 17: Specific Gravity Test performed in Laboratory

## 3.4.5 Compaction

Standard compaction test was conducted on soil which was dried in electric oven beforehand. This is to explore moisture density relationship for soil used in this research.  $\omega_{opt}$  and  $\gamma_d$  of soil were determined. The diameter of standard compaction mold was 4 in and height was 4.584 in. Oven dried soil was used for this test. The soil used was passing sieve#16. At first soil was mixed with water for 10 minutes. It was then left for 2 h to completely homogenize the sample. For each test, at least five compaction points were taken to get reproducible results.

### 3.4.6 Swell Percent

Swell percent test was conducted to understand the swelling behavior exhibited by untreated soil. The test was conducted following the guidelines mentioned in ASTM 4546D. Soil used was passing sieve#16. Soil sample for this test was placed in a ring of dimensions ( $60 \times 20$ ) mm. Ring assembly was then put in oedometer cell. A vertical dial gauge was then set on oedometer apparatus to note the vertical movements. Water was then poured in oedometer cell until it is completely filled with water.



Figure 18: Swell Percent Test performed in the Laboratory

Dial gauge readings were continuously recorded over a span of few days until a point was reached where no significant changes in the readings were observed.



Figure 19: Swelling Pressure Test carried out in Laboratory

#### 3.4.7 Swelling Pressure

Swell pressure test was performed by oedometer apparatus. The test was conducted following the guidelines mentioned in ASTM 4546D. Soil used was passing sieve#16. Soil sample for this test was placed in a ring of dimensions (60×20) mm. After swell percent test, sample was subjected to incremental loads until the sample got to its original height.

#### 3.4.8 Unconfined Compression Test

To evaluate the undrained strength of soil, UCS test was conducted. The test was conducted following the guidelines mentioned in ASTM D2166. UCS test samples were in mold of diameter 70 mm and of height 140mm. Oven dried soil was used for this test. Soil used was passing of sieve#16. Soil was mixed with water for more than ten minutes. Then it was put in the mold and compacted according to OMC and MDD values of soil as obtained in standard compaction test. Sample were tested in automatic loading machine. Its loading rate is set at 1.25 mm/min. Three UCS samples were made for each test. This was done to get consistent results.

#### 3.4.9 Direct Shear Test

To evaluate the shear strength parameters of soil, DST test was conducted. The test was conducted following the guidelines mentioned in ASTM D3080. Oven dried soil was used for this test. Soil used was passing of sieve#16. Soil sample was compacted to OMC and MDD. The normal stresses experienced by specimen were 50 kPa & 100 kPa and then increased to 200 kPa.



Figure 20: Unconfined Compression Test Performed in the Laboratory



Figure 21: Direct Shear Test Performed in Laboratory

3.4.10 X-ray Fluorescence (XRF)

To evaluate and assess elemental composition of untreated soil, CCR & WSA, the test conducted was XRF. This test was performed at Bestway Cement Farooqia. XRF test is based on mechanism that when material is exposed to high energy X-rays, it will produce fluorescent X-rays, which are distinctive of its elemental composition.

### 3.4.11 X-ray Diffraction (XRD)

To evaluate and assess mineralogical composition and crystalline structure of untreated soil, CCR & WSA, the test conducted was X-ray diffraction. This test was performed to identify clay minerals found in the untreated soil. This helped in understanding the engineering characteristics of materials that were strength and reaction to moisture. This test helped in better understanding of materials behavior.

#### 3.5 Phase-3: Soil Stabilization with WSA

Third phase is related with determination of properties of untreated soil mixed with WSA. This phase is related with optimization of untreated soil with WSA. WSA was blended with untreated soil in varying dosages (5%, 7.5%, 10%, 12.5% and 15%). This was done to find the optimum WSA content for untreated soil. Optimum WSA content was decided based on UCS results. Major testing performed were compaction and strength analysis.

- 1. Compaction (ASTM D698)
- 2. UCS (ASTM D2166)

### 3.5.1 Compaction

The test was conducted following the guidelines mentioned in ASTM D698. Standard compaction test was conducted on soil which was dried in electric oven beforehand. This is to explore moisture density relationship for soil used in this research.  $\omega_{opt}$  and  $V_d$  of soil mixed with varying fractions of WSA (5%, 7.5%, 10%, 12.5% and 15%), were determined. The diameter of standard compaction mold was 4 in and height was 4.584 in. Oven dried soil was used for this test. The soil used was passing sieve#16. At first soil was mixed with water for 10 minutes. It was then left for 2 h to completely homogenize the sample. At each dosage of WSA for untreated soil, at least five compaction points were taken to get reproducible results. These results will be used in UCS tests.

## 3.5.2 Unconfined Compression Test

To evaluate the undrained strength of soil, UCS test was conducted. The test was conducted following the guidelines mentioned in ASTM D2166. UCS of soil mixed with varying fractions of WSA (5%, 7.5%, 10%, 12.5% and 15%), were determined. UCS test samples were in mold of diameter 70 mm and of height 140mm. Oven dried soil was used for this test. Soil used was passing of sieve#16. Soil was mixed with water for more than ten minutes.



Figure 22: UCS Test Performed on the Soil Mixed with WSA

Then it was put in the mold and compacted according to OMC and MDD values of soil blended with WSA, as obtained in standard compaction test. Sample were tested in automatic loading machine. Its loading rate is set at 1.25 mm/min. Three UCS samples were made for each test. This was done to get consistent results. Finally, the highest UCS value was chosen as optimum WSA content for untreated soil.

### 3.6 Phase-4: Soil Stabilization with (CCR:WSA) Binder

Fourth phase is related with determination of behavior of untreated soil mixed with composite binder (CCR:WSA). Optimum WSA content was fixed as dosage for composite binder ratios. WSA will be replaced with CCR in varying percentages (0%, 10%, 25%, 50% and 75%) in binder dosages. WSA was blended with untreated soil in varying dosages (5%, 7.5%, 10%, 12.5% and 15%). This was done to find the optimal ratio of CCR:WSA for untreated soil. Major testing performed were plasticity, compaction, swelling properties, strength & deformation analysis and microstructural analysis.

- 1. Atterberg Limits (ASTM D4318)
- 2. Compaction (ASTM D698)
- 3. Swell Percent (ASTM D4546)
- 4. Swell Pressure (ASTM D4546)
- 5. UCS (ASTM D2166)
- 6. DST (ASTM D3080)
- 7. Stress-Strain & E<sub>50</sub>
- 8. XRD
- 9. SEM
- 10. EDX

### 3.6.1 Atterberg Limits

Liquid limit, plastic limit and plasticity index test was conducted on the soil by using Casagrande apparatus. These parameters were determined to understand the plasticity behavior of untreated soil. The test was conducted following the guidelines mentioned in ASTM D4318. The soil sample was first dried in electric oven before the testing procedure. Soil used was passing of sieve#40. These limits of soil mixed with varying proportions of CCR:WSA (0:100, 10:90, 25:75, 50:50 and 75:25) binder, were determined.

### 3.6.2 Standard Proctor Test

Standard compaction test was conducted on soil which was dried in electric oven beforehand. This is to explore moisture density relationship for soil used in this research.  $\omega_{opt}$  and  $V_d$  of soil were determined. The diameter of standard compaction mold was 4 in and height was 4.584 in. Oven dried materials were used in this test. The soil used was passing sieve#16. At first soil was mixed with CCR and WSA for 10 minutes. Then soil binder mixture was blended with water for 10 minutes. It was then left for 2 h to completely homogenize the sample. For each of five ratios of binder, at least five compaction points were taken to get reproducible results.

### 3.6.3 Swell Percent

Swell percent test was conducted to understand the swelling behavior exhibited by untreated soil. The test was conducted following the guidelines mentioned in ASTM 4546D. Soil used was passing sieve#16. Soil sample for this test was placed in a ring of dimensions ( $60 \times 20$ ) mm. Ring assembly was then put in oedometer cell. A vertical dial gauge was then set on oedometer apparatus to note the vertical movements. Water was then poured in oedometer cell until it is completely filled with water.



Figure 23: Swell Percent Test Performed on the Soil Mixed with CCR:WSA Binder3.6.4 Swelling Pressure

Swell pressure test was performed by oedometer apparatus. The test was conducted following the guidelines mentioned in ASTM 4546D. Soil used was passing sieve#16. Soil sample for this test was placed in a ring of dimensions ( $60 \times 20$ ) mm. After swell percent test, sample was subjected to incremental loads until the sample got to its original height.

#### 3.6.5 Unconfined Compression Test

To evaluate the undrained strength of soil, UCS test was conducted. The test was conducted following the guidelines mentioned in ASTM D2166. UCS of soil mixed with varying ratios of CCR: WSA (5%, 7.5%, 10%, 12.5% and 15%), were determined. UCS test samples were in mold of diameter 70 mm and of height 140mm. Oven dried soil was used for this test. Soil used was passing of sieve#16. Soil was mixed with water for more than ten minutes. Then it was put in the mold and compacted according to OMC and MDD values of soil as obtained in standard compaction test. Sample were tested in automatic loading machine. Its loading rate is set at 1.25 mm/min. Three UCS samples were made for each test. This was done to get consistent results. UCS sample



Figure 24: UCS Test Carried out on the Soil Stabilized with CCR:WSA Binder



Figure 25: DST Test Carried out on the Soil Stabilized with CCR:WSA Binder

was covered in cling wrap and labelled. It was then it was put in sealed box for dry curing. The curing was done for 7, 14, 28 and 90 days. Thus was done to understand the long term behavior of materials.

### 3.6.6 Direct Shear Test

To evaluate the shear strength parameters of soil, DST test was conducted. The test was conducted following the guidelines mentioned in ASTM D3080. Oven dried soil was used for this test. Soil used was passing of sieve#16. Soil sample was compacted to OMC and MDD. The normal stresses experienced by specimen were 50 kPa & 100 kPa and then increased to 200 kPa.

#### 3.6.7 Stress-Strain & E<sub>50</sub>

Stress-strain response was determined for soil stabilized with CCR:WSA binder. This was obtained from UCS results. These results were plotted to understand the stabilized soil behavior against external forces. To evaluate and assess the stiffness behavior of soil stabilized with CCR:WSA binder, secant modulus of elasticity was measured. It is termed as  $E_{50}$ . This parameter is important for settlement calculations.

### 3.6.8 X-ray Diffraction (XRD)

To evaluate and assess mineralogical composition and crystalline structure of crystalline structure of soil stabilized with CCR:WSA binder, XRD test was carried out. To understand the formation of cementitious hydrates in the improved soil. XRD is very useful to identify the crystalline phases of materials. It also gives information about the amorphous nature of material, which gives us clue about the reactivity of material involved in the stabilization process. Materials having mineral nature possess crystalline structure. Which can be identified using available diffraction patterns in the JCPDS database, for most of minerals known to this date. The test was conducted on

Bruker D8 Advance Diffractometer. The parameters set in the diffractometer were following:

- $\succ$  2θ range → (10° to 80°)
- > Voltage  $\rightarrow 20kV$
- > Step Size  $\rightarrow 0.02^{\circ}$
- > Time/Step  $\rightarrow$  0.5s



Figure 26: Samples for XRD Testing



Figure 27: XRD Test performed on Bruker D8 Advance Diffractometer

Samples for XRD were taken from middle portion of UCS samples, which were already tested. Samples were dried for 24h in oven and then pulverized to pass sieve#200. Mineralogical composition of soil is very important to asses the behavior of soil and this is very helpful in selecting the suitable additive for soil stabilization. It is know from literature that certain additives are very effective for soils with specific mineral types. So by using JCPDS database and patterns obtained by XRD tests on materials in this study, it gave better insight about the reactivity of additives with soil and helped in selecting dosage of additive material for stabilization process. X'Pert HighScore software was used for interpreting the XRD data obtained from test results. This software helped in understanding the new compounds formed as result of stabilization process. This provided better understanding of stabilization process and this ultimately improved the engineering properties of soil.

# 3.6.9 Scanning Electron Microscopy (SEM)

To assess the morphological alterations occurred due to stabilization of soil with CCR:WSA binder, SEM test was carried out. It also helps in identify the different phases of stabilization process. Specimen was collected from the UCS samples already tested. It was obtained from the middle section of samples. Samples were oven dried before taken for testing. SEM samples were of size less than 10 mm. Samples were coated with Gold before putting it inside the SEM device chamber. The voltage was set at 20kV. The test was conducted by JEOL JSM-6490A, which also had EDS detector installed in it.



Figure 28: SEM & EDS Test performed on JEOL JSM-6490A

## 3.6.10 Energy-Dispersive X-ray Spectroscopy (EDS)

EDS test was conducted to determine the elemental composition of soil-binder mixture. This test is performed by JEOL JSM-6490A apparatus. This apparatus can conduct on spot chemical analysis, which helped in giving information about the new compounds formed. EDS detector is fitted inside the SEM machine. EDS helped in identifying the chemical reactions which occurred as result of additives addition to soil. This test enabled us to understand the composition of new materials formed as a result of stabilization of untreated soil with CCR:WSA composite binder.

## **CHAPTER 4: RESULTS AND DISCUSSIONS**

### 4.1 General

This study dealt with investigation of stabilization of high plastic soil with composite binder (CCR:WSA). A series of experiments were carried out for investigating the stabilization process in detail. Engineering properties of untreated soil were determined. Then, untreated soil was stabilized with WSA. Compaction and UCS were major tests performed in this phase. Optimum quantity of WSA was determined based on the results of UCS results. This optimum dosage was fixed as binder for stabilization of soil. After that, soil was stabilized with composite binder (CCR:WSA). The compaction, plasticity, swelling, strength & deformation and microstructural behavior of stabilized soil were observed.

This chapter deals with test findings and their related discussions to study stabilization of untreated soil with composite binder (CCR:WSA). It explains the effect of influential factors (i.e. additive type, additive dosage & curing period) on the stabilization of untreated high plastic soil.

## 4.2 Untreated soil Characterization

### 4.2.1 Sieve Analysis

Sieve analysis of untreated soil was performed to determine its grain size distribution. Figure 29 depicts the results obtained after sieve analysis of untreated soil. The test was performed according to ASTM D422. There was no gravel found in the soil. The sand content was 1.9 % and fine content was 98.1 %. So, there is high fine content in the soil.

### 4.2.2 Hydrometer Analysis

Hydrometer analysis was performed to classify the fine content found in the untreated soil. The test was performed according to ASTM D422. It was observed that clay content in the soil was 45.5 % and silt content found was 52.6 %. The results of Hydrometer analysis is shown in Figure 29.



Figure 29: Particle size distribution of Soil

### 4.2.3 Atterberg Limits

Liquid limit obtained for untreated soil was 52 %. Plastic limit came out as 22.5 % and Plasticity index (PI) was came out as 29.5. According to Unified Soil Classification System (USCS), soil falls in the category of High Plastic (CH) soil, as shown in Figure 30 illustrates the outcomes of the Atterberg limit test and classifies soil as High Plastic (CH) soil by following the guidelines of USCS . Clay soils which lie above A-line, with LL > 40 and containing (>5)% smectite content are termed as swelling soils [35].



Figure 30: Plasticity Chart for Soil Classification (ASTM D-2487)

# 4.2.4 Specific Gravity

Specific gravity obtained for untreated soil came out as 2.68, which lies in typical range of specific gravity values for clays and montmorillonite. The standard followed was ASTM D854 during the test.



Figure 31: Compaction Result Obtained for Untreated Soil

### 4.2.5 Compaction

The compaction test was performed on soil according to ASTM D698. Optimum moisture content (OMC) was found out as 21.3 % and maximum dry density (MDD) came out to be 1.69 g/cm<sup>3</sup>. Figure 31 exhibits the moisture-density relationship for untreated soil.

### 4.2.6 Swell Percent (ASTM D4546)

The test was conducted following the guidelines mentioned in ASTM 4546D. The value of swell percent for untreated soil came out as 5.7 %. It has high degree of expansion [35].

## 4.2.7 Swell Pressure (ASTM D4546)

The test was conducted following the guidelines mentioned in ASTM 4546D. The value of swelling pressure for untreated soil came out as 142 kPa.



Figure 32: UCS Test Result of Untreated Soil

#### 4.2.8 UCS (ASTM D2166)

To evaluate the undrained strength of soil, UCS test was conducted. The test was conducted following the guidelines mentioned in ASTM D2166. The UCS of untreated soil came out as 126.5 kPa. Figure 32 depicts the result obtained after test for untreated soil.

#### 4.2.9 Direct Shear Test (DST)

To evaluate the shear strength parameters of soil, DST test was conducted. The test was conducted following the guidelines mentioned in ASTM D3080. The friction angle ( $\phi$ ) found out to be 14.3 ° and cohesion (C) was found out as 36.1 kPa.

#### 4.2.10 X-ray Fluorescence (XRF)

To evaluate and assess mineralogical composition and crystalline structure of untreated soil. This helped in assessing the mineralogical composition of soil. The XRF results of CCR and WSA is also found out. The XRF results of materials used in this study are shown in Table 1 illustrates the results obtained after execution of XRF of untreated soil. It can be seen from the results that major constituent of soil is silica  $(SiO_2 \rightarrow 58.8\%)$  and alumina  $(Al_2O_3 \rightarrow 17.2\%)$ , which are common for clay soils. Major constituent of WSA was found to be silica  $(SiO_2 \rightarrow 66.8\%)$ . It is characteristic of agricultural ashes to contain silica in large amounts. It is stated that if  $SiO_2$ ,  $Al_2O$  and  $Fe_2O_3$ , was added together and their sum is greater than 70% then by following ASTM C618 guidelines, the material has pozzolanic properties. So WSA has a pozzolanic character. This will help in its reaction with soil and CCR. The major constituent of soil is Calcium Oxide (CaO  $\rightarrow$ 77.2%) and the next major one is silica (SiO<sub>2</sub> $\rightarrow$ 8.1%). Calcium based compound reacts effectively with silica based contents [36].

Chemical		Expansive	CCD	WCA
Composition	Soil		CCK	WSA
SiO <sub>2</sub>		58.8	8.18	66.8
Al <sub>2</sub> O <sub>3</sub>		17.27	1.31	4.21
Fe <sub>2</sub> O <sub>3</sub>		6.49	0.74	3.38
CaO		1.26	77.24	8.2
MgO		2.2	0.8	2.05
$SO_3$		0.01	0.18	1.05
Na <sub>2</sub> O		0.65	0.25	3.67
K <sub>2</sub> O		3.06	0.11	4.4
Cl <sub>2</sub> O		0.05	0.04	2.5
LOI		9.68	2.74	4.06

Table 1: Chemical composition obtained after XRF test of Soil, CCR and WSA

### 4.2.11 XRD

To evaluate and assess mineralogical composition and crystalline structure of untreated soil, the test conducted was X-ray diffraction. Figure 33 portrays the results obtained after XRD pattern of soil contains quartz (Q), montmorillonite (M) and illite (I) in major quantities. The presence of swelling mineral in soil is an indication of its swelling behavior. XRD test also validates the results of XRF test, both tests showed the major constituent as quartz (SiO<sub>2</sub>). Peak of quartz (Q) is highest in whole XRD pattern of soil. Swelling mineral is present in soil XRD pattern, which caused the swellshrink characteristics in untreated soil from Nandipur.

XRD pattern of CCR is shown in Figure 33. It indicates that the major components of CCR are Portlandite (Ca(OH)<sub>2</sub>) and Calcite (CaCO<sub>3</sub>). This result further
validates the result of XRF, which also showed that the major constituent is calcium oxide. Its composition is like lime. Large amount of portlandite will be helpful in reaction with silica-based soil and WSA. XRD pattern of WSA shows that main constituent of it is Quartz (SiO<sub>2</sub>) and minor ones are cristobalite and rankinite, as shown in Figure 33. Results of XRD are in confirmation with results of XRF.



Figure 33: XRD Patterns for Soil, WSA and CCR

Value	Standard
52 (%)	ASTM D-4318
22.5 (%)	ASTM D-4318
29.5 (%)	ASTM D-4318
1.69 (kN/m3)	ASTM D-698
21.3 (%)	ASTM D-698
2.68	ASTM D-854
15.5 (%)	ASTM D-2216
126.5 (kPa)	ASTM D-2166
36.10 (kPa)	ASTM D-3080
14.3	ASTM D-3080
СН	ASTM D-2487
5.7(%)	ASTM D-4546
142(kPa)	ASTM D-4546
	Value         52 (%)         22.5 (%)         29.5 (%)         1.69 (kN/m3)         21.3 (%)         2.68         15.5 (%)         126.5 (kPa)         36.10 (kPa)         14.3         CH         5.7(%)         142(kPa)

 Table 2: Engineering Properties of Untreated Soil

# 4.3 Soil Stabilization with WSA

Third section deals with determination of properties of untreated soil mixed with WSA. WSA was blended with untreated soil in varying dosages (5%, 7.5%, 10%, 12.5% and 15%). Optimum WSA content was decided based on UCS results. Major testing performed were compaction and strength analysis.

- 1. Compaction (ASTM D698)
- 2. UCS (ASTM D2166)

#### 4.3.1 Compaction

The test was conducted following the guidelines mentioned in ASTM D698. This is to explore moisture density relationship for soil used in this research. For each percentage of WSA added to soil, at least five points were taken on compaction curve to get reliable results. Figure 34 depicts the compaction curves obtained for soil modified by addition of WSA in soil. It concluded that when WSA is added to soil, MDD of soil get reduced. While on the same time, OMC of soil increases. This indicated by the compaction curves moving towards the down right side of graph.

Figure 34 also illustrates that with addition of WSA in soil, the specific gravity of soil get reduced. The effect of WSA content on the specific gravity of soil stabilized is shown in the graph. Specific gravity of soil decreases as percentage of WSA increases in the soil. This is because specific gravity of WSA is 2.21 g/cm<sup>3</sup>, which ultimately have decreasing effect on the specific gravity of soil.  $V_d$  of soil decreased with addition of WSA in soil. This because of lesser specific gravity of WSA content than soil.

It is reported that WSA particles are very fine in nature and have very large surface area. This increased the water holding capacity of WSA particles. Due to this water adsorption, packing effect in the soil increased. This makes soil-WSA mixture more closely packed. It reduced the air voids in the soil matrix, which will eventually make soil more compact and denser. The increase in the moisture holding capacity had caused increase in the OMC of soil. The studies conducted on soil stabilization with agricultural ashes have shown similar kind of results [32], [37], [38].



**Figure 34:** Compaction curves for soil stabilized with WSA content *4.3.2 UCS* 

UCS test was conducted on soil stabilized with WSA content to find the optimum WSA content for untreated soil. UCS test result of soil stabilized with different WSA content. Figure 35 illustrates that with the addition of WSA in the soil, UCS of soil is enhanced until it reaches its optimum. UCS of soil increased with WSA content until it reaches its optimum strength at WSA (12.5 %). Upon further addition of WSA, strength decreased.

UCS increased from 126.5 kPa (original soil) to 280 kPa with WSA content at 12.5 %. This was occurred after 7 days of curing. At 28 days of curing, the strength of soil is enhanced to 280 kPa with WSA content at optimum (12.5%). This is almost 2.71

times increase in strength from original soil strength. This increase in UCS is caused by decrease in the air voids. As particles of WSA are very fine in size, this will fill voids and increase the compactness of soil structure. Soils resistance to deformation caused by external forces will be enhanced.



Figure 35: UCS test on soil stabilized with varying WSA Content

The decrease in the soil strength at WSA (15%) is caused by porous nature and impurities present in the WSA particles. WSA was waste product of oil mill boiler. It contained impurities. These factors caused weak bonds with soil particles. These findings align with the results of studies already done on soil stabilization by agricultural ashes [32], [38].



Figure 36: Cohesion of WSA Stabilized Soil



Figure 37: Friction Angle of WSA Stabilized Soil

## 4.3.3 Direct Shear Test

To evaluate the shear strength parameters of soil, DST test was conducted. The test was conducted following the guidelines mentioned in ASTM D3080. The results of cohesion for WSA stabilized soil. Figure 36 depicts that cohesion of soil stabilized with WSA is enhanced with rise in the addition of WSA content. The cohesion of soil improved raised from 36.1 kPa to 42.5 kPa at WSA content of 12.5 %. This increase in cohesion is caused by porous morphology of WSA particles. These pore adsorbed water and decreased the available moisture from soil. This will eventually decrease the distance between particles of soil and attraction between particles will increase.

The variation of Friction angle for soil stabilized with WSA. Figure 37 depicts that  $\phi$  of soil stabilized with WSA is enhanced with rise in the addition of WSA content. It increased from 14.3 ° for untreated soil to 25.8 ° at WSA content of 12.5 %. WSA particles are very fine in nature, will fill voids in between soil particles. This will increase connection between soil particles and increase the friction angle of soil.

## 4.3.4 Swell Percent

Swell percent of stabilized with WSA was determined to understand its swelling behavior. The results of swell percent for WSA stabilized soil. Figure 38 illustrated that swell percent values decreases for soil stabilized with the rise in the addition of WSA in soil. It decreased from 5.7 % for untreated soil to 3 % at WSA content of 12.5 %. This decrease in the swell percent is caused by increased non plastic WSA particles. The soil particles which are high plastic in nature are replaced with non-plastic WSA particles.



Figure 38: Swell percent of WSA Stabilized Soil



Figure 39: Swell Pressure of WSA Stabilized Soil

## 4.3.5 Swelling Pressure

Swelling pressure of stabilized with WSA was determined to understand its swelling character. The results of swelling pressure for WSA stabilized. Figure 39 depicted that swelling pressure exhibited a declining trend with rise in the WSA proportion in the soil. It decreased from 142 kPa for untreated soil to 76 kPa at WSA content of 12.5 %. Untreated soil contains particles which are swelling in nature and due to addition of WSA particles in place of soils swelling particles will make the stabilized soil with less water affinity [39]. These findings align with the results in previous researches in literature [40], [41].



Figure 40: Atterberg Limits of WSA Stabilized Soil

<sup>4.3.6</sup> Atterberg Limits

To evaluate plasticity of soil, Atterberg limits was found out by Casagrande apparatus. Figure 40 illustrated that liquid limit exhibited a declining trend with rise in the WSA proportion in the soil. Plasticity index exhibited a declining trend with rise in the WSA proportion in the soil. Plastic limit exhibited inclining trend with rise in the WSA proportion in the soil. There was a reduction of liquid limit from 52 % to 45 %. Additionally there was a reduction of PI from 29.5 to 19. This decrease in LL and PI is caused by water adsorption due to pores present on WSA particles. This reduces the water affinity of soil. This will ultimately reduce the LL and PI of soil. Soil stabilization with WSA content has made soil more resistant to deformation by reducing plasticity and water affinity of soil.

#### 4.4 Soil Stabilization with (CCR:WSA) Binder

This section is related with results and discussions related with untreated soil mixed with composite binder (CCR:WSA). Optimum WSA content was fixed as dosage for composite binder ratios. WSA will be replaced with CCR in varying percentages (0%, 10%, 25%, 50% and 75%) in binder dosages. Major testing performed were plasticity, compaction, swelling properties, strength & deformation analysis and microstructural analysis.

- 1. Atterberg Limits
- 2. Compaction
- 3. Swell Percent
- 4. Swell Pressure
- 5. UCS
- 6. DST
- 7. Stress-Strain & E<sub>50</sub>
- 8. XRD

9. SEM

10. EDX

#### 4.4.1 Standard Compaction Test

The test was conducted following the guidelines mentioned in ASTM D698. This is to explore moisture density relationship for soil used in this research. The compaction curves obtained after test were plotted as shown in Figure 41, for CCR:WSA binder stabilized soil. OMC of soil indicated a declining trend with addition of CCR proportion in soil.



Figure 41: Compaction Curves of Composite Binder (CCR:WSA) Stabilized Soil

OMC of soil decreased from 24.1 % to 21.72 %. This decrease in OMC is caused by a decrease in WSA content in soil binder mixture. WSA particle has pores in its surface. These pores act as water reservoirs [42]. As WSA content decreases in soil

binder mixture, these porous particles will become lesser in number and will the water holding capacity of soil-binder mixture.

MDD of soil-binder mixture increases with increase in CCR content in soils stabilized with CCR:WSA binder. There is difference in specific gravities of CCR and WSA, with CCR specific gravity (2.32 g/cm<sup>3</sup>) being higher. This will increase the specific gravity of overall mixture. MDD of soil-binder composite increases from 1.57 g/cm<sup>3</sup> at 0:100 to 1.61 g/cm<sup>3</sup> at CCR:WSA = 75:25 ratio. This increase in MDD is good for soil structure as it will increase the resistance to deformation of soil skeleton. Specific surface area of soil binder composite decreased.

#### 4.4.2 Atterberg Limits

Casagrande apparatus was used for carrying out these tests for soil stabilized with CCR:WSA binder. The results of Atterberg limit obtained after test, were plotted as shown in Figure 42, for CCR:WSA binder stabilized soil. LL of soil indicated a declining trend with addition of CCR proportion in soil. Plasticity index exhibited a declining trend with rise in the CCR proportion in the soil. Plastic limit exhibited inclining trend with rise in the CCR proportion in the soil. There was a reduction of liquid limit from 52 % to 37 %. Additionally there was a reduction of PI from 29.5 to 7.

CCR has portlandite as its main constituent, which was determined through its XRD patterns. The main components of portlandite are calcium and hydroxyl ions. After mixing water with soil and composite binder mixture, hydration process happens. This will cause  $Ca(OH)^2$  to split up into  $Ca^{+2}$  and  $OH^-$ .  $Ca^{+2}$  will increase in the mixture. Calcium ions will react negative charged clay surface. These ions will take the place of Na<sup>+</sup> and other monovalent cations present in the water solution around clay platelets. This process will cause cation exchange in clay-binder composite blend. This will affect

the double layer around the clay surfaces. Replacement of monovalent cations by divalent cations will cause a reduction in the thickness of double layer. This will further affect the distance between the clay particles and will reduce it. A declining trend will be exhibited by repulsive forces. An increase in attraction between soil particles will occur.

Soil particles will make bonds and will start making aggregates. These aggregates will make flocs in soil structure. This will change the water demand in the soil stabilized with binder composite. LL and PI will be reduced. All of this will affect positively to the strength and deformation characteristics of soil stabilized improved by composite binder.



Figure 42: Atterberg Limits of Composite Binder (CCR:WSA) Stabilized Soil 4.4.3 Swell Percent

Swell percent of soil stabilized with CCR:WSA binder was determined to understand its swelling behavior. The results of swell percent for CCR:WSA binder stabilized soil. Figure 43 illustrated that swell percent indicated a declining trend with addition of CCR proportion in soil. There was a reduction of swell percent from 5.7 % for untreated soil to 3 % at binder ratio 0:100 (Pure WSA). This decrease in the swell percent is caused by increased non plastic WSA particles. The soil particles which are high plastic in nature are replaced with non-plastic WSA particles. It further decreased to 0.75 %, at binder ratio of 75:25.



Figure 43: Swell Percent Analysis of Binder (CCR:WSA) Stabilized Soil

Cation exchange process is caused by the presence of  $Ca^{+2}$  ions due to CCR content in the mixture. Which will formation of aggregates and flocs in the soil stabilized with composite binder. This will change the water demand in the soil stabilized with binder composite. PI will be reduced. This will have great impact on the water affinity of soil stabilized. It is reported that decrease in the PI have decreasing effect on swelling characteristics of soil.

## 4.4.4 Swell Pressure

Swelling pressure of soil stabilized with CCR:WSA binder was found out by oedometer apparatus. This test was done to understand swelling behavior of soil stabilized with CCR:WSA binder. The results of swelling pressure for CCR:WSA binder stabilized soil. Figure 43 illustrated that swell pressure values indicated a declining trend with addition of CCR proportion in soil. There was a reduction of swell pressure from 142 kPa for untreated soil 76 kPa at binder ratio 0:100 (Pure WSA). This decrease in the swelling pressure is caused by increased non plastic WSA particles. The soil particles which are high plastic in nature are replaced with non-plastic WSA particles.



Figure 44: Swelling Pressure Analysis of Composite Binder (CCR:WSA) Stabilized Soil

It further decreased to 19 kPa at binder ratio of 75:25. Cation exchange process is caused by the presence of  $Ca^{+2}$  ions due to CCR content in the mixture. Which will formation of aggregates and flocs in the soil stabilized with composite binder. This will change the water demand in the soil stabilized with binder composite. PI will be reduced. This will have great impact on the water affinity of soil stabilized. It is reported that decrease in the PI have decreasing effect on swelling characteristics of soil.

## 4.4.5 Unconfined Compression Strength (UCS)

To evaluate the undrained strength of soil, UCS test was conducted. The test was conducted following the guidelines mentioned in ASTM D2166. UCS test was conducted for soil improved with addition of varying CCR:WSA binder ratios. Figure 45 depicted that UCS indicated a rising trend with addition of CCR proportion in soil. UCS test was conducted at different curing periods to understand the long term behavior of CCR:WSA binder on strength of soil stabilized. The curing for samples was done for 7, 14, 28 and 90 days. These were kept at ambient temperature in closed box in laboratory. UCS increases from 126 kPa to 4647 kPa at CCR:WSA ratio of 75:25. This was achieved for samples which were cured for 3 months. So the composite binder has enhanced UCS of soil stabilized by more than 35 times of untreated soil strength.

UCS of soil stabilized with CCR:WSA (0:100), improved by almost 3.5 times than untreated soil. This was achieved for samples which were cured for 28 days. This rising trend in UCS is caused by a decrease in the air voids. As particles of WSA are very fine in size, this will fill voids. Soil will be more resistant to deformation. WSA also contained calcium content in it as depicted in its XRF results. This calcium content will take part in cation exchange process. This will cause rise in the strength of soil.



Figure 45: Variation of UCS of Composite Binder (CCR:WSA) Stabilized Soil

Barman et al. mentioned that affinity for calcium content by clays [16]. It was also reported that there are two stages for lime stabilization, namely Modification and Solidification and lime content has an important role in both of processes. Modification by cation exchange and flocculation happens, if lime content is not enough. But when lime content is present in large quantity, modification occurs at first. Then pozzolanic reaction takes place and which leads to solidification phase of lime stabilization.

Gradient of lines for composite binder 0:100 gradually lowered over time as depicted in figure. This line then becomes almost horizontal, after 28 days of curing. This was because of very low calcium content in the soil. The gradient of line for binder 10:90 also show similar behavior as 0:100. The gradient of line for binder 25:75 exhibited a steepness, which is an indication of more strength gain over time. This was due to fact that it contains more CCR content in it as compared to the 10:90 and 0:100. As there was more calcium content so more reactivity and strength over time. But the line nearly becomes horizontal after 4 weeks, which was due to reason that calcium content in binder was used already and not enough is present for further reaction. Similar behavior is shown by 50:50 binder, as 25:75.

Optimum strength was found at binder ratio of 75:25. The line of which was very steep from start and remained steep till the end of curing period. This binder contained most CCR content of any other binder ratio. The calcium content remained in the soil-binder matrix even after modification process and took part in pozzolanic process. Which lead to solidification phase of stabilization.

WSA contains amorphous silica content, which is very effective for pozzolanic reaction with calcium content in CCR. WSA provided that amorphous silica content for reaction with excess calcium present in soil binder matrix. All the silica content form soil side was used at this stage. So calcium reacts with silica from WSA. That is reason for gradient steepness after 28 days of curing. It was similar in trends to findings reported in literature [36].

#### 4.4.6 Direct Shear Test

To evaluate the shear strength parameters of soil, DST test was conducted. The test was conducted following the guidelines mentioned in ASTM D3080. Figure 46 illustrated that cohesion of soil stabilized with composite binder is enhanced with rise in the addition of CCR proportion. The cohesion of soil improved raised from 36.1 kPa to 42.5 kPa at 0:100.



Figure 46: Cohesion of Composite Binder (CCR:WSA) Stabilized Soil



Figure 47: Friction Angle of Composite Binder (CCR:WSA) Stabilized Soil

This increase in cohesion is caused by porous morphology of WSA particles. These pores adsorbed water and decreased the available moisture from soil. This will eventually decrease the distance between particles of soil and the attraction between particles will increase. With other binder ratios, as CCR content is introduced in the mixture, bonding between soil particles will increase. This is caused by reduction in diffuse layer thickness. Cohesion increases to 46 kPa after increase in CCR content in mixture. This was found out at binder 75:25. The variation of friction angle for soil stabilized with composite binder is shown in Figure 37. Friction angle indicated a inclining trend for soil stabilized with the rise in the CCR proportion in soil. It increased from 14.3 ° for untreated soil to 25.8 ° at 0:100. WSA particles are very fine in nature, will fill voids in between soil particles. This will increase connection between soil particles and increase the friction angle of soil. Liu et al. studied the stabilization soil with composite binder and reported similar kind of behavior [23].

Friction angle increased to 34 °, after CCR was introduced in the mixture. This CCR will affect the structure of soil by making cation exchange and flocculation. This will reduce the PI of soil stabilized. Which will increase the friction angle.

#### 4.4.7 Stress-Strain & E<sub>50</sub>

To understand the deformation behavior of soils stabilized with composite binder, stress -strain behavior was determined. This was obtained from UCS results, which were done on automatic loading machine. These results were plotted to examine the stabilized soil resistance to deformation as shown in Figure 48. These results are samples which were kept in closed box for 28 days of curing. Results demonstrated that the gradient of line for untreated soil is very low and does not change abruptly over time. The strength doesn't increase quickly but in slow manner. This also happens when it fails, with slow and steady failure. This shows the untreated soil acted like ductile material. This often is caused by hardening under the action of strain in soil.

For soil stabilized with binder ratios, there was increase in peak strength values. Gradient of line was getting high with increase in the CCR content. Peak stress reaches 1974.3 kPa after CCR increases to 75% of whole binder content in soil. It was due pozzolanic reaction that occurred after the bonding between soil particles due cementitious products formation between them. This value was found at 28 days of curing. These stress-strain plots are very important for settlement calculations. Due to increase in the CCR content in mixture, curves moves to higher stiffness section of figure. This helped in the resistance against stain conditions for soil stabilized.



Figure 48: Stress- Strain Behavior of Composite Binder (CCR:WSA) Stabilized Soil at 28 Days of Curing

Stress-stain plot for 90 days was observed to understand the long-term performance of modified soil. UCS results were used for this purpose, which were done on automatic loading machine set at rate 1.25 mm/min. These results were plotted to examine the stabilized soil resistance to deformation as shown in Figure 49. These results are for soil stabilized at 90 days of curing. Results demonstrated that the gradient of line for soil stabilized with 0:100 is very low and does not change abruptly over time. The strength doesn't increase quickly but in slow manner. This also happens when it fails, with slow and steady failure. This shows the untreated soil acted like ductile material. This often is caused by hardening under the action of strain in soil.



Figure 49: Stress- Strain Behavior of Composite Binder (CCR:WSA) Stabilized Soil at 90 Days of Curing

For soil stabilized with binder ratios, there was increase in peak strength values. Gradient of line was getting high with increase in the CCR content. Peak stress reaches 4647 kPa after CCR increases to 75% of whole binder content in soil. It was due pozzolanic reaction that occurred after the bonding between soil particles due cementitious products formation between them. This value was found at 90 days of curing. These stress-strain plots are very important for settlement calculations. Due to increase in the CCR content in mixture, curves moves to higher stiffness section of figure. This helped in the resistance against stain conditions for soil stabilized.



**Figure 50:** Secant Modulus E50 of Composite Binder (CCR:WSA) Stabilized Soil Stiffness of soil stabilized with binder was plotted as shown in Figure 50. E<sub>50</sub> is secant modulus of stiffness of soil. It is ratio of stress to strain at 50% of maximum

stress in curve. Stiffness of soil can be assessed from this parameter. Results indicated a rising trend in stiffness values with addition of CCR proportion in soil. It was due pozzolanic reaction that occurred after the bonding between soil particles due cementitious products formation between them. Stiffness was 212.25 kPa for Nandipur soil without any additive.

Stiffness increased to 245 MPa after 28 days of curing by binder. This increased stiffness results in lower strain in soil stabilized under stress conditions. Secant modulus increases to 541 MPa after 90 days of curing due to binder addition in soil. This occurred at 75:25 binder. At all curing conditions, this binder had highest stiffness values. Al-Jabban et al. also reported similar kind of behavior in soil modification study on soil improvement [43].

# 4.4.8 XRD

This test was conducted on soil stabilized with binder 75:25, which was cured in sealed box at ambient temperature for 90 days. This test was done to observe the mineralogical changes that occurred after binder was added to soil in ratios. Figure 51 illustrated the outcome of this analysis. There are two patterns shown in the figure, upper one is for stabilized with 75:25 and lower one is for untreated clay soil. The maximum strength of soil occurred at 75:25 binder. This occurred at all curing periods. XRD test was performed to identify the products formed that increased the strength in soil binder mixture. It can be seen from the figure that there two main products formed as results of soil stabilization with binder, which are following:

- Sismondine (G)  $\rightarrow$  (29.4°, 37.4°, 51°)
- ➤ Calcium Silicate Hydrate (CSH)  $\rightarrow$  (27.2°, 36°, 47.5°)

CCR has portlandite as its main constituent, which was determined through its XRD patterns. Portlandite  $(Ca(OH)^2)$  contains  $Ca^{+2}$  and  $OH^-$ . After mixing water with

soil and composite binder mixture, hydration process happens. This will cause  $Ca(OH)^2$  to split up into  $Ca^{+2}$  and  $OH^-$ .  $Ca^{+2}$  will increase in the mixture. Calcium ions will react negative charged clay surface. These ions will take the place of Na<sup>+</sup> and other monovalent cations present in the water solution around clay platelets. This process will cause cation exchange in clay-binder composite blend. This will affect the double layer around the clay surfaces. Replacement of monovalent cations by divalent cations caused a declining trend in the thickness of double layer. This decreases the distance between the clay particles. Repulsive forces depicted a declining trend. An increase in attraction between soil particles will occur.

Gismondine occurred three times on XRD pattern of soil stabilized. While CSH also found at three different locations in XRD pattern of soil stabilized with binder 75:25. These two products linked the soil particles together and formed strong bonds between them.

There were other hydration products which were formed and are given below:

- Ettringite  $\rightarrow$  (15.8°)
- Calcite (C)  $\rightarrow$  (29.4°, 43.1°)
- Cowlesite  $\rightarrow (10.4^\circ, 17.4^\circ)$
- Calcium Chloroaluminate hydrate which is also called Friedel's Salt (Fs)  $\rightarrow$  (11.15°)

Ettringite was found at one locations on XRD pattern of stabilized soil. Calcite was found at two locations. Calcite was formed after the reaction of calcium content with air molecules. As samples for XRD were taken from UCS sample. This occurred during the experimentation and curing phase of UCS samples. Air gets in contact with (Ca(OH)<sup>2</sup>), which was present in soil binder mixture and is main constituent of CCR.

Calcite formed increased the bonding between the soil particles and strength of soil increased.

It is reported in literature that Friedel's salt fills the empty void spaces between particles [44]. This property will help in increasing the compactness of soil stabilized. Cowlesite peaks were located at two locations. It is a CASH product and helps in cementing soil particles [45]. These findings are aligned with the previous studies [46], [47].



Figure 51: XRD Patterns of Composite Binder (CCR:WSA) Stabilized Soil

The peaks of montmorillonite, illite and kaolinite had lowered in soil stabilized as compared to original soil. This is similar with the peaks of Quartz. As a result of stabilization of soil, the peaks intensity depicted a reducing trend. The intensity of these peaks decreased. It can be seen that portlandite peak had disappeared from the XRD pattern of stabilized soil. CCR has two main constituents. There were many peaks of Portlandite in the XRT pattern of CCR but it is not present in the stabilized soil XRD pattern. It can be concluded that calcium content from CCR has reacted with silica content available in the mixture. Pozzolanic products are formed as a result of reaction between them. 4.4.9 SEM

This was conducted to understand the alterations that occurred in the morphology of stabilized soil. Composite binder 75:25 performed the best out of all the binder ratios as demonstrated by previous sections. The maximum strength was achieved at this binder ratio. So to understand the mechanism behind this, SEM test was performed. The was conducted for samples cured for 7, 28 and 90 days. Figure 52 demonstrated the SEM images of these samples,

The first two images (a), (b) were of samples that were cured for one week. These images show that after the addition of CCR in the soil, particles have bonded with each other in face-to-face orientation, after curing for 1 week. These aggregations will then make Edge to edge and edge to face associations, that are called flocs, as shown in Figure 52 (a). These connections will reduce the available surface area for water to attach and decrease the water demand in the soil stabilized. This will reduce the plasticity behavior of soil.

In Figure 52 (c), it can be seen that there are tube like structures present. These are particles of WSA. WSA particles have pores on the surface. And these have mean diameter of around 5  $\mu$ m. The tube-like structures are approximately 90  $\mu$ m long. The diameter of these structures is around 10  $\mu$ m. The porous nature of these particles make them store the available moisture. These pores then provide space for reactions that occur in soil binder matrix. Calcium content will react with silica content present in ash in the presence of moisture on its surface.

These reactions remove silica from ash content. It was found out that these reaction have caused etched patches on the ash particles, as depicted in Figure 52 (c). The silica content reacted with calcium content and formed cementitious hydrates. EDS test was conducted to verify this and it was found that Calcium silicate hydrate was

formed near these tube ash particles. Calcium aluminosilicate hydrate was also formed. These products are the cause of increase strength in the binder stabilized soil.

In Figure 52 (d) & (e), it can be seen that crystalline surfaces are prevalent in soil matrix. Observations revealed that calcite compounds were detected in these locations. These images are of sample which was cured for 90 days. It was reported in literature that there are three crystal forms in which calcium carbonate is found in nature [27], [48].

- 1. Aragonite (which are needle like in crystal structure)
- 2. Vaterite (which are globular like in crystal structure)
- 3. Calcite (which are rhombohedral and scalenohedral like in crystal structure)

The most common crystal form of them is calcium carbonate. It has been found in literature that calcium carbonate occurs in scalenohedral crystal structure at specific conditions [48], which are following:

- High pH $\rightarrow$  (12-13)
- Calcium rich environment

EDX test was performed and its was found from the results that it was calcite that occurred in the sample cured for 90 days as shown in Table 3. When the section of Figure 52 (d) was zoomed in, it can be seen that scalenohedral crystals are found in it. Observations revealed that the length of these scalenohedral crystals was around 4.5  $\mu$ m. These calcite crystals filled the spaces between particles and made the soil fabric denser and stronger.



Figure 52: SEM Results Analysis of Composite Binder (CCR:WSA) Stabilized Soil at Curing Periods: (a) 7 Days , (b) 7 Days , (c) 28 Days , (d) 90 Days

4.4.10 EDS

EDS test was performed to determine the elemental and mineralogical changes that occurred due to soil stabilization process. It was performed on four spots on samples which are indicated in the Figure 52, as A-1, A-2, A-3 & A-4. EDS detector was fitted inside the SEM device. It can perform on spot chemical analysis. Table 3 illustrated the findings of EDS analysis. It can be concluded from the table that at location A-1, soil particle is present. At location A-2, Calcium aluminosilicate hydrate (CASH) product is formed as a result of soil stabilization with binder. At location A-3, Calcium silicate hydrate is formed, which is hydration product. At location A-4, elemental composition indicate the presence of Calcite. These findings validate the results obtained in strength and microstructural analysis. It can be concluded that composite binder (CCR: WSA) provides an effective solution for stabilization of untreated soil from Nandipur.

		Elemental Composition %							
Figure No.	Spot ID								
		Si	Al	Ca	K	Mg	Fe	Na	0
11 (b)	A-1	17.6	9.8	3	1.4	1.3	1	2.8	63.1
				10.0	• •				
11 (c)	A-2	17.9	8.9	10.3	2.9	-	5.1	-	54.9
11 (c)	Λ_3	177	ЛЛ	178	0.4	_	11	_	58.6
11 (0)	A-3	1/./	7.7	17.0	0.4	-	1.1	-	50.0
11 (e)	A-4	1.2	1.1	32.9	_	_	_	_	64.8

Table 3: EDS Analysis of Composite Binder (CCR:WSA) Stabilized Soil

# **CHAPTER 5: CONCLUSIONS & RECOMMENDATIONS**

## 5.1 Conclusions

The main aim of this study was to evaluate the effects of different CCR:WSA binder ratios on the stabilization of expansive soil. The experimental analysis performed in this research study yields the conclusions that are given below:

- The maximum dry density of soil stabilized with the addition of WSA decreased while at same time OMC of soil increased. The compressive strength of soil increased with addition of wheat straw ash in soil due to filler effect of ash particles. Optimum was found at 12.5 % ash content.
- Soil swelling characteristics reduced by 47% at same WSA content. Plasticity index of soil reduced by 35% after the addition of optimum WSA in soil. There was enhancement of almost 80% in the friction angle of soil with the addition of optimum WSA content.
- Plasticity index of soil reduced by 76% after the addition of optimum CCR:WSA binder in soil. Soil changed from high plastic swelling soil to low plastic soil.
- CCR:WSA binder has proved to be very efficient for swelling soils. It reduced the swelling parameters by 85%.
- The UCS of soil stabilized with CCR:WSA composite binder, improved remarkably. It increased by 35 times at CCR:WSA ratio of 75:25. While stiffness (E<sub>50</sub>) improved by 46 times at optimum composite binder ratio.
- Friction angle improved by 2.4 times at optimum CCR:WSA ratio. While there is almost 1.4 increase in cohesion of soil stabilized with optimum CCR:WSA ratio.
- XRD results revealed the formation of CSH and Gismondine as main cementitious product in soil stabilized with optimum CCR:WSA ratio. XRD results were

validated by SEM & EDS results with appearance of CSH & CASH products. Calcite was found in sample cured for 90 days in scalenohedral form.

- As CCR content increased in binder mixed soil, the plasticity and swelling properties of soil reduced. On the other hand, with rise in CCR content in binder mixed soil, the strength characteristics of soil increased.
- Optimum ratio of composite binder CCR:WSA was found to be 75:25 with 12.5
   % binder content for stabilization of expansive soil from Nandipur.

These findings clearly proves the effectiveness of CCR:WSA composite binder. This study helps in sustainable use of these additives in construction sector. This helps in saving precious land, which was going to be used for disposal of these wastes. This composite binder provides efficient solution for expansive soil problem by using wastes.

#### 5.2 **Recommendations**

- Expansive soil was used in this study for stabilization objectives. It is recommended to use different soils with low and high plasticity for further investigation of the effect of CCR:WSA binder.
- The peak stress of soil stabilized with CCR:WSA binder increased tremendously making the soil very stiff. It is recommended to make efforts to counter this by using additives or other suitable solutions.
- Mechanical and microstructural behavior of soil was observed in this study. It is recommended to include durability testing in future studies.
- Cost comparison should be carried out between expansive soil improved with addition of CCR:WSA binder and lime.

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