# A STUDY ON STABILIZATION/SOLIDIFICATION OF OIL-CONTAMINATED SOILS

BY

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IN THE NAME OF ALLAH, MOST GRACIOUS, MOST MERCIFUL

This humble work is dedicated to:

My beloved parents, wife and brothers for all their love, encouragement and support

`

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# Abstract


Full name : **MOHAMMED SALEM MUBARAK BA-NAIMOON**

Thesis title : **A STUDY ON STABILIZATION/SOLIDIFICATION OF OIL-CONTAMINATED SOILS**

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During the Gulf War in 1990-91, the shoreline spanning around 755 km up to the industrial city of Jubail in Saudi Arabia was heavily polluted with the crude oil posing a serious threat to the environment and flora and fauna in the sea. Recently, some companies have been assigned projects related to recovery of the flora and fauna using some specific technique. The technique adopted for restoration of the flora and fauna requires excavation of the contaminated soil along the shoreline for forming channels so that the sea water can move forth and back through these channels during high tides. As a result, a huge quantity of the contaminated spoils is being generated which cannot be dumped in landfills without proper treatment.

Stabilization/Solidification (S/S) technique has been effectively used for treating and reclaiming soils contaminated by petroleum oil utilizing various chemical additives, termed as 'stabilizers', such as Portland cement, lime, fly ash, etc. When contaminated soil is mixed with stabilizer, the soil gets chemically stabilized and contaminants are immobilized eliminating the leaching problem. The binding effect of stabilizer physically solidifies the mixture which improves the structural and geotechnical properties of the treated soil. The treated soil can be utilized in many ways such as: for landfilling and backfilling, for road construction, and for making brick blocks, etc. However, for a given contaminated soil, there is need to select a suitable type and optimum dosage of a stabilizer which can be used for S/S treatment to achieve maximum technical benefits and economy.

The main objective of the present study was to explore the possibility of treatment of spoils using S/S technology so as to ensure either the safe disposal of the treated spoils or utilization of the treated spoils for engineering applications such as formation of sub-base or base courses in road construction. To achieve the objectives, three levels of the oil-contaminated soils (high, medium and low) were collected from different locations and characterized using different laboratory tests. For S/S treatment, Portland cement and different cementitious waste materials such as CKD, EAFD and LSP were used as alternative stabilizers. The treatment of high oil contaminated soil (HOC) was first considered using 23 combinations of the stabilizers and their different dosages. The effectiveness of the S/S treatment was evaluated based on the unconfined compressive strength (UCS), California bearing ratio (CBR), toxicity characteristics leaching

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procedure (TCLP), X-ray diffraction (XRD) and scanning electron microscopy (SEM) tests. Test results indicated that the HOC mixtures stabilized with 7 % cement alone, 30% CKD plus 5 % cement, and 15% LSP plus 7% can be utilized in sub-base course in the construction of rigid as well as flexible pavements. Therefore, for S/S treatment of the mixtures of medium oil contaminated soil (MOC) and low oil contaminated soil (LOC), only these three combinations of stabilizers were adopted and it was found that the S/S-treated MOC and LOC soils can also be used as sub-base materials in the construction of pavements. The TCLP results indicate that the concentration of heavy metals in all the S/S-treated soil mixtures were tremendously reduced and were found to be within EPA limits.

**MASTER OF SCIENCE DEGREE**

**KING FAHD UNIVERSITY OF PETROLEUM & MINERALS**

**Dhahran, Saudi Arabia**

# Abstract (Arabic)

## ملخص الرسالة

**الاسم الكامل: محمد سالم مبارك بانعيمون**

**عنوان الرسالة: دراسة على تثبيت وتصليب التربة الملوثة بالبترول الخام**

**التخصص: هندسة مدنية (جيوتقنية)**

**تاريخ الدرجة العلمية: ديسمبر 2013**


خلال حرب الخليج في عام 1990م-1991م، تلوث الخط الساحلي الذي يمتد حوالي 755 كم حتى مدينة الجبيل الصناعية في المملكة العربية السعودية تلوثا شديدا بالنفط الخام مما يشكل تهديداً خطيراً على البيئة والنباتات والحيوانات في البحر. مؤخراً، بعض الشركات وقعت على مشاريع متعلقة بحماية البيئة لهذه النباتات والحيوانات باستخدام بعض تقنيات محددة. أعتمدت التقنية لأستعادة الحياة النباتية والحيوانية الملوثة على حفر مناسب على طول الخط الساحلي لتشكيل قنوات بحيث يمكن أن تتحرك مياه البحر ذهاباً وإياباً من خلال هذه القنوات أثناء المد العالي.نتيجةً لذلك، فأنّ كميات هائلة من التربة الملوثة تم تجميعها في أماكن خاصة من جرّاء هذه الحفريات حيث أنه لا يمكن أن تُرمى في المقالب الخاصة دون علاج مناسب لها.

إنّ تقنية التثبيت والتصلب (S/S) أصبحت تقنية فعالة تُستخدم لعلاج واستصلاح التربة الملوثة بالبترول الخام باستخدام إضافات كيميائية مختلفة، تُوصف بأنّها "مثبتات"، مثل الأسمنت والجير والرماد المتطاير، الخ. عندما تُخلط التربة الملوثة مع هذه المثبتات ، فأنّ التربة تصبح مثبتة ومستقرة كيميائياً مما يؤدي إلى تقليل والحد من مشكلة الرشح. إنّ التأثيرالفيزيائي لهذه المثبات يعمل على تصلب الخليط مما يُحسّن من الخصائص التركيبية والجيوتقنية للتربة المعالجة. هذه التربة المعالجة يُمكن الأستفادة منها في نواحٍ كثيرة مثل: الردميات، الطرق، صناعة الطوب، إلخ. وعليه ، فأنّه لعلاج التربة الملوثة يحتاج الى تحديد نوع مناسب من المثبتات وجرعة أمثل يُمكن استخدامها لعلاج هذه التربة بواسطة تقنية التثبيت والتصلب S/S لتحقيق أقصى قدر من الفوائد الفنية والأقتصادية.

إنّ الهدف الرئيسي من هذه الدراسة هوالبحث عن إمكانية علاج هذه التربة الملوثة باستخدام تكنولوجيا S/S وذلك لضمان التخلص الآمن من للتربة المعالجة أو الأستفادة منها في بعض التطبيقات الهندسية مثل الطرق كطبقة ثانوية أو رئيسة. من أجل تحقيق هذه الأهداف، تم جمع ثلاثة مستويات من التربة الملوثة بالنفط ( عالية ومتوسطة ومنخفضة ) من مواقع مختلفة ، وتصنيفها باستخدام اختبارات معملية مختلفة . من أجل تطبيق تقنية التثبيت والتصلبS/S للعلاج، تمّ استخدام الأسمنت البورتلاندي و مختلف المواد الناتجة من الصناعة مثل CKD، EAFD وLSP كمثبتات بديلة.في هذه الدراسة تمّ أولاً معالجة التربة عالية التلوث بالبترول HOC باستخدام 23 مجموعة مكوّنة باستخدام المثبتات وبجرعات مختلفة. تمّ تقييم فعالية تقنية التثبيت والتصلب S/S لمعالجة التربة الملوثة بواسطة اختبارات شملت الدمك،مقاومة الإنضغاط الغير محصور(UCS) ،التحمل (CBR)،أشعة اكس (X-RD) والتصوير الألكتروني الميكروسكوبي (SEM) وخصائص الرشح السمي (TCLP). أشارت نتائج هذه الدراسة بأنّ التربة التي تحتوي على نسبة عالية من التلوث بالبترول الخام HOC المعالجة بـ7% من الأسمنت،30% CKD بالأضافة إلى 5% من الأسمنت و15%LSP بالأضافة إلى 7% من الأسمنت يُمكن أن تكون مناسبة لأستخدامها للطبقة الثانوية للطرق الخرسانية والأسفلتية. أيضاً تمّ معالجة التربة متوسطة التلوث بالبترول MOC و منخفضة التلوث بالبترول LOC باستخدام فقط هذه المجموعات الثلاث من المثبتات حيث تبين أيضاً أنّ المعالجة بواسطة تقنية التثبيت والتصلب S/S لهما يُمكن أيضاً أن تكون مناسبة لأستخدامها للطبقة الثانوية للطرق الخرسانية والأسفلتية. كما أشارت نتائج الرشح TCLP أن تركيز المعادن الثقيلة في جميع خلطات التربة المعالجة بواسطة تقنية التثبيت والتصلب S/S قد انخفضت بشكل كبير وأصبحت ضمن الحدود المسوح بها وفقاً لمعاييروكالات حماية البيئة EPA.


**درجة الماجستير**

**جــامعـة المـلك فـهـد للبترول والـمعـادن**

**الظـهران، المـملكة العربيـة السـعوديـة**

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# CHAPTER 1

# INTRODUCTION

## 1.1 General

In fact, there are numerous techniques for soil waste remediation can be used for treatment but stabilization/solidification (S/S) appears to be more effective and accurate because it binds the compounds of hazardous waste stream into a stable insoluble form (stabilization) or entrapping the waste within a solid cementitious matrix (solidification) (Wiles.1987). In addition, The S/S technology has been described by the U.S. Environmental Protection Agency (EPA) as the greatest accessible technology for 57 RCRA (Resource Conservation and Recovery Act) listed hazardous wastes (Paria and Yuet, 2006).

The Stabilization/Solidification can be defined as a clean-up technology which involves mixing of soil with contaminated sludge and additives like Portland cement, lime/fly ash and cement/fly ash  so as to immobilize the contaminants within the soil from being released to the environment causing groundwater pollution (Paria and Yuet ,2006). Stabilization refers to those techniques which reduce the hazard potential of by changing the soil contaminants so that they become less harmful or less mobile (U. S. Army Corps of Engineers, 1995). Solidification refers to techniques that encapsulate the waste in a monolithic solid of high structural integrity. Solidification changes the physical properties of a contaminated substance; these desired changes include increase in compressive strength, decrease in permeability and encapsulation of hazardous constituents (U. S. Army Corps of Engineers, 1995).

## 1.2 S/S Technology Advantages and Applications

In fact, the Stabilization/Solidification does not remove the contaminants from the soil but it only prevents soil contaminants from spreading into the surrounding environment. The S/S process has found applications in the treatment of liquids, soils and sludge contaminated with inorganic materials but may not be employed for organically contaminated soil due to their volatility and interference with the reagent setting process. Mainly, there are two types of S/S reagents, organic and inorganic (U. S. Army Corps of Engineers, 1995). Organic reagents are rarely used the normal processing steps when using inorganic reagents are to (a) chemically react with all the water present, (b) chemically react with the contaminants to render them insoluble, and then (c) encapsulate the products while inorganic reagents most often used for S/S include Portland cement, fly ash, lime, phosphates, and kiln dust from lime and cement production. All of these reagents have basically the same general types of active ingredients as far as S/S reactions are concerned. These active ingredients include: $SiO_2$, CaO, MgO, $Al_2O_3$, and $Fe_2O_3$.

There are many advantages of the S/S technology over other traditional remediation technologies. Some of the advantages are listed below (Wiles, 1986):

i. Good long-term stability, both physically and chemically,
ii. Good impact and comprehensive strength,
iii. High resistance against biodegradation,
iv. Low water permeability relatively, and
v. Non-toxicity of the chemical ingredients used for S/S.

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It is reported that the oil contamination of soil decrease the maximum dry density by 4%, cohesion by 66%, angle of internal friction by 23% and unconfined compressive strength (UCS) by 35%, and increase in liquid limit by 11%. Challenges and achievements have been made to stabilize oil contaminated soils using various additives such as cement, lime, and fly ash independently as well as an admixture of different combinations (Shah et al., 2002).

The results of tests by using S/S technology indicate that the stabilization agents improve the geotechnical properties of the soil by the way of cation exchange, agglomeration, and cementing. The improvement in unconfined compressive strength, cohesion and angle of internal friction can be attributed to neo-formations, such as calcium silicate hydrates (C-S-H) that coat and bind the soil particles (Shah et al., 2002).

The treatment processes by S/S technology are designed to achieve one or more of the following:

i. Improve the handling and physical characteristics of the waste;
ii. Decrease the surface area of the waste mass across which loss of contaminants can occur; and
iii. Reduce the solubility of hazardous constituents in the waste.

The degree of effectiveness of S/S treatment requires the measurement of physical, engineering, and chemical properties of the Stabilized/Solidified material. Some of the tests that are normally carried out to evaluate the effectiveness of the S/S technology include the following:

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i. Strength test such as unconfined compressive strength.

ii. Permeability test.

iii. Leachability using Toxicity Characteristics Leaching Procedure (TCLP).

Additional tests relevant to the evaluation of the effectiveness of S/S treated soil include:, distribution of particle, compaction, California bearing ratio (CBR), porosity and bulk density tests.

The applications of S/S technology involves various steps the in-situ, as follows: (a) collection and characterization of sample (b) excavation of the contaminated soil, (c) screening and crushing of over-sized pieces, (d) buffering of soil pH, (e) mixing contaminated soil with the S/S binding reagents, (f) testing of the treated soil to verify the success of the treatment, and (g) using the treated soil as sub-base material for road construction and other usages (Wiles, 1986). Some of the applications of the S/S technology for in-situ treatment of polluted soils and their ex-situ examples are as follows:

i. Remediation of lead-and-petroleum-contaminated soils at a Boston Brownfield site using cement-based S/S.
ii. Redevelopment of a former manufactured gas plant site as a research park in Cambridge, Massachusetts using cement-based S/S treatment
iii. Reuse of the S/S treated arsenic-and-creosote-impacted soil at a former wood-treating site as base for pavement in Port Newark, New Jersey.
iv. Augusta manufactured gas plant clean-up using cement-based S/S in Augusta, Georgia.
v. Reuse of New York Harbor sediments after S/S treatment of the sediments.
vi. S/S of contaminated soil at 90th South Battery Site, West Jordan, Utah.

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## 1.3 Background of Soil Contamination by Oil spill in Saudi Arabia

Between January 21 and mid of March, 1991, more than twenty years ago, the northern half of the Arabian Gulf was hit by the heaviest and worst oil spill in history. On January 21, 1991, a few days after the Coalition Forces launched an air campaign against Iraq, the Iraqi military forces in Kuwait opened valves at the Sea Island oil terminal near Kuwait City and released large quantities of crude oil into the Gulf, an act of environmental warfare. About 1,700,000 million cubic meters of light Kuwaiti oil was intentionally released. Of this volume, 40% was assumed to have evaporated and 10% to have been dissolved in the seawater body. About 185,000 cubic meters was recovered by Saudi Arabian forces. It was estimated that about 400,000 cubic meters of the released oil polluted more than 755 km of the Saudi shorelines.

Eighteen years later, an estimated 8,100,000 cubic meters of oiled sediments remained within shoreline habitats ranging from exposed rocky shores to highly sheltered mud flats. Approximately 70% of the oiled sediments occur in sheltered habitats (mud tidal flats and salt marshes), mostly as oiled crab burrows, with liquid oil remaining in the burrows to depths exceeding 50 cm. Habitats exposed to the greatest amount of wave activity, such as the outer sand beaches, contain the smallest but a major amount of oil, which is buried by few centimeters (about 20 cm) of clean sediment. Ecological recovery was lowest in mangroves and salt marshes, with over 80% of the upper intertidal zones having reduced species richness and a disturbed community structure.

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## 1.4 Problem Statement

As it was mentioned in the background of this study, the environmental pollution, which runs from Kuwait beaches to the shores borders of Saudi Arabia, which is located near Jubail industrial area, due to crude oil, is dangerous on the environmental. As a result, the possibility of dumping the spoils in landfills is ruled out because of the huge quantity of the spoils (approximately 500,000 $m^3$) and also due to the complexity of the contaminated sediments. Therefore, it is essential to explore the feasibility of treatment of spoils using S/S technology for possible reuse of the treated sediments and the ability for engineering application such as using them as sub-base or base material for road construction, back fill or construction material, etc. Huge quantity of contaminated soil after treatment should be consumed as much as possible according to the recommendation and tests results of the present study as the appropriate solution for reducing the cost of disposal.

## 1.5 Objectives

The main objective of the proposed study was to explore possibilities of an effective and economical treatment and utilization of soils found contaminated by oil spill on three different sites in the coastal area of Jubail in Saudi Arabia utilizing various available stabilizers such as, CKD, EAFD and LSP, generally used for S/S treatment.

The specific objectives are as follows:

1. To characterize the soils collected from all the three sites for classifying the soils based on geotechnical properties and pollution levels.

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2. To select the ranges of stabilizers (Cement, CKD, EAFD and LSP) for S/S treatment of highest class of soil.
3. To carry out different tests after S/S treatment to evaluate the effectiveness of the treatment for each class of soil.
4. To conduct statistical analysis of the test results to identify the effect of each factor on effectiveness of S/S treatment.
5. To evaluate the technical and economic viability of the selected S/S treatments based on the analysis of test results
6. To suggest suitable applications of S/S treated soils.

The flow chart for achieving the above objectives is shown in Figure 1.1.

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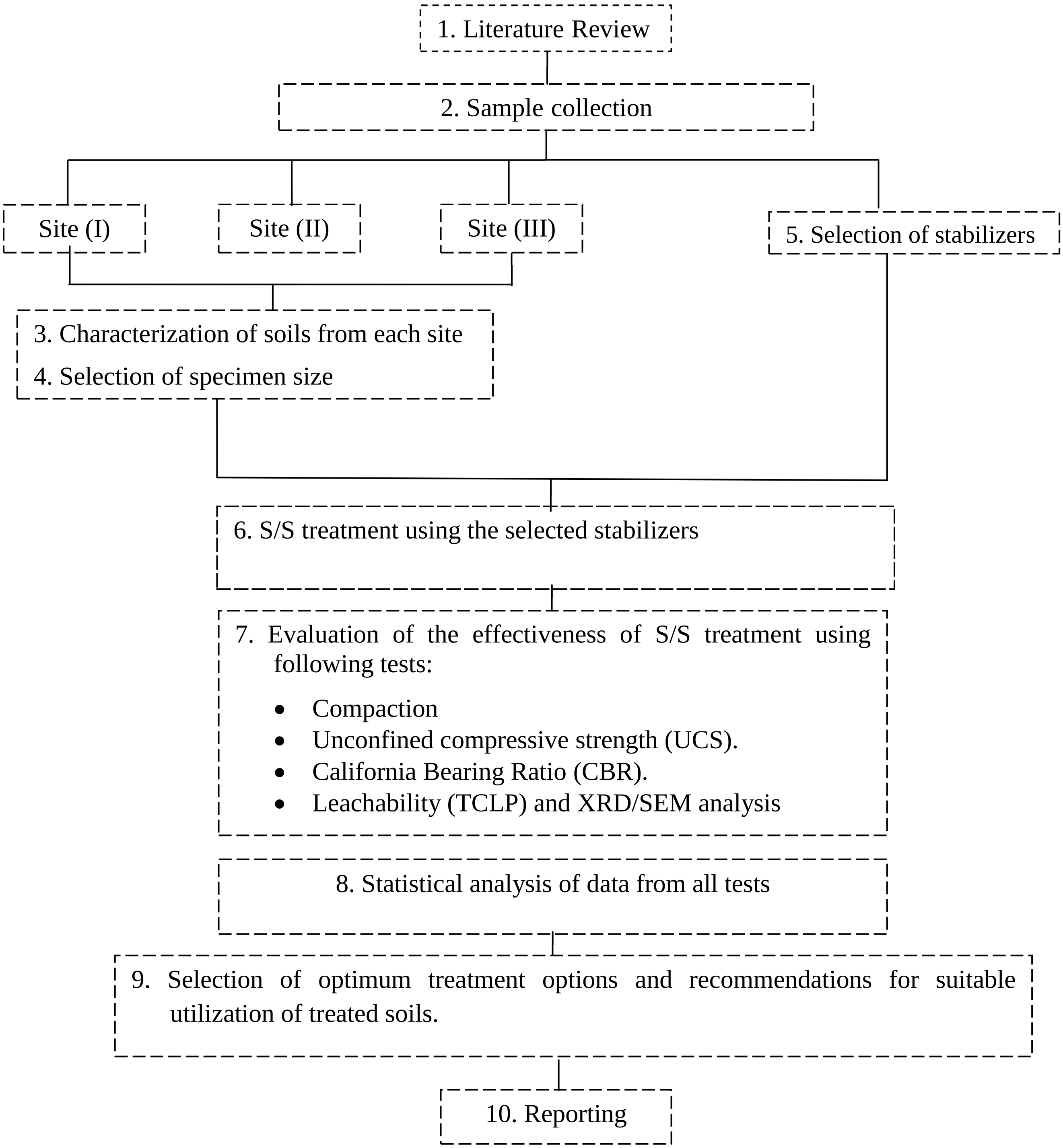


Figure 1.1: Flow chart showing different tasks

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# CHAPTER 2

# LITERATURE REVIEW

The protection of the environment from hazardous pollutants associated with the soils contaminated with oil is a major concern in today's industrialized world, especially the developing nations. Therefore, there is necessity to cure the contaminated soils to reduce the potential release of crude oil into the environment. The S/S technology had been adopted by governmental agencies and individuals and it has proven to be reliable. The S/S treatment process consists of the addition of cementitious binders like cement, cement kiln dust, lime and fly ash, etc. to contaminated soils to form a slurry or liquid waste so that the contaminants from the soil can be prevented from affecting the groundwater and subsequently the environment.

## 2.1 Stabilization/Solidifications (S/S) technology

In the 1950's, S/S technology was originally developed but has been used recently as treatment for industrial, hazardous and some selected radioactive wastes. S/S defined as treatment processes designed to improve waste-handling and physical characteristics, decrease surface area across which pollutants can transfer or leach, or limit the solubility or reduce the toxicity of hazardous constituents. S/S treatment mainly consists of mixing the contaminated material with the suitable stabilizers. Lime, cement, and other cementitious industrial waste materials are commonly used for S/S treatment.

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## 2.2 Stabilizers used in (S/S) Technology

Stabilizers used in the S/S technology are either inorganic and the organic stabilizers, and should be safely for the environmental, offered locally and economically worthwhile. Initially, lime and cement were conventionally used for S/S treatment but later on the utilization of waste materials having cementing properties for S/S treatment has become a common practice. Tuncan et al., 2000 conducted a study to stabilize petroleum-contaminated soils with cement, fly ash and lime for use as sub-base material for road. They found that the addition of 5% cement, 10% fly ash and 20% lime showed the better strength. The following sub-sections discuss about some locally available waste materials that can be used as stabilizers.

### 2.2.1 Cement Kiln Dust (CKD)

In the Kingdom of Saudi Arabia, there are many factories that produce thousands of tons of cement daily. Large quantities of cement kiln dust (CKD), which is a very fine powder, are generated from these cement factories posing the problem of disposal and also environmental pollution. In 2010, the production of cement in the Kingdom was 53 million tons (Vasehi, 2010). Around 6 to 7% of CKD (by mass of cement produced) is generated (Al-Refeai et al, 1999) from a cement factory. The Arabian Cement Company Ltd. (ACCL), Jeddah, is currently producing about one thousand tons of CKD per day. Many manufacturers of cement are reluctant to recycle CKD into the production line due to the high chlorides level and alkalis in CKD, (Kessler, 1995; USEPA, 1998).

Haynes and Kramer (1982) have reported an approximate phase composition of CKD as shown in Table 2.1

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Table 2.1: Approximate Composition of CKD (Haynes and Kramer, 1982)

| **Compound** | **% by weight** |
|---|---|
| $CaCO_3$ | 55.5 |
| $SiO_2$ | 13.6 |
| CaO | 8.1 |
| $K_2SO_4$ | 5.9 |
| $Fe_2O_3$ | 2.1 |
| KCI | 1.4 |
| MgO | 1.3 |
| $Na_2SO_4$ | 1.3 |

CKD is categorized as waste material can be used in many applications including the following (Bhatty, 1995):

i. Agriculture: potash/lime source and animal feed.

ii. Civil engineering: fill, soil stabilization, fly ash stabilization and blacktop filler.

iii. Building materials: lightweight aggregates, blocks, low strength concrete and masonry cement.

iv. Sewage and water treatment: coagulation aid and sludge stabilization.

v. Pollution control: sulfur absorbent, waste treatment and solidification.

Al-Amoudi et al. (2006) studied the stabilization of four eastern Saudi soils by using CKD. The results indicated decrease of the dry density and increase in the optimum moisture content when CKD was mixed to the different types of soil, namely sandy sabkha, white marl with low plasticity, cohesionless marl and plastic marl. The addition

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of 50% CKD to the sandy sabkha, white marl with low plasticity, cohesionless marl and plastic marl soils increased the unconfined compressive strength by about 5.66, 1.69, 1.41 and 13.2 times, respectively.

Shabel (2006) studied the stabilization of sabkha soil of Jizan, Saudi Arabia, by using CKD and cement. The results indicated improvement in engineering properties of sabkha soil of Jizan by addition of CKD stabilizer. Sabkha soil of Jizan treated with 2% cement + 20% CKD satisfied the requirements of the USACE 7-days strength that can be used as sub-base material in rigid pavements.

Al-Aghbari and Dutta (2008) studied the effect of cement and cement by-pass dust on the engineering properties of sand. They found that sand with ordinary Portland cement represented a good material and can be used for base or sub-base course application while the sand with cement by-pass dust can be used for improving the bearing capacity of sand to support low to moderate rise building.

Maslehuddin et al. (2009) studied the properties of CKD blended cement concrete specimens with 0%, 5%, 10%, and 15% CKD, replacing ASTM C 150 Type 1 and Type V cements. The results indicated that 28-day compressive strength of CKD cement mortar is higher than that of Type I cement mortar and the shrinkage of CKD cement mortar increases with an increase in the amount of CKD.

### 2.2.2 Electric Arc Furnace Dust (EAFD)

Electric arc furnace dust (EAFD) is a fine grained, high-density material containing high amounts of zinc and iron and large amounts of calcium, manganese, magnesium, lead and chromium. It is in the form of very fine powder forming major part of the smoke or

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fume from the furnace. The powder from the furnace is drawn through cooling pipes and collected in specially designed bag filters and it is also called as bag house dust.

It is reported that about 15 to 20 kg of EAFD is generated through production of one ton of steel (Recupac, 2012). In In Saudi Arabia, there are four groups of companies for steel production: SABIC, Al-Ettefaq, Al-Rajhi and Al-Yamama. The annual steel production in Saudi Arabia is estimated to be about 471,000 tons (Article, 2010). Therefore, the annual production of EAFD in the Kingdom will approximately be 8,242 tons. The fine dust particles of EAFD are released in atmosphere and it forms a major pollution problem.

According to United States Environmental Protection Agency (US EPA), the EAFD is considered as hazardous material, it must be stored in specialized landfill. On the other hand, EAFD can be used as secondary raw material for production of zinc or other products due its high content of zinc. So, the presence of zinc and pozzolanic materials will improve the properties of concrete. Hence, EAFD can be used for the production of medium to low strength concrete where it is found that the increase in EAFD contents would significantly delay the setting time of the concrete while the compressive and shearing strengths as well as resistance to abrasion will enhance (Carlos Alberto Caldas de Souza, 2010).

The chemical composition and physical properties of EAFD were investigated by Xuefeng and Yuhong (Xuefeng X, 1998). It was reported that the use of EAFD in cement is more economical than the use of iron ore. In addition, the quality of cement produced with EAFD meets the requirements for Chinese specifications.

It is reported that the effect of the addition of 10, 15 and 20 % EAFD on the mechanical

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properties and chloride penetration of Portland cement (De Sauza, et al., 2010). The researchers found that the addition of EAFD from 10 to 20 % will increase the axial compressive strength of the concrete as well as increase the tensile strength.

Maslehuddin et al. (2011) evaluated the mechanical properties and durability characteristics of ordinary Portland cement (OPC) and blended cement (with silica fume and fly ash) concrete specimens with electric arc furnace dust (EAFD). Results of their study indicated that the setting time and slump retention inclined to increase with the addition of EAFD. However, there was a gain in strength with the addition of EAFD. Further, the water absorption and chloride permeability were found to decrease and there was an increase in the corrosion resistance of concrete with EAFD when compared to OPC and blended concretes.

Alexandre et al. (2006) studied the effect of EAFD in pozzolana-modified Portland cement paste. To understand the residue effect and properties of cement paste in fresh and hardened states, setting time and heat of hydration were determined as well as mineralogical and micro structural characteristics were evaluated. Results indicated that the EAFD retards the Portland cement’s hydration reaction. At initial stages, the compressive strength was found to be less than control specimen but at advanced age significant gain was noted. The compressive strength with 5% EAFD was found to be similar to the reference MP cement paste at age of 28 days.

Carlos Alberto et al. (2010) studied the effect of EAFD on the mechanical and chemical performance of Portland cement concrete. They found that the increase in compressive strength of concrete specimen by the addition of EAFD in the range of 10 to 20 wt. (%). Also, the tensile strength and setting time of specimens increased with the addition of

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EAFD and the chloride penetration decreased. The acetic acid leaching and water solubility test results show low movement of potentially toxic elements from EAFD-based concrete.

### 2.2.3 Limestone Powder (LSP)

LSP is obtained from the crushing of carbonate rocks which are primary sources of coarse aggregates in the Central and Eastern Provinces of the Kingdom. LSP is available in abundance in Saudi Arabia.

LSP develops the hydration rate of cement compounds and increases the strength at early ages. It reacts with the alumina phase of cement to form a calcium mono carbo aluminate hydrate without substantial changes on the strength of blended cement. LSP has good ability of packing cement granular skeleton and a large dispersion of cement grains (Bonavetti, 2003) which helps in improving the quality of concrete.

The effect of replacement of LSP in pozzolanic cement was studied by Heikal et al. (2000). They reported reduction in the initial and final setting times by addition of LSP. Also, the porosity reduced due to filling of the pores between cement particles by carbo aluminate formed in the presence of LSP. However, the content of free lime and combined water increased with LSP content. The addition of LSP results an increase in the heat of hydration and compressive strength.

Dhir et al. (2007) studied the performance and mechanical properties of concrete produced by blending Portland cement and LSP. They replaced 15%, 25%, 35% and 45% of cement by LSP keeping cement contents in a range of 235 to 410 kg/m$^3$, and free water content of 185l/m$^3$. They found that there were minor differences in the performance between Portland cement and 15% LSP blended cement concretes at the

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same cement content and water-to-cement ratio. On the other hand, there was a decrease in the strength when the LSP content increased.

From the above information regarding use of EAFD and LSP, it is clear that unlike CKD, the utilization of EAFD and LSP is mostly explored in producing concrete. However, in the present study, the effect of EAFD and LSP on S/S treatment is considered along with other stabilizers.

## 2.3 Tests for Evaluating Effectiveness of S/S Treatment

For evaluating effectiveness of S/S treatment, it requires the measurement of physical, engineering and chemical properties of the stabilized/solidified material. It was reported by Malviya and Chaudhary (2006) that the degree of effectiveness of S/S treatment can be defined basically by two parameters, the strength and the leach resistance of the treated product. The micro-structural examination of the stabilized/solidified mass in the study on evaluation of effectiveness of S/S treatment, which makes better understanding of the nature of the S/S process, is also useful (Grega et al, 2001). Further, scanning electron microscope (SEM) and X-ray diffraction (XRD) tests were used for the micro-structural examination (Means et al, 1995). Some of the tests that are normally carried out to evaluate the effectiveness of the S/S technology include the following:

i. Compressive strength tests
ii. California Bearing Ratio tests(CBR)
iii. Permeability test
iv. Leachability tests using TCLP tests
v. XRD/SEM Tests

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### 2.3.1 Compressive strength

Strength testing is often used during a treatability study to indicate how well a material will endure stresses created by overburden and earth moving equipment (Wiles, 1987). Strength test data often provide a baseline comparison between stabilized and unstabilized waste materials. Unstabilized wastes generally do not exhibit good shear strength, but with cement stabilization, the strength is expected to increase significantly. The minimum required unconfined compressive strength for a stabilized/solidified material is evaluated to be 0.35 MPa (EPA), but in the UK, acceptable 28-day strength is 0.7 MPa, this might be increased depending on what the waste will be used of after S/S process which will be to increase the cement and other binder materials to be used in the waste. This test is usually accomplished with help of unconfined compression machine and varying maximum load, performed according to ASTM D2166-85. UCS tests are usually performed at different time intervals of 7, 14, 28, 90 days and one year to monitor the effect of the changes in the mineralogical composition of waste, with increasing time, and environmental exposure.

### 2.3.2 California Bearing Ratio tests (CBR) test

This test is performed by measuring the pressure required to penetrate a soil sample with a plunger of standard area. The measured pressure is then divided by the pressure required to achieve an equal penetration on a standard crushed rock material. The CBR test is described in ASTM Standards D1883-05 (for laboratory-prepared samples) and D4429 (for soils in place in field), and AASHTO T193. In this research the mixtures will subject to CBR test to know the ability of using treatment soils on road applications.

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### 2.3.3 Leachability test

The United States Environmental Protection Agency (USEPA) The Contaminant Leaching Procedure (TCLP) method is commonly used to determine if a waste is hazardous or otherwise (Qian et al., 2006). The TCLP is designed to simulate the leaching potential of a waste within an unmanaged landfill designed for municipal refuse. Following strength testing, samples derived from failed specimens or crushed specimens, were tested for their heavy metal leachability using the US Environmental Protection Agency (EPA) TCLP (US EPA,1995) test. During this leaching test, the solid was pulverized and mixed with an acetic acid solution (pH = 3 if solid pH is higher than 5 or pH= 5 if solid pH is lower than 5) at a solution to solid ratio of 20. The suspension was then tumbled for 18 h and following this, separation of the extract solution from the solids was achieved by filtration. Soluble contaminants concentrations in the solution were measured using an inductively coupled plasma atomic emission spectrometer. Hexavalent chromium concentrations in solution were evaluated using a UV–Vis spectrophotometer (Dermatas et al, 2006). All TCLP testing was performed on sample duplicates and average values were used. In addition, all analyses were performed by using two different quality control standards, as well as the method of standard additions (spiking), to ensure proper quality control of the reported results (Dermatas et al, 2006). After homogenization, the suspension was allowed to stand for 7 days, and soluble contaminants concentration will be determined according to the standard methods.

### 2.3.4 Permeability test

Maximum allowable permeability is usually specified by the regulating agency for treated materials. The permeability test is not a measure of leachability because having

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higher permeability does not infer that the waste has not been treated well. The permeability test for stabilization/solidification is usually carried out in accordance with the ASTM D 5084-90 standard. Consideration must be given to the confining pressure, gradient and the permeating fluids which will reflect the field condition.

### 2.3.5 XRD/SEM tests

X-ray diffraction (XRD) and Scanning Electron Microscopic (SEM) techniques are usually performed to measure and identify the new minerals and compounds formed due to the action of stabilization agents.

## 2.4 Applications of stabilization/solidifications (S/S) technology

The review of literature pertaining to application of S/S technology for treatment of oil-contaminated soils is as follows.

Petroleum Development Oman (PDO) carried out study on evaluation of effectiveness of S/S treatment of its petroleum-contaminated soil (PCS) using cement as stabilizer for the purpose of utilizing the treated PCS in engineering applications in beneficial and economical ways ( Hassan et al., 2004). PCS was subjected to the toxicity characteristic leaching procedure (TCLP). Unconfined compressive strength of the cement-stabilized PCS increased with the addition of 5% cement and remained relatively constant with the addition of higher cement content. The oil present in PCS had an adverse effect on cement hydration. In general, the blend of PCS with crushed stone, as a road base/sub-base, caused a reduction in CBR in comparison with 100% crushed stone. Blends of up to 10% PCS replacement can be used as a base material, while higher percentages of PCS substitution can be used for road sub-bases. Higher percentages of PCS replacement (up

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to 40% PCS) may be used for medium or light traffic surfaces or base course layers. For mixes containing 30% and 40 % PCS, air voids are higher than the typical specified limits (Hassan et al., 2004).

The results of a research aimed to investigate the effect of cement and cement by-pass dust (CBPD) as a stabilizer on the geotechnical properties of oil-contaminated soils resulting from leaking underground storage tanks, or soils surrounding petroleum refineries and crude oil wells indicate that cement and cement by-pass dust improve the properties of oil-contaminated soils. (Al-Rawas et al., 2005). It is found that, the addition of cement or CBPD resulted in an increase in strength as measured by the unconfined compression test. The strength is higher with a longer curing period. The presence of oil acts as a hydration retarder and reduces the strength. In general, the cohesion increases as the percentage of cement or CBPD increases and as the curing period increases. Higher cohesion values resulted from the use of cement as compared with CBPD. No specific trend was observed for the variation in the angle of internal friction with the addition of either cement or CBPD or with different curing periods. A higher percentage of cement and a longer curing period resulted in a decrease in permeability. The stabilization of oil-contaminated soils resulted in improved soil properties (Al-Rawas et al., 2005).

A research aimed to investigate the effects of hydrocarbon on engineering properties of residual soils developed from geranitic and metasedimentary rocks is reported (Rahma et al, 2010). The addition of hydrocarbon to soil was varied by 0, 4, 8, 12 and 16 % of dried weight of soil sample. From piratical size distribution analysis showed the granitic rock soil contains 64% sand 34% silt and 2% clay whereas the meta-sedimentary soil consists of 34% gravel, 37% sand and 2% clay. The specific gravity for granitic was 2.56 and for

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meta-sedimentary was 2.61. The types of minerals present in granitic soil were quartz, kaolinite and gibbsite whereas the meta-sedimentary soil consisted of quartz, kaolinite. It is found through this research that the Atterberg limits decrease as hydrocarbon amount increases. Also, the maximum dry density and optimum water content decrease as hydrocarbon amount increases. The maximum deviator stress for granitic soil ranged between 6-28 kPa and for meta-sedimentary soil ranged between 8-27 kPa. The overall unconsolidated un-drained shear decreased with an increase in the hydrocarbon amount (Rahma et al., 2010).

A study aimed to evaluate of the efficiency of remediation of a Botucatuis residual soil contaminated by diesel in terms of unconfined compressive strength (UCS) and column leaching (ASTM D4874) tests (Knope et al., 2005). The UCS and leaching tests on soil treated with up to 50% of Portland cement were carried out after 3 and 7 days of curing. It was noticed that the higher the amount contaminant added the lower was effectiveness of the cement addition to the contaminated soil.

A research on estimation of the potential of limestone dust (LSD) and coal fly ash (CFA) to stabilize contaminated soils was reported ( Brooks et al, 2011).The geotechnical characteristics of the soils investigated included: Atterberg limits, compaction, California bearing ratio (CBR), swell, and unconfined compressive strength (UCS) tests. Results of the study showed that the plasticity and swell of the soils were reduced by 40% and between 40 and 70%, respectively. The results further showed a marked increase in strength of the soils for CBR and UCS when stabilized with the additives (Brooks et al., 2011).

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Another study on S/S treatment of oil-contaminated soil using different stabilization agents like lime, fly ash and cement either independently or as an admixture showed an improvement in the geotechnical properties (Shah et al, 2002). This improvement can be attributed to dispersion of oil, cation exchange, agglomeration, and pozzolanic actions of additives namely lime, fly ash and cement (Shah et al, 2002). It is found that throughout the experimental program, the best results were observed when soil was treated with a combination of 10% lime, 5% cement and 5% fly ash. In the process of stabilization fuel oil might have formed a stable complex with metals. Increase in the strength of the soil can be attributed to neo-formation of compounds, like CSH, CSH-1, that coat and bridge soil grains (Shah et al., 2002).

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# CHAPTER 3

# EXPERIMENTAL INVESTIGATION

The experimental investigation was carried out mainly in three phases. Firstly, contaminated soils collected from three different locations having heavy, medium and low oil contamination levels were characterized and an optimum specimen size was selected by conducting compaction tests considering different alternatives. Secondly, different types of stabilizers and their different dosages were selected for S/S treatment and specimens for different tests were prepared. Finally, the UCS, CBR, TCLP, XRD/SEM tests were carried out on prepared and cured specimens for evaluating effectiveness of the S/S-treated soils. The results of the experimental work were presented in tabular and graphical forms for discussion and statistical analysis leading towards useful conclusions and recommendations.

## 3.1 Collection of Soil Samples

Samples of contaminated soils were collected from three storage sites belonging to a project contractor (located in the coastal area of Jubail, Saudi Arabia) having different levels of oil contamination. The oil-contaminated sediments were excavated from the intertidal zone of that area, Kazzami Peninsula, which is located between Mardumah and Al Freyah, as shown in Figure 3.1 .

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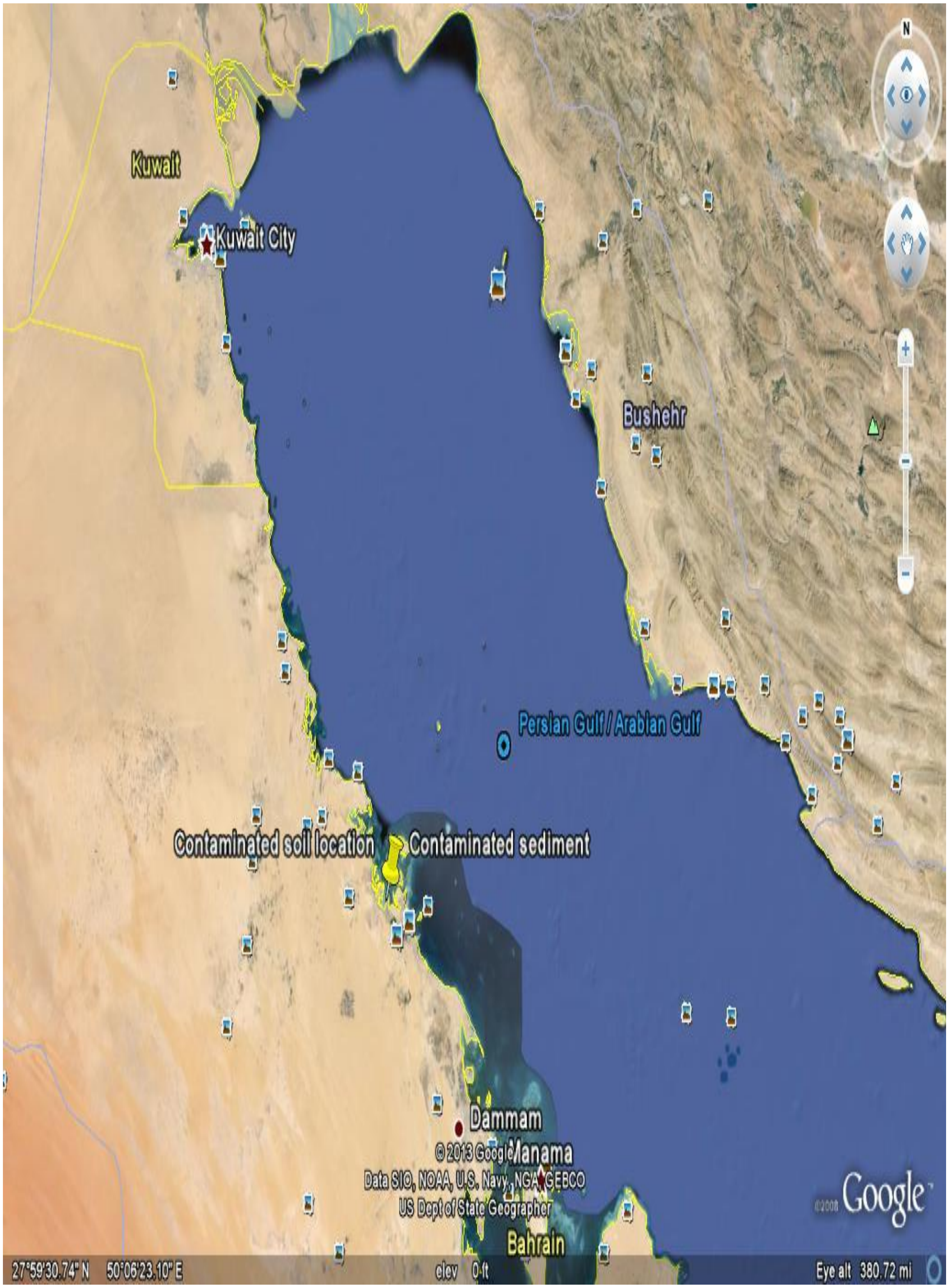


Figure 3.1: Satellite image showing locations from where oil-contaminated sediments were excavated and stored by the project contractor

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## 3.2 Characterization of Oil Contaminated Soils

The chemical and physical tests were conducted on the collected samples for characterization for contaminated soils.

### 3.2.1 Total Petroleum Hydrocarbons (TPH) Test

The term "total petroleum hydrocarbons" (TPH) is generally used to describe the measurable amount of petroleum-based hydrocarbons in the environment; and thus the TPH information obtained depends on the analytical method used. TPH tests were performed to determine the amount of oil contents of the contaminated soil samples using Agilent Technology, 7890 GC system, (Gas Chromatograph). Test results showing total TPH of contaminated soils are presented in Table 3.1 along with the relative classification of the contaminated soil based on the measured average total TPH, terming the soil with relatively highest TPH as "high oil contaminated soil (HOC)", having medium TPH as medium oil contaminated soil (MOC)" and having relatively lowest TPH as "low oil contaminated soil (LOC)".

Table 3.1: TPH results for contaminated soils.

| **Description (relative classification)** | **Total average TPH (mg/kg)** |
|---|---|
| High Oil Contaminated Soil (HOC) | 2682 |
| Medium Oil Contaminated Soil (MOC) | 2466 |
| Low Oil Contaminated Soil (LOC) | 1833 |

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### 3.2.2 X-Ray diffraction (XRD) Analysis

The mineralogical composition of the contaminated soils was investigated using the X-ray diffraction (XRD) technique. The contaminated soils were initially air dried, sieved using sieve #200 and thoroughly mixed for homogenization. About 10 grams of each soil sample was utilized for the mineralogical analysis. X-ray diffractometer (RIGAKU ULTIMA IV X-RAY DIFFRACTOMETER) was used.

Figures 3.2 through 3.4 show the XRD patterns for HOC, MOC and LOC soils, respectively. Each pattern consists of a series of reflections of different intensities (count per second, cps) at different values of 2θ. Table 3.2 shows the percentages of different minerals present in the contaminated soils, taken from Figures 3.2 through 3.4.

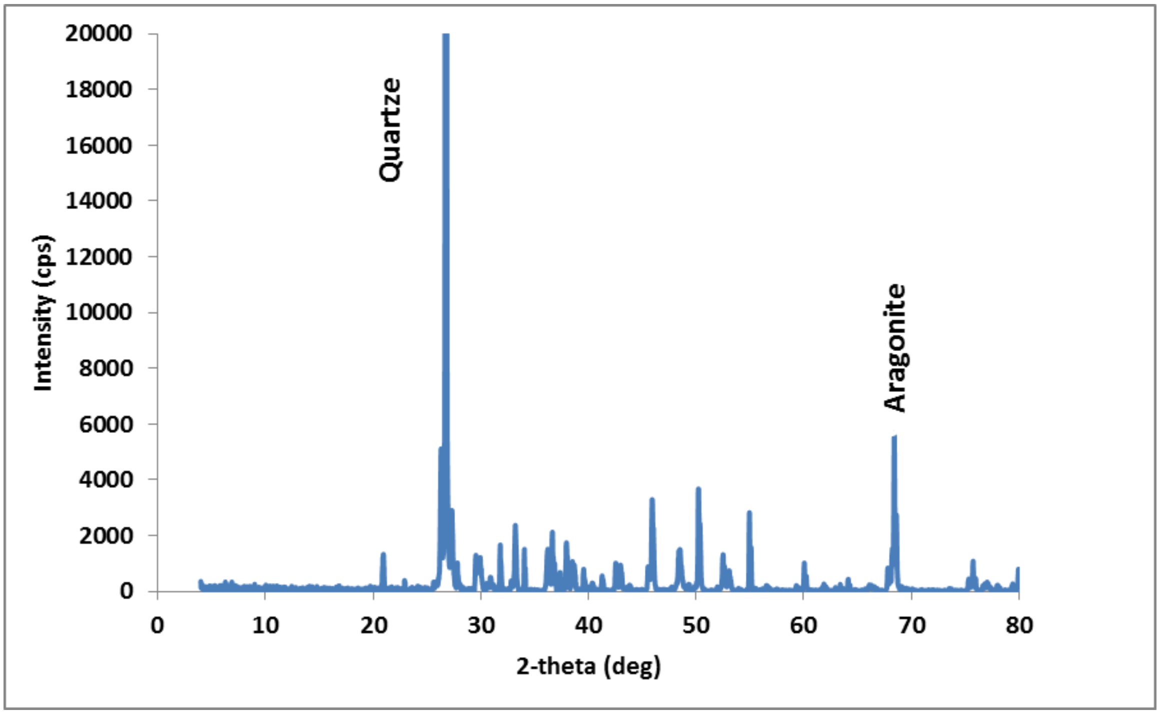


Figure 3.2: XRD Micrographs for HOC soil

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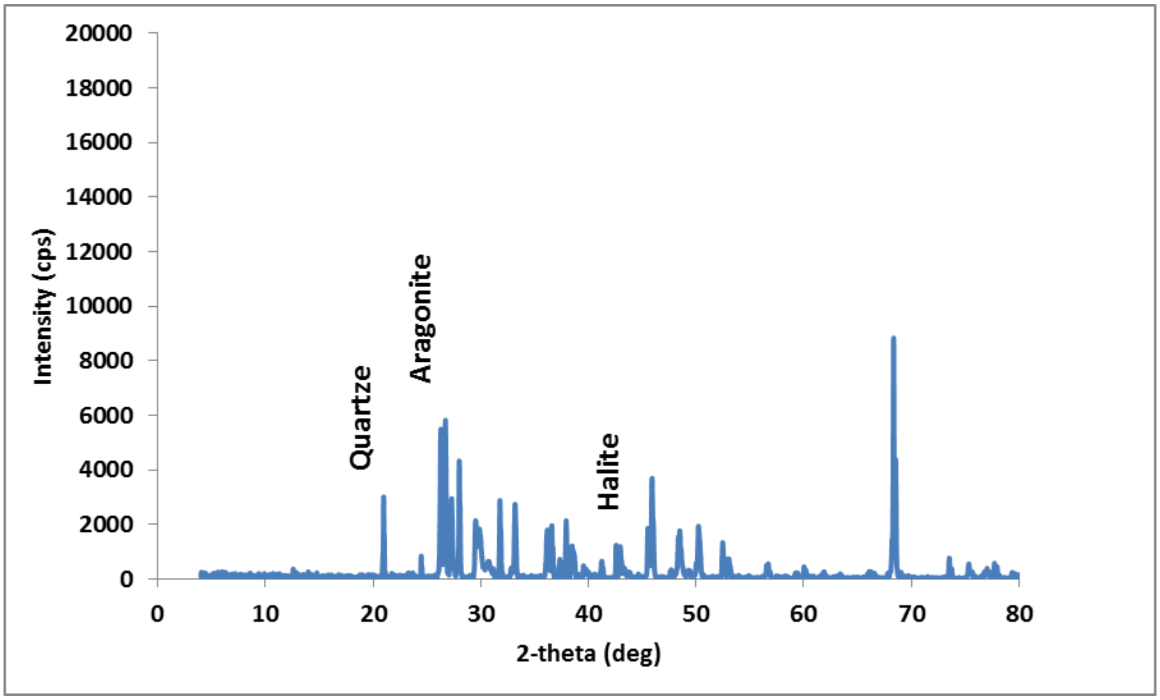


Figure 3.3: XRD Micrographs for MOC soil

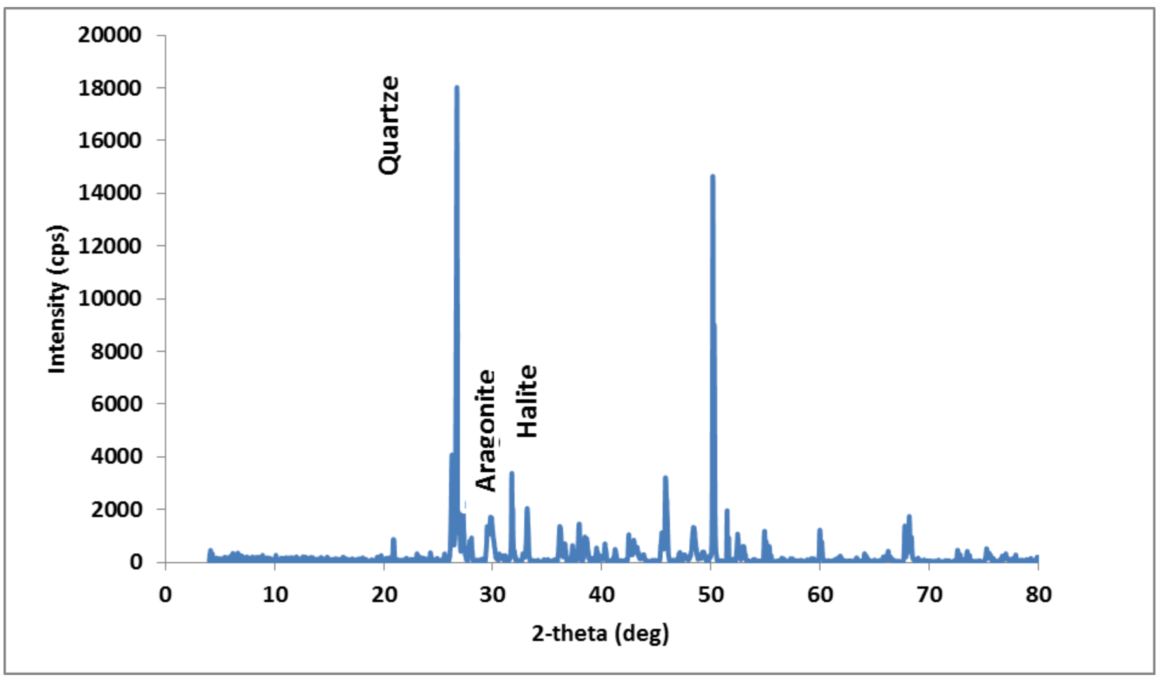


Figure 3.4: XRD Micrographs for LOC soil

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Table 3.2: Mineralogical Analysis of Selected Contaminated Soil

| Compound | HOC (% by mass of soil) | MOC (% by mass of soil) | LOC (% by mass of soil) |
|---|---|---|---|
| Aragonite [$CaCO_3$] | 26 | 20 | 24 |
| Quartz [$SiO_2$] | 74 | 74.3 | 69 |
| Halite [NaCl] | 0 | 5.7 | 7 |

It can be observed from Table 3.2 that the contaminated soil has quartz as its main constituent. About three-fourth of the soil is constituted by quartz and one-fourth is constituted by aragonite, i.e., calcium carbonate. A small percentage of halite, i.e., sodium chloride is also present in the contaminated soil.

### 3.2.3 Scanning Electron Microscopy (SEM) Analysis

The scanning electron microscope (SEM) is an ideal tool to observe the features of the fabric of soil. For carrying out SEM analysis, the contaminated soils were initially air-dried, sieved using sieve #200 and thoroughly mixed for homogenization. About 10 grams of each soil sample was used to conduct SEM analysis with the help of JEOL 500LV scanning electron microscope (SEM). Figures 3.5 through 3.7 show the micrographs of HOC, MOC and LOC soils, respectively.

From the SEM micrographs, as shown in Figures 3.5 through 3.7, it is found that the HOC soil has most dense microstructure and the LOC soil has least dense microstructure. The reason behind differences in the porosity of the same soil can be attributed to the presence of oil particles in the pores of contaminated soils.

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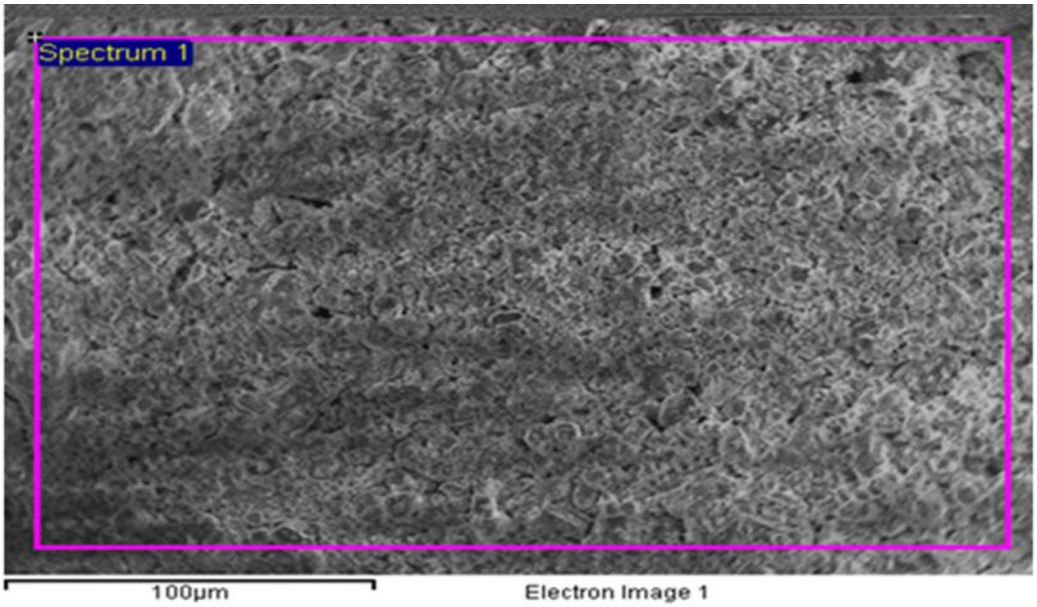


Figure 3.5: SEM Micrographs for HOC soil

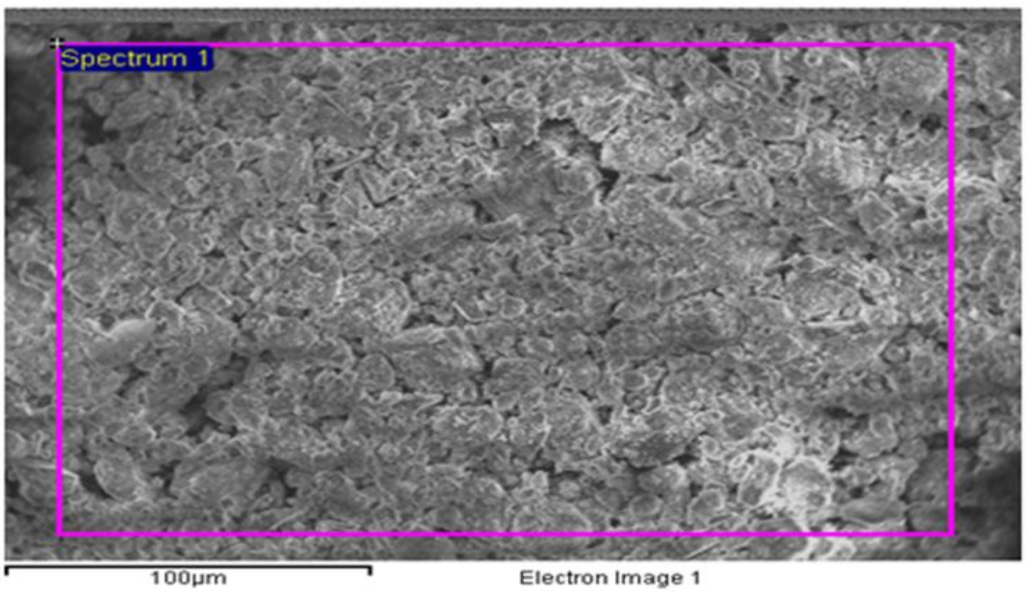


Figure 3.6: SEM Micrographs for MOC soil

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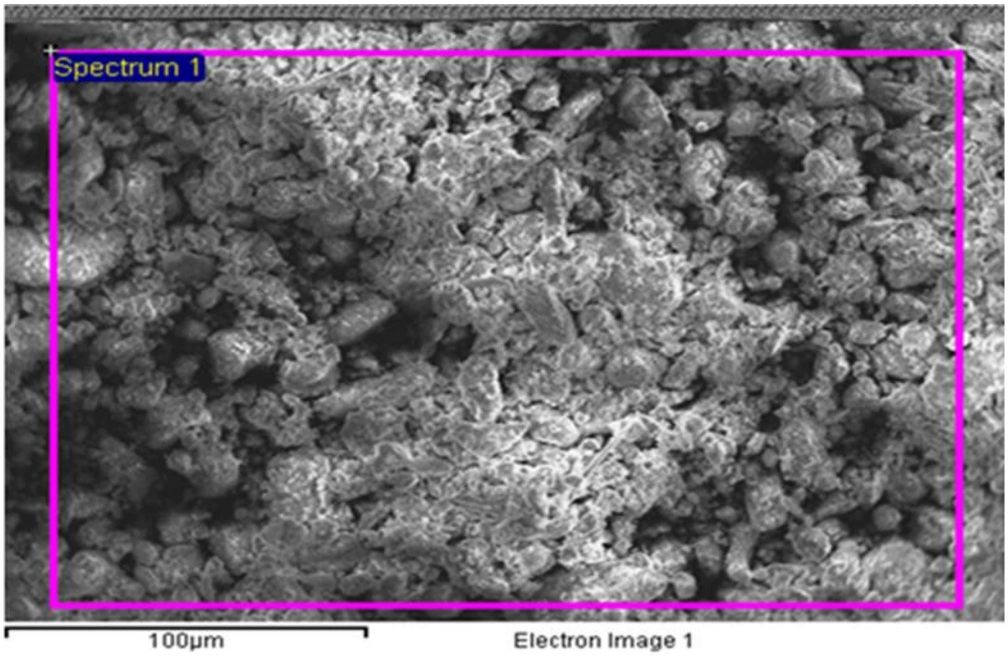


Figure 3.7: SEM Micrographs for LOC soil

### 3.2.4 Specific Gravity

The specific gravity is used as a parameter to determine some important properties of the soil such as void ratio, unit weight, soil particle size and determination of the saturation of the soil during the consolidation process. The specific gravity of the soil samples, passing sieve No. 4, were determined in accordance with ASTM D 854. Specific gravities of high, medium and low contaminated samples were found to be 2.64, 2.62 and 2.65, respectively.

### 3.2.5 Atterberg Limits

For determining liquid limit and plastic limit, the Atterberg limit test was conducted on the three contaminated soil samples, passing through ASTM sieve No. 40, according to ASTM D4318. Since the soil is a cohesion less mainly consisting quartz, it was not possible to get the number of blows for the liquid limit test, so the liquid limit is reported to be nil. Also, the soil samples could not be rolled to a thread of 1/8 in (3.18 mm), therefore, the soil was classified as non-plastic.

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### 3.2.6 Grain Size Distribution

For conducting sieve analysis test as per the ASTM D 422, high, medium and low contaminated soil samples were first sieved throughout #4 sieve (4.75 mm in diameter) and mixed properly. Then, the samples were dried in the oven at 110 °C. It was observed that all contaminated soil samples contains some particles passing through sieve No. 200, therefore, the hydrometer test was also conducted to check the presence of silt in soil. The results of the sieve analysis (dry and wet) and hydrometer tests were plotted as shown in Figures 3.8 through 3.11 for HOC, MOC and LOC soils. It can be observed that about 18.5%, 27%, and 28.5% of HOC, MOC and LOC soils, respectively, passes throughout the #200 sieve when soils were in wet state. However, when the contaminated soil samples were sieved in dry state, the percentages passing through # 200 sieve were recorded as 1.5%, 3% and 2.7% for HOC, MOC and LOC soils, respectively.

Figures 3.8through 3.11 indicate that the grain size curve, obtained using the results of wet sieving, was consistently above the one when dry sieving method was used. This is attributed to the fact that water tends to dissolve the salt particles of the soil, increasing the percentage of fine particles.

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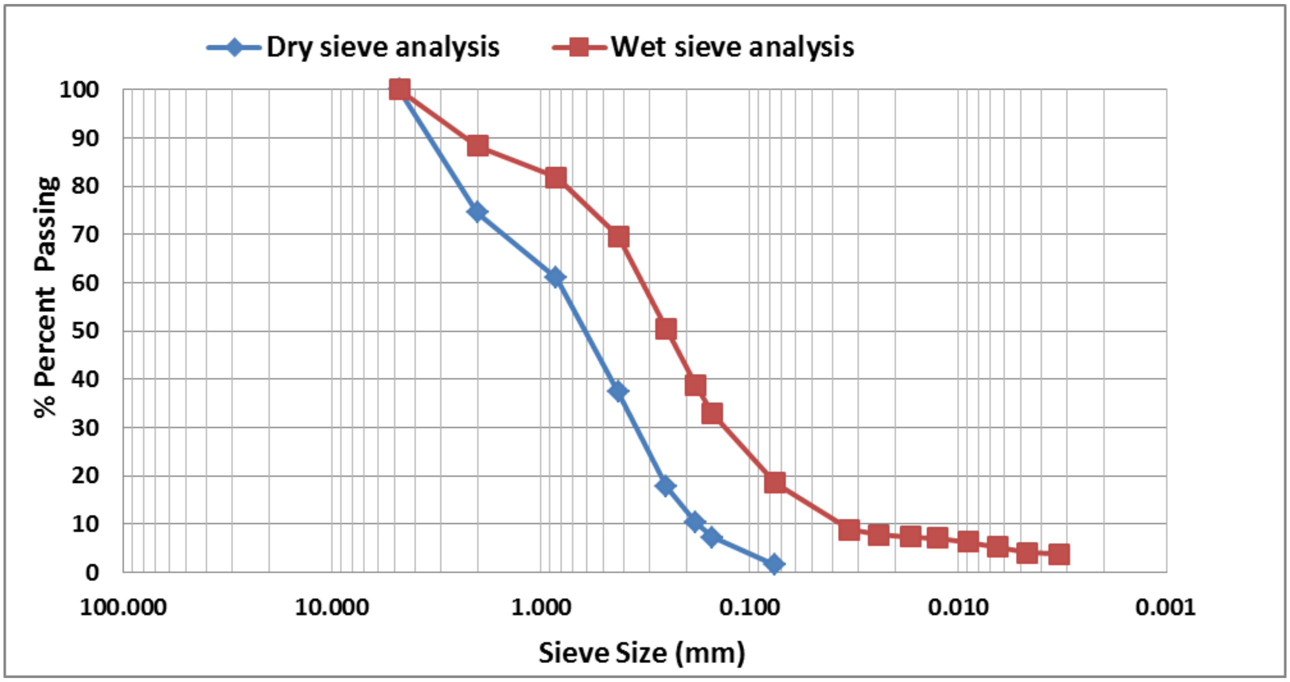


Figure 3.8: Grain size distribution curve for HOC

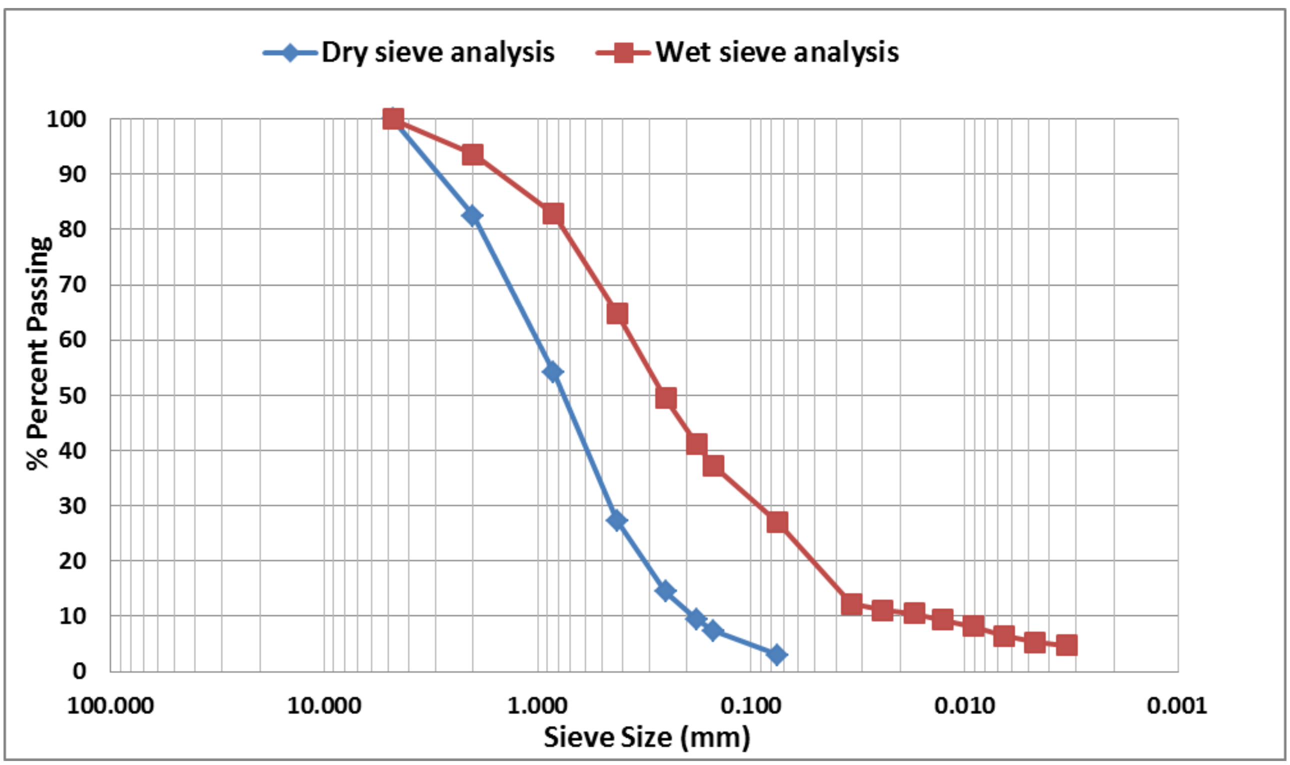


Figure 3.9: Grain size distribution curve for MOC

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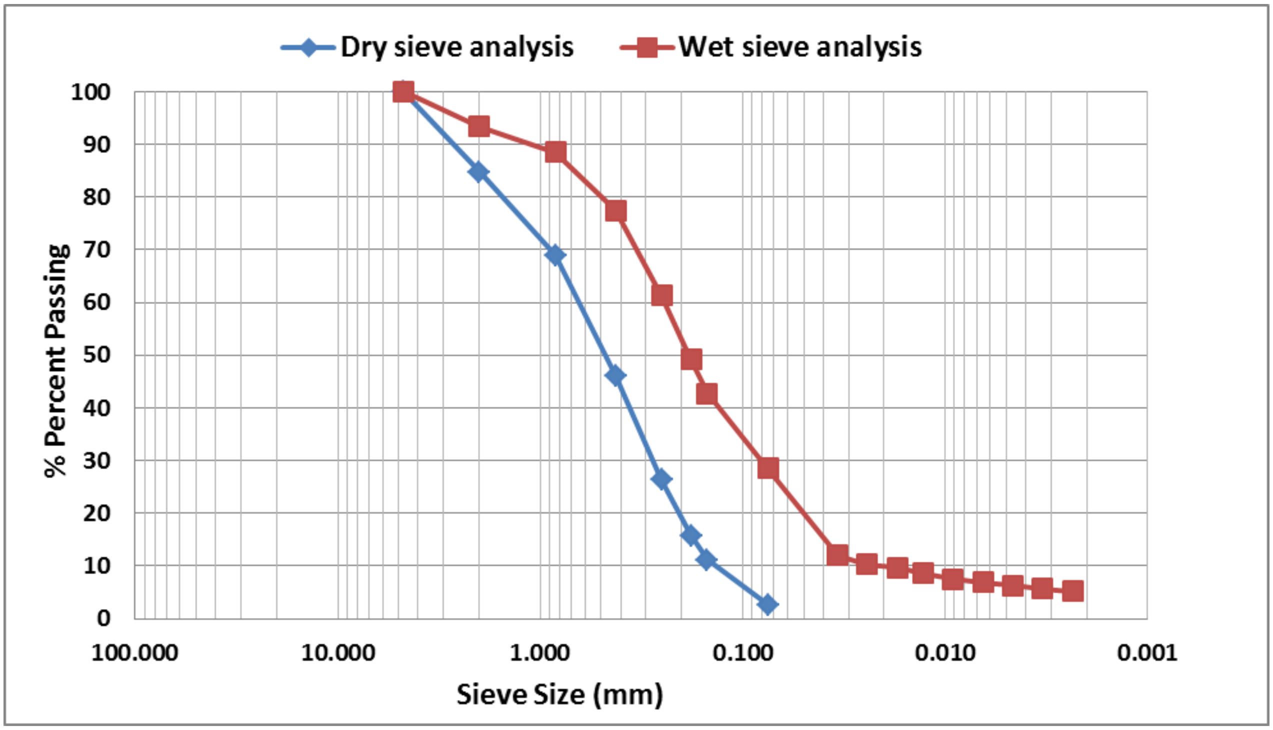


Figure 3.10: Grain size distribution curve for LOC

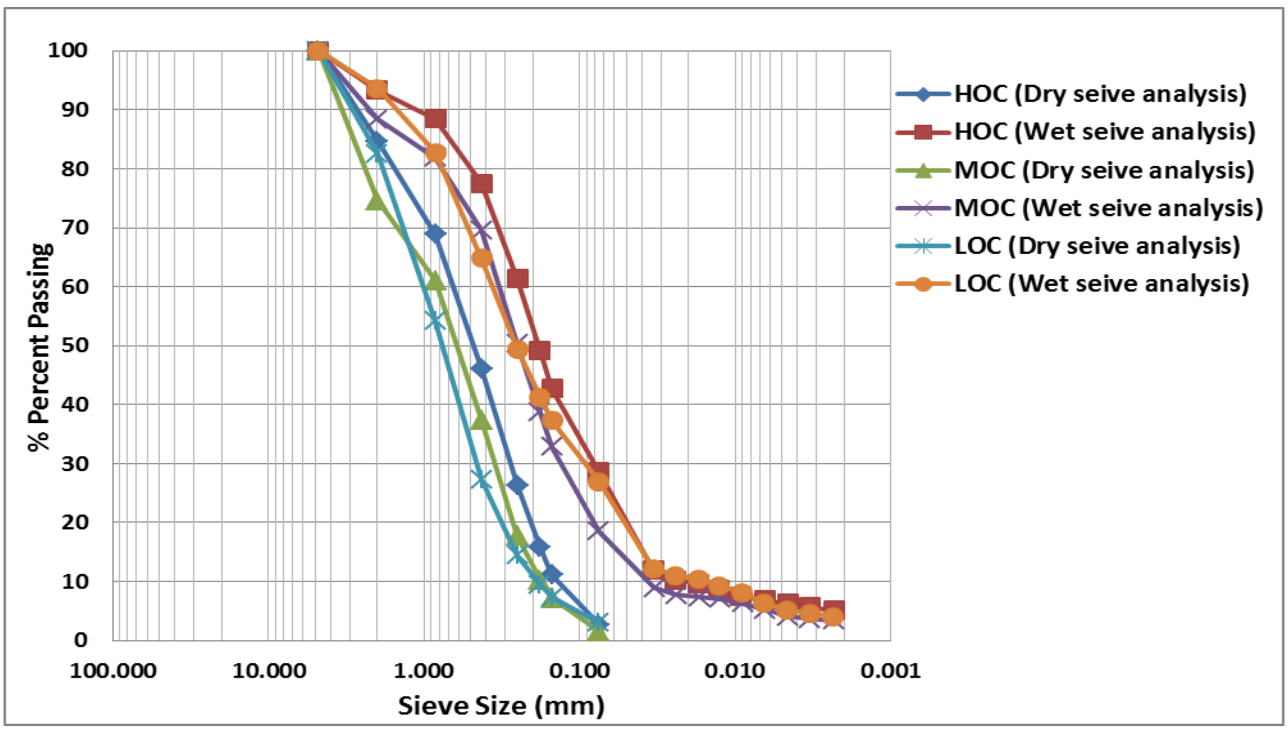


Figure 3.11: Grain size distribution curve for all Oil Contaminated

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### 3.2.7 Classification of the Selected Soils

Based on the grain size distribution analysis, the soil can be classified as “silty sand (SM)” according to the USCS classification criteria and the same can be classified as “A-3” (based on washed sieving) according to the AASHTO classification criteria, as shown in Table 3.3.

Table 3.3: Soil Characteristics

| **Property** | **Designation** | **HOC** | **MOC** | **LOC** |
|---|---|---|---|---|
| Specific Gravity | ASTM D 854 | 2.64 | 2.62 | 2.65 |
| Liquid Limit | ASTM D 4318 | Non Plastic | Non Plastic | Non Plastic |
| Plastic Limit | ASTM D 4318 | Non Plastic | Non Plastic | Non Plastic |
| Classification | USCS | SM (silty sand) | SM (silty sand) | SM (silty sand) |
| | AASHTO | A-3 | A-3 | A-3 |

## 3.3 Selection of Optimum Mold Size using Compaction Test

In order to reduce the consumption of materials, time and energy in conducting the UCS tests an optimum size of the test specimen was selected by conducting trial Proctor compaction tests. Two specimen sizes were considered (as shown in Figure 3.12): (i) cylindrical mold having 4 inch diameter and 6 inch height as normally used for standard and modified Proctor tests and (ii) miniature cylindrical mold having 1½ inch diameter and 3 inch height for reducing the effort without compromising with the compaction energy and keeping the height to diameter ratio within the range of 2 to 2.5 as recommended for UCS test. The weights of hammers for compacting in cases of standard, modified, and miniature tests were taken as 5.5, 10, and 1.32 lb, respectively. The numbers of layers considered for compacting in cases of standard and

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miniature tests were 3 whereas 5 layers were used in case of modified Proctor test. While standard 25 number of blows was kept for standard and modified Proctor tests, the number of blows for miniature size was varied from 25 to 45.

Figure 3.12: Standard and miniature molds size and hammers

From the observation of the moisture-density relationship curves, as shown in Figure 3.13, the maximum dry unit weights of modified specimen size, standard specimen size and selected miniature specimen size with different number of blows were found as: modified-18.2 kN/m$^3$, standard -16.8 kN/m$^3$, miniature 16.3 kN/m$^3$ (45 blows), 16.3 kN/m$^3$ (35 blows), 16.4 kN/m$^3$ (30 blows) and 16.5 kN/m$^3$ (25 blows) corresponding to optimum moisture contents of 8.1% , 10.5%, 12% , 12%, 11.5% and 11.5%, respectively. The summary of all the compaction tests carried out for selecting optimum mold size is presented in Table 3.4.

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Based on the results presented in Table 3.4, the miniature specimen with 25 number of blows was selected as an optimum mold with optimum degree of compaction because of the following reasons:

i. The maximum dry density and compaction energy of the miniature specimen with 25 number of blows (16.5 kN/m$^3$ and 12,368 ft-lb/ft$^3$, respectively) were almost similar to that for the standard specimen (16.8 kN/m$^3$ and 12,375 ft-lb/ft$^3$, respectively).

ii. As observed from Figures 3.14 and 3.15, for miniature size specimen there was no significant increase in the maximum dry density and compaction energy with increase in the number of blows beyond 25.

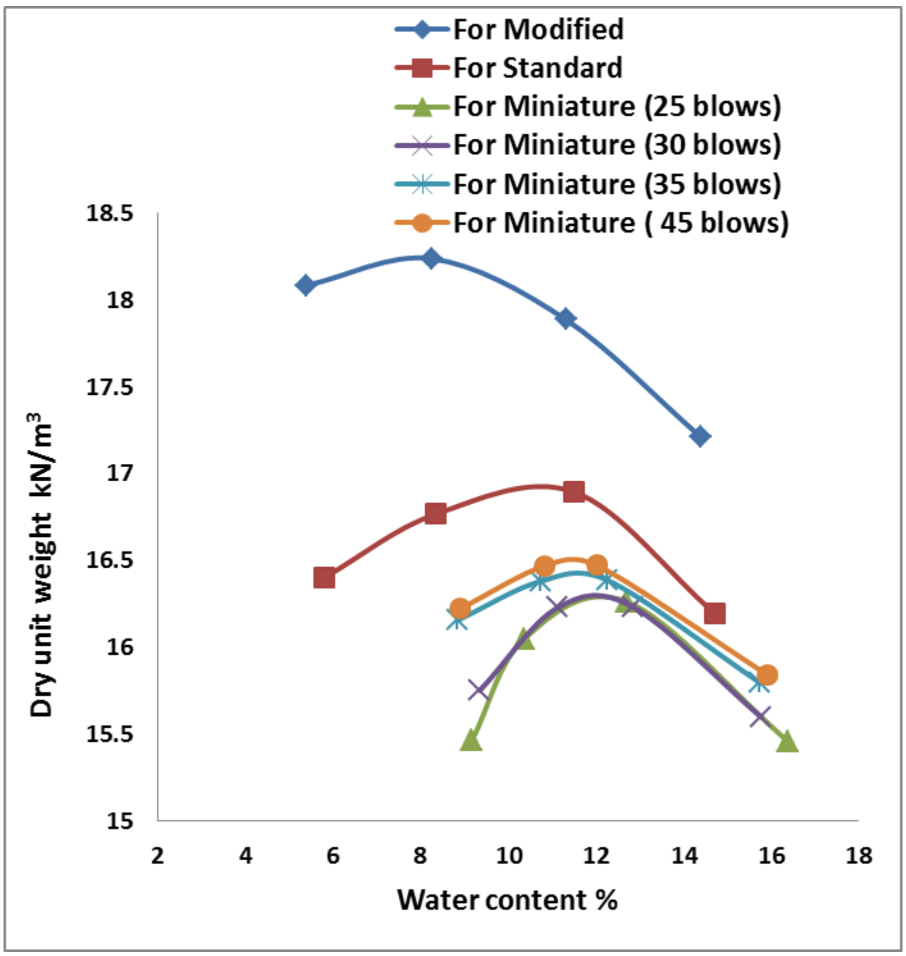


Figure 3.13: Compaction curves for modified, standard and selected size specimens

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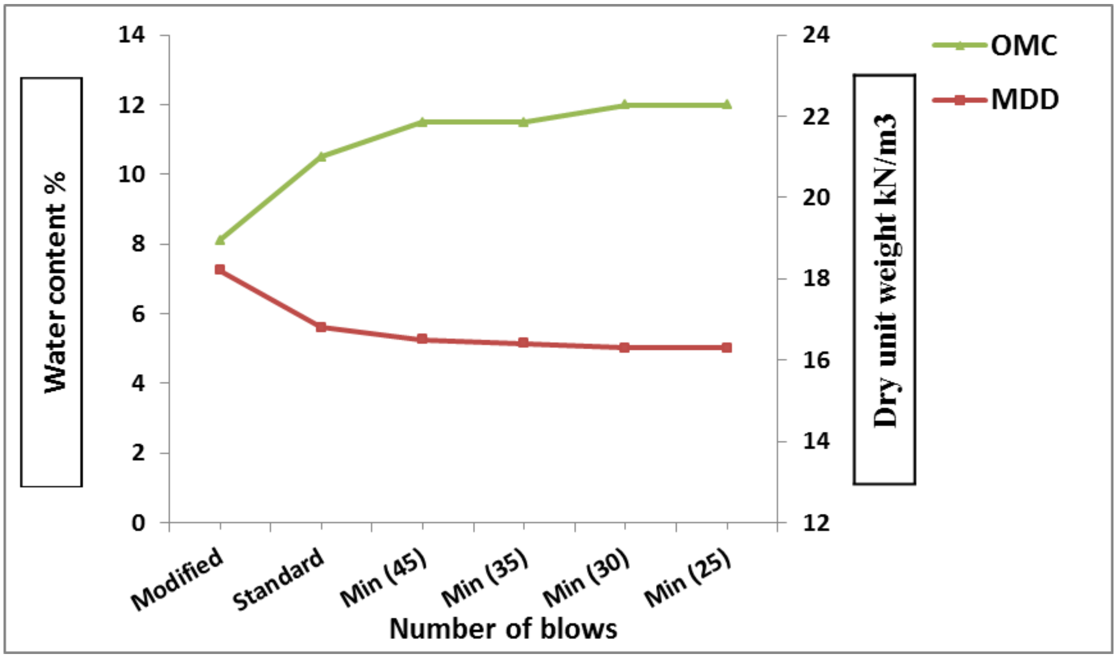


Figure 3.14: Moisture-density relationship for modified, standard and selected size specimens

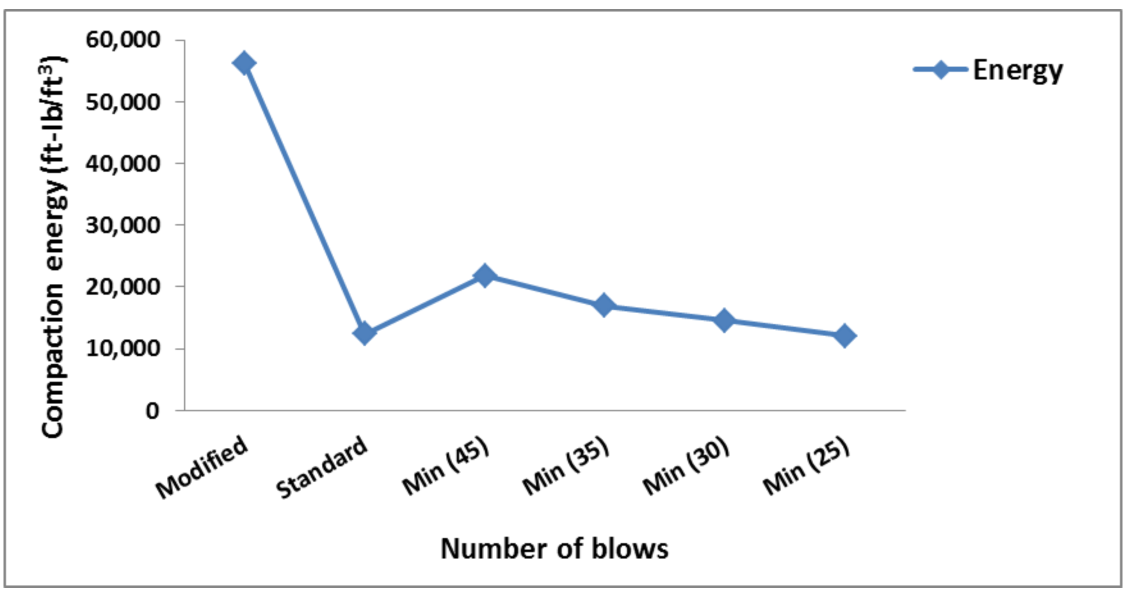


Figure 3.15: Compaction energy curve for modified, standard and selected size specimens

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Table 3.4: Summary of compaction tests

| **Compaction test parameters** | **Modified size (4 in × 6 in)** | **Standard size (4 in × 6 in)** | **Miniature size (1.5 in × 3 in)** | | | |
|---|---|---|---|---|---|---|
| | **Modified** | **Standard** | **Miniature (45)** | **Miniature (35)** | **Miniature (30)** | **Miniature (25)** |
| **Number of layers** | 5 | 3 | 3 | | | |
| **Weight of hammer (lb)** | 10 | 5.5 | 1.32 | | | |
| **Height of drop of hammer (ft)** | 1.5 | 1 | 0.383 | | | |
| **Number of blows** | 25 | 25 | 45 | 35 | 30 | 25 |
| **Volume ($ft^3$)** | 0.0333 | 0.0333 | 0.0031 | | | |
| **Compaction energy( ft-lb/$ft^3$)** | 56,250 | 12,375 | 22,263 | 17,315 | 14,842 | 12,368 |
| **Energy Compared to Modified** | 4.6 | 1 | 1.8 | 1.4 | 1.2 | 1 |
| **Optimum moisture content (%)** | 8.1 | 10.5 | 11.5 | 11.5 | 12 | 12 |
| **Maximum dry density( $kN/m^3$)** | 18.2 | 16.8 | 16.5 | 16.4 | 16.3 | 16.3 |

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## 3.4 Selection of Stabilizers

Following four types of stabilizers were used for S/S treatment in the present study:

i. Portland cement (Type I conforming to ASTM C 150)

ii. CKD was obtained from Saudi Arabian Cement Company, as a waste material.

iii. EAFD generated as waste from Saudi Iron and Steel Company (HADEED).

iv. LSP obtained from the crushing of carbonate rocks at a quarry in Abu Hadriyah, Eastern Province, Saudi Arabia.

The chemical composition of cement, CKD, EAFD and LSP are presented in Tables 3.5 through 3.8, respectively. The specific gravities of cement, CKD, EAFD and LSP were found to be 3.15, 2.78, 2.75 and 2.66, respectively.

Table 3.5: Chemical Composition of Portland Type I Cement (Najamuddin, 2011)

| **Constituent** | **Weight (%)** | **Constituent** | **Weight (%)** |
|---|---|---|---|
| CaO | 64.35 | $SO_3$ | 2.10 |
| $SiO_2$ | 22.0 | Loss on ignition | 0.7 |
| $Al_2O_3$ | 5.64 | $C_3S$ | 55 |
| $Fe_2O_3$ | 3.80 | $C_2S$ | 19 |
| $K_2O$ | 0.36 | $C_3A$ | 10 |
| MgO | 2.11 | $C_4AF$ | 7 |
| $Na_2O$ | 0.19 | | |
| Equivalent alkalis ($Na_2O$ + 0.658$K_2O$) | 0.33 | | |

`

Table 3.6: Chemical Composition of CKD (Najamuddin, 2011)

| Constituent | Weight (%) | Constituent | Weight (%) |
|---|---|---|---|
| CaO | 49.3 | Chloride | 6.90 |
| $SiO_2$ | 17.1 | Loss on ignition | 15.8 |
| $Al_2O_3$ | 4.24 | BaO (µg/g (ppm)) | 78.2 |
| $Fe_2O_3$ | 2.89 | $Cr_2O_3$ | 0.011 |
| $K_2O$ | 2.18 | CuO | 0.029 |
| MgO | 1.14 | NiO | 0.012 |
| $Na_2O$ | 3.84 | $TiO_2$ | 0.34 |
| $P_2O_5$ | 0.12 | $V_2O_5$ | 0.013 |
| Equivalent alkalis ($Na_2O$ + 0.658$K_2O$) | 5.27 | ZnO (µg/g (ppm)) | 65.8 |
| $SO_3$ | 3.56 | $ZrO_2$ | 0.011 |

Table 3.7: Chemical Composition of EAFD (Najamuddin, 2011)

| Constituent | Weight (%) |
|---|---|
| Aluminium | 0.7 |
| Calcium | 9.39 |
| Cadmium | 0.0004 |
| Copper | 0.06 |
| Iron | 33.6 |
| Potassium | 1.70 |
| Magnesium | 2.3 |
| Manganese | 1.8 |
| Sodium | 2.6 |
| Nickel | 0.01 |
| Lead | 1.31 |
| Phosphorous | 0.13 |
| Silicon | 2.38 |
| Tin | 0.03 |
| Sulphur | 0.57 |
| Titanium | 0.09 |
| Zinc | 10 |

`

Table 3.8: Chemical Composition of LSP (Najamuddin, 2011)

| **Constituent** | **Weight (%)** |
|---|---|
| $SiO_2$ | 11.79 |
| CaO | 45.7 |
| $Al_2O_3$ | 2.17 |
| $Fe_2O_3$ | 0.68 |
| MgO | 1.80 |
| $K_2O$ | 0.84 |
| $Na_2O$ | 1.72 |
| Equivalent alkalis ($Na_2O$+0.658$K_2O$) | 2.27 |
| Loss on Ignition | 35.10 |
| Moisture | 0.20 |

Figure 3.16 shows the comparison of the percentages of four major oxides (CaO, $SiO_2$, $Al_2O_3$ and $Fe_2O_3$) present in the selected stabilizers. It can be observed from Figure 3.16 that while the percentages of the major oxides in CKD and LSP are comparable with that of cement, the EAFD has very small percentages of CaO, $SiO_2$, and $Al_2O_3$. However, the percentage of $Fe_2O_3$ in EAFD is very high (48.1%), more than 12 times as compared to cement. This is because EAFD is obtained from iron industries. Since EAFD possessed insignificant cementitious constituents, its major effect on S/S treatment is not expected. However, it was considered to explore the possibility of its action when combined with the other stabilizer.

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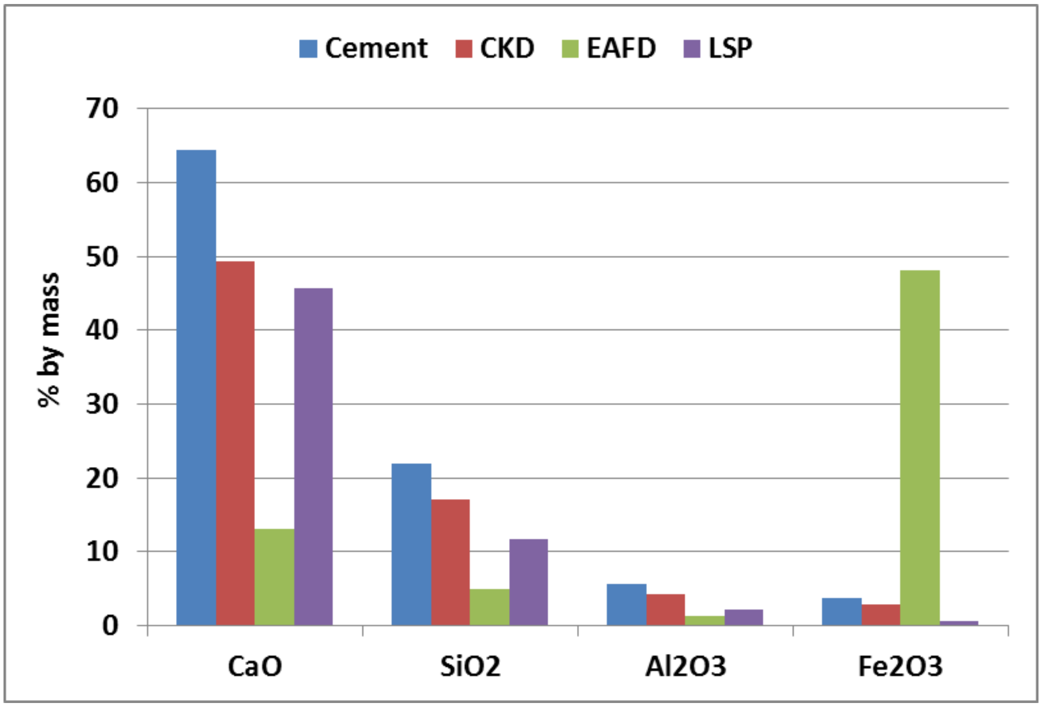


Figure 3.16: Comparison of the percentages of four major oxides ($CaO$, $SiO_2$, $Al_2O_3$ and $Fe_2O_3$)

## 3.5 Selection of the Combinations of stabilizers for S/S Treatment

Based on the findings of the studies conducted on stabilization of local plain soils using cement, CKD, EAFD and LSP (AL-Homidy, 2013), various combinations of cement, CKD, EAFD and LSP in different ranges of their dosages were selected for S/S treatment of the oil-contaminated soils considered in the present work. As shown in Table 3.9, 23 combinations of stabilizers were first considered for HOC soil and the performance of the S/S treatment was evaluated. Three combinations of stabilizers, which were found to be successful for treating HOC soil with possibility of utilizing the treated HOC in road construction, were selected for treatment of MOC and LOC soils, as given in Table 3.10.

`

Table 3.9: Trial mixtures of HOC soil for S/S treatment

| Mix No. | Stabilizer |
|---|---|
| 1 | Plain (untreated) |
| 2 | 2% Cement |
| 3 | 5% Cement |
| 4 | 7% Cement |
| 5 | 10% Cement |
| 6 | 30% CKD |
| 7 | 30% CKD + 2% Cement |
| 8 | 30% CKD + 5% Cement |
| 9 | 30% CKD + 7% Cement |
| 10 | 30% CKD + 10%LSP |
| 11 | 20% EAFD |
| 12 | 20% EAFD + 2% Cement |
| 13 | 20% EAFD + 5% Cement |
| 14 | 20% EAFD + 7% Cement |
| 15 | 20% EAFD + 10% LSP |
| 16 | 5% LSP |
| 17 | 10% LSP |
| 18 | 15% LSP |
| 19 | 5% LSP + 2% Cement |
| 20 | 10% LSP + 2% Cement |
| 21 | 15% LSP + 2% Cement |
| 22 | 15% LSP + 5% Cement |
| 23 | 15% LSP + 7% Cement |

Table 3.10: Trial mixtures of MOC and LOC soil for S/S treatment

| Mix No. | Stabilizer |
|---|---|
| 1 and 2 | Plain (0% Stabilizer) |
| 3 and 4 | 7% Cement |
| 5 and 6 | 30% CKD + 5% Cement |
| 7 and 8 | 15% LSP + 7% Cement |

`

## 3.6 Procedure for Mixing Soil with Stabilizer

The mixer as shown in Figure 3.17 was used for mixing the soil with stabilizer for preparing specimens for compaction tests and all other tests for evaluating the effectiveness of S/S treatment such as UCS, CBR, TCLP, etc. Mixing was carried out in a dry state for about first 3 minutes to achieve homogeneity, then water, as required to maintain the optimum moisture content (OMC), was added to the dry mixture and mixing was continued for about another 10 minutes.

Figure 3.17: Photograph showing mixer used

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## 3.7 Compaction Test for Determining Required OMCs

Before preparation of the test specimens, trial compaction tests were carried out on all the proposed mixtures to determine the OMC individually for each of the 31 mixtures listed in Tables 3.9 and 3.10, based on maximum dry densities. The compaction tests were conducted using the optimally selected miniature mold of size 1.5 in (38.1mm) × 3 in (76.2 mm) and using compaction in 3 layers with 25 number of blows per layer.

## 3.8 Unconfined Compressive Strength (UCS) Test

UCS test is commonly used for the evaluation of the performance of S/S-treated soils. The UCS is used in the structural design of pavements as the strength criterion for the base and the sub-base courses. Usually, a minimum UCS value is specified by codes of practice for different courses of a pavement.

In the present study, UCS tests were carried out according to ASTM D 2166. Specimens were prepared using the selected miniature molds having height/diameter ratio of 2. The soil and stabilizer for individual trial mixtures were mixed first in dry state and then mixed in presence of water (using OMC) as mentioned in section 3.6. Thereafter, the mix was compacted in the selected miniature mold in 3 layers, each layer given a 25 number of blows. The mold used had a split to make sure that the samples extruded as perfect as possible. After the removal of the specimens, they were wrapped in three layers of nylon to prevent any loss of moisture from the specimens, as shown in Figure 3.18. The samples were then kept on the table in the laboratory for air-curing for different periods (7, 28 and 90 days) at the laboratory temperature (23 ± 3$^{o}$C). Then each specimen was

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subjected to unconfined loading till the failure, as shown in Figure 3.19. The deformation rate of the test was 0.95 mm/min. The test was carried out using the compression machine 300 kN. For each mixture, three replicate specimens were tested and the average UCS value was considered.

Figure 3.18: Some of UCS Sealed Specimens throughout the Curing Period

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Figure 3.19: Photo showing the UCS testing

The criteria for minimum 7-day UCS of stabilized soils required for their utility in sub-base and sub-grade layers in rigid and flexible pavements construction, as recommended by ACI Committee (1990), are presented in Table 3.11.

Table 3.11: Minimum UCS for Stabilized Soils (ACI Committee, 1990)

| **Stabilized Layer** | **Minimum required UCS after 7-days of sealed curing, kPa** | |
|---|---|---|
| | **Concrete Pavement** | **Flexible Pavement** |
| Base Course | 3450 | 5175 |
| Sub-base Course | 1380 | 1725 |

`

## 3.9 Soaked CBR Test

California Bearing Ratio (CBR) of the materials used in base and sub-base courses of pavements has been commonly used in the structural design and evaluation of pavements. The test is recognized worldwide because of its simplicity and applicability. Therefore, the test can easily be used to evaluate the material for use in pavement construction and adapted by the engineers as a test to empirically measure the strength of soil under controlled moisture and density conditions.

In this study, soaked CBR tests were conducted according to ASTM D 1883 to simulate the field conditions in which the soil is flooded with water where flooding can either be from the ground water or from the rain water infiltrating the layers. Like UCS testing, all the trial mixtures were subjected to soaked CBR tests for assessing the adequacy of the CBR value of each mixture for the utilization of the treated mixtures in pavement construction. The CBR mold had a height of 5 in (127 mm) and a diameter of 6 in (152 mm). After casting the prepared mixtures in the CBR mold, the CBR molds were sealed by plastic sheets and left to air-curing in laboratory conditions (23 ± 3 $^{o}$C) for 7 days, as shown in Figure 3.20. Then, the samples still in molds were soaked in water by placing the unsealed molds in a water tank along with the dial gage for 4 days as shown in Figure 3.21. No swelling was observed during soaking. After completion of soaking period, the soaked specimens were subjected to CBR testing according to ASTM D 1883, as shown in Figure 3.22.

Figure 3.20: Sealed specimens subjected to air-curing for 7 days

Figure 3.21: Specimens soaked in water for 4 days after air-curing

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Figure 3.22: Photo showing the Soaked CBR testing

The criteria for minimum CBR requirements for road construction, as recommended by Asphalt Institute (1970) and presented in Table 3.12, were used for exploring the possibility of utilization of S/S-treated soils in the road construction

Table 3.12: Minimum CBR Requirements for Road Construction (Asphalt Institute, 1970):

| **CBR, %** | **General rating** | **Uses** |
|---|---|---|
| 0-3 | Very poor | Sub-grade |
| 3-7 | Poor to fair | Sub-grade |
| 7-20 | Fair | Sub-base |
| 20-50 | Good | Base or Sub-base |
| > 50 | Excellent | Base |

`

## 3.10 Toxicity Characteristic Leaching Procedure Test (TCLP)

The toxicity characteristics leaching procedure (TCLP) method evaluates mobility of metals in a landfill (LaGrega et al, 2001). The test simulates worst case scenario where hazardous waste is co-disposed with municipal waste. The test was carried out according to the EPA Method 1311(US EPA, 1998). The TCLP was performed on specimens of untreated and treated soils that satisfied the minimum requirements of unconfined compressive strength and CBR. The specimens were prepared using the same condition of mixing and compaction and then subjected to air-curing for 7 days. After curing, the specimens were crushed and passed through a standard sieve of 9.5 mm size (i.e., ASTM sieve 3/8 inch). Then, the specimens were stored in plastic bags for the extraction of metals (US EPA Method 1311). The pH was measured and it was found greater than 5; extraction fluid with pH 2.88 ± 0.05 was used. The TCLP extraction of specimens was carried out using the rotary extractor device, as shown in Figure 3.23. The photograph showing extracted samples is depicted in Figure 3.24. The concentrations of the metals that leached out from the stabilized soil specimens through simulated extraction process were measured and compared with the maximum permissible concentrations of metals set by the EPA. Table 3.13 shows the maximum permissible concentration of contaminants, as per the EPA standards (EPA, 1998).

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Figure 3.23: Rotary extractor device


Figure 3.24: Extracted samples for TCLP tests

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Table 3.13: The maximum permissible concentration of contaminants (EPA, 1998)

| Contaminant | Regulatory Level (mg/l) |
|---|---|
| Arsenic (As) | 5 |
| Barium(Ba) | 100 |
| Cadmium(Cd) | 1 |
| Chromium(Cr) | 5 |
| Lead(Pb) | 5 |
| Mercury(Hg) | 0.2 |
| Selenium(Se) | 1 |
| Silver(Ag) | 5 |
| Nickel(Ni) | Not Regulated by EPA |
| Vanadium(V) | Not Regulated by EPA |

## 3.11 X-Ray Diffraction/Scanning Electron Microscopic (XRD/SEM)

In this study, JEOL 500LV scanning electron microscope (SEM) utilizing the secondary electron mode and X-ray diffraction (XRD) were used for testing the mixtures that satisfied the requirements of minimum UCS and CBR and maximum TCLP. Cylindrical specimens (38.1 mm in diameter and 76.2 mm in height) were cast, and cured in the same way as for the UCS, CBR and TCLP tests. Around 20 mm cubical specimens were cut out from the prepared cylindrical specimens for conducting the XRD/SEM tests.

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# CHAPTER 4

# RESULTS AND DISCUSSION

In this chapter, the results of all the tests conducted are presented and discussed. The effectiveness of the selected stabilizers for S/S treatment of oil-contaminated soils was evaluated based on the minimum requirements of UCS and CBR for possible utilization of the treated soils in road construction. TCLP test results, obtained for those mixtures which passed the minimum requirements of UCS and CBR, were used to examine the safety against environmental pollution in case if the treated soils were used in road construction.

## 4.1 Compaction Test Results

The values of optimum moisture content (OMC) obtained corresponding to maximum dry density (MDD) values using the moisture content versus dry density plots for all 31 S/S-treated mixtures are presented in this section. As mentioned earlier, the OMC values obtained individually for each soil mixtures were used in preparation of specimens for UCS, CBR, TCLP and XRD/SEM tests.

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### 4.1.1 Compaction Test Results of HOC Soil

#### 4.1.1.1 Compaction Test Results of Cement-Stabilized HOC Soil

Figure 4.1 shows the plots of compaction test results (i.e., water content versus dry density) obtained for plain soil (i.e., HOC soil without stabilizer) as well for HOC soil mixed with cement contents of 2, 5, 7 and 10%. As can be seen from Figure 4.1, the maximum dry density increases marginally with increase in cement content. This can be attributed to the higher specific gravity of cement as compared to that of HOC soil. Also, it can be noted from Figure 4.1 that there is an increase in the optimum moisture content by the addition of cement. This is due to the increase in demand for water for hydration with increase in cement content.

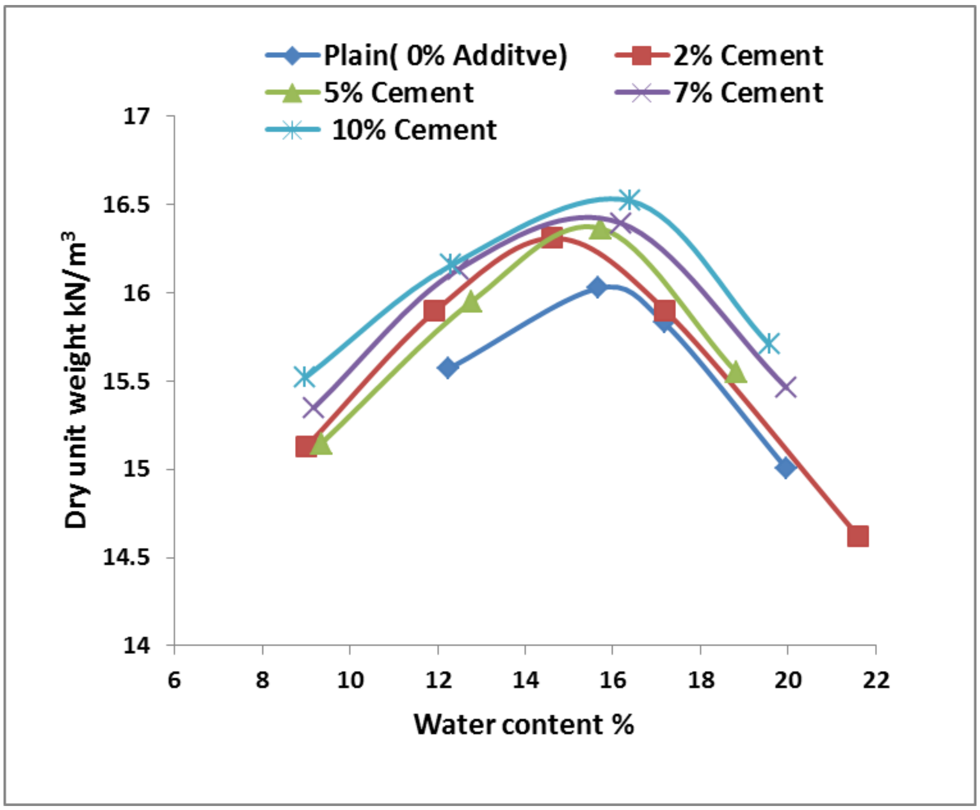


Figure 4.1: Plots of water content and dry density for HOC soil mixed with cement

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### 4.1.1.2 Compaction Test Results of CKD-Stabilized HOC Soil

Figure 4.2 shows the plots of compaction test results obtained for plain soil (i.e., HOC soil without stabilizer) as well for HOC soil mixed with 30% CKD, 30% CKD plus 2% cement, 30% CKD plus 5% cement, 30% CKD plus 7% cement, and 30% CKD plus 10% LSP.

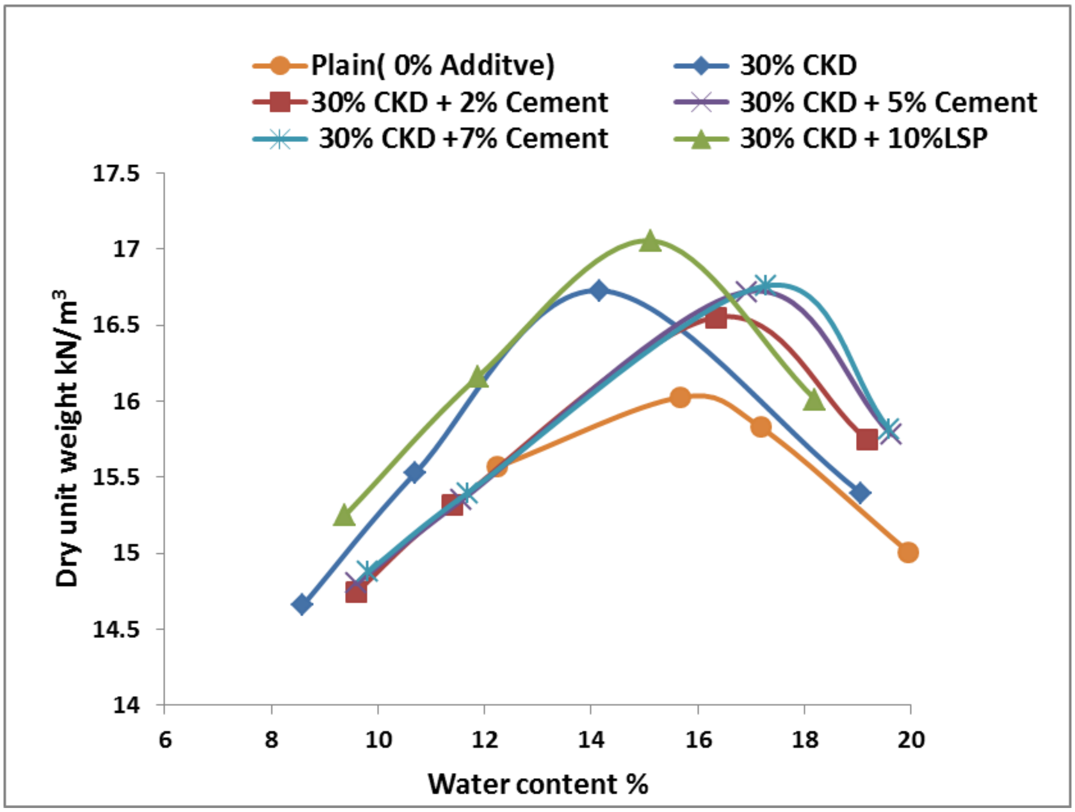


Figure 4.2: Plots of water content and dry density for HOC soil mixed with cement and CKD

### 4.1.1.3 Compaction Test Results of EAFD-Stabilized HOC Soil

Figure 4.3 shows the plots of compaction test results obtained for plain soil (i.e., HOC soil without stabilizer) as well for HOC soil mixed with 20% EAFD, 20% EAFD plus 2% cement, 20% EAFD plus 5% cement, 20% EAFD plus 7% cement, and 20% EAFD plus 10% LSP.

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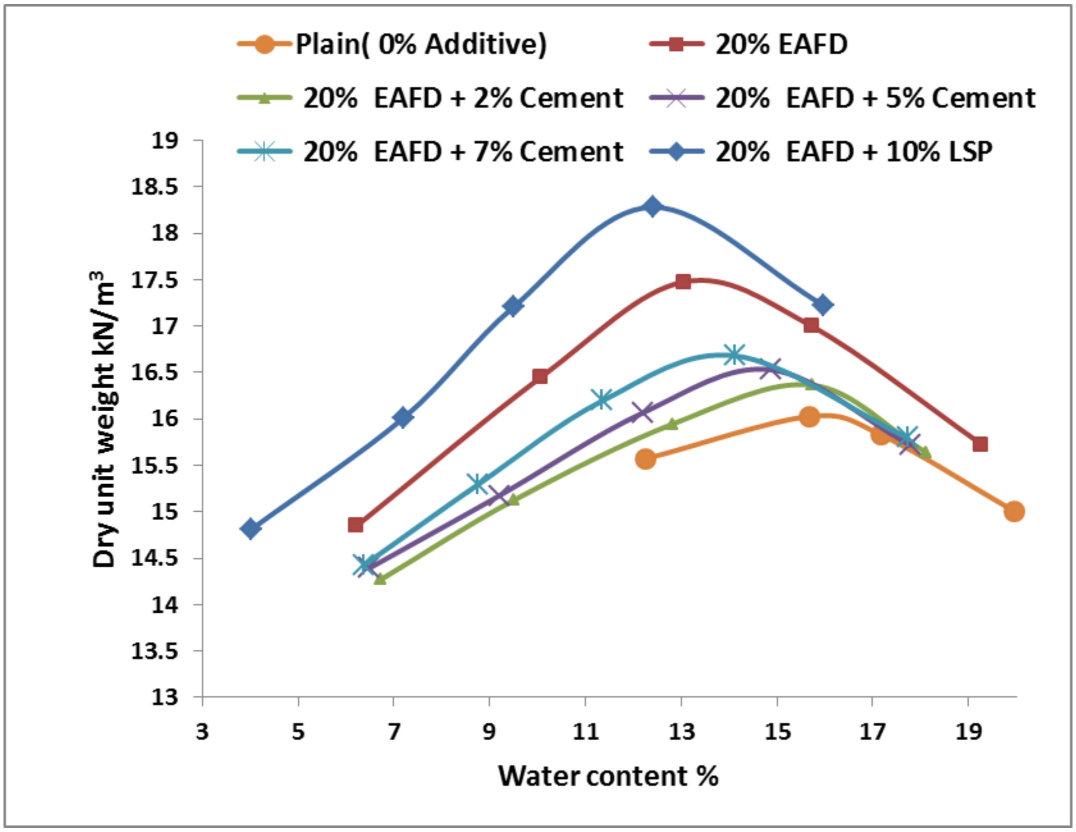


Figure 4.3: Plots of water content and dry density for HOC soil mixed with cement and EAFD

### 4.1.1.4 Compaction Test Results of LSP-Stabilized HOC Soil

Figure 4.4 shows the plots of compaction test results obtained for plain soil (i.e., HOC soil without stabilizer) as well for HOC soil mixed with LSP contents of 5, 10, and 15%. As can be seen from Figure 4.4, maximum dry density increases marginally with increase in LSP content. This can be attributed to the filling of voids in HOC soil by the fine particles of LSP. Also, it can be noted from Figure 4.4 that there is an decrease in the optimum moisture content by the addition of LSP due to reduction in the water requirement because reduction in the voids of HOC soil by filling effect of LSP.

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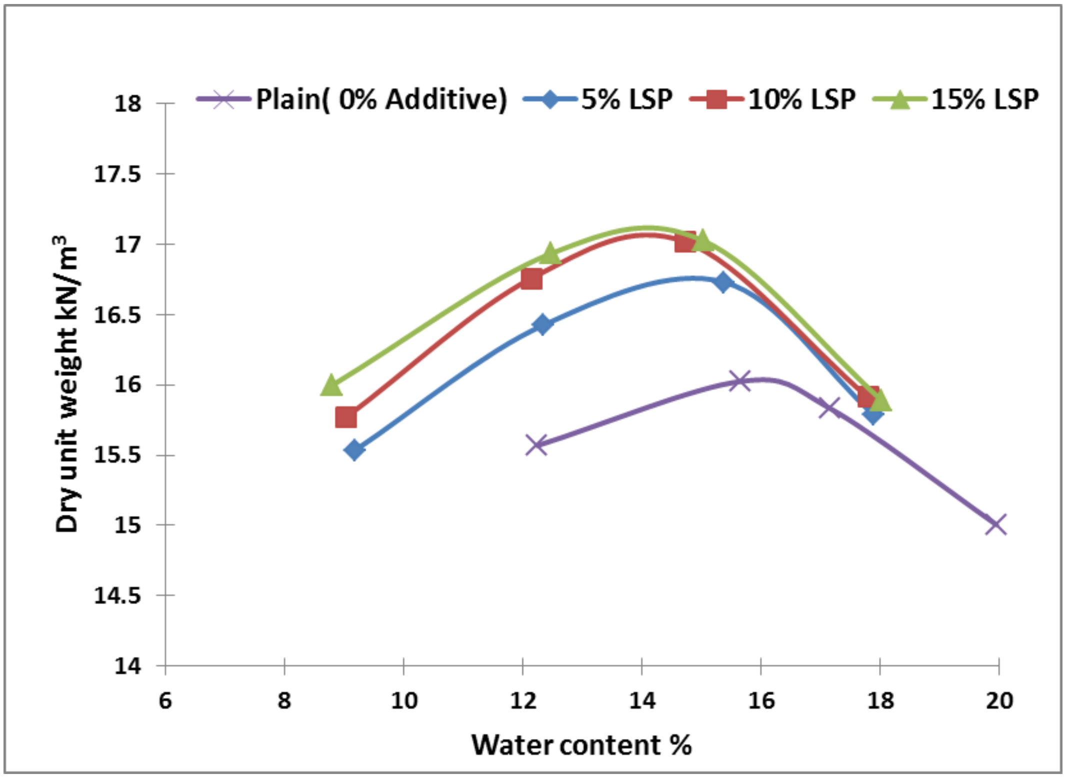


Figure 4.4: Plots of water content and dry density for HOC soil mixed with LSP

Further, the results of compaction tests conducted on the mixtures of HOC soil, cement and LSP are shown in Figure 4.5. It can be observed from Figure 4.5 that the MDD marginally increased with increase in LSP content keeping cement content constant at 2%. At 15% LSP content, the MDD increased when the cement content was increased from 2 to 5% and from 5 to 7%.

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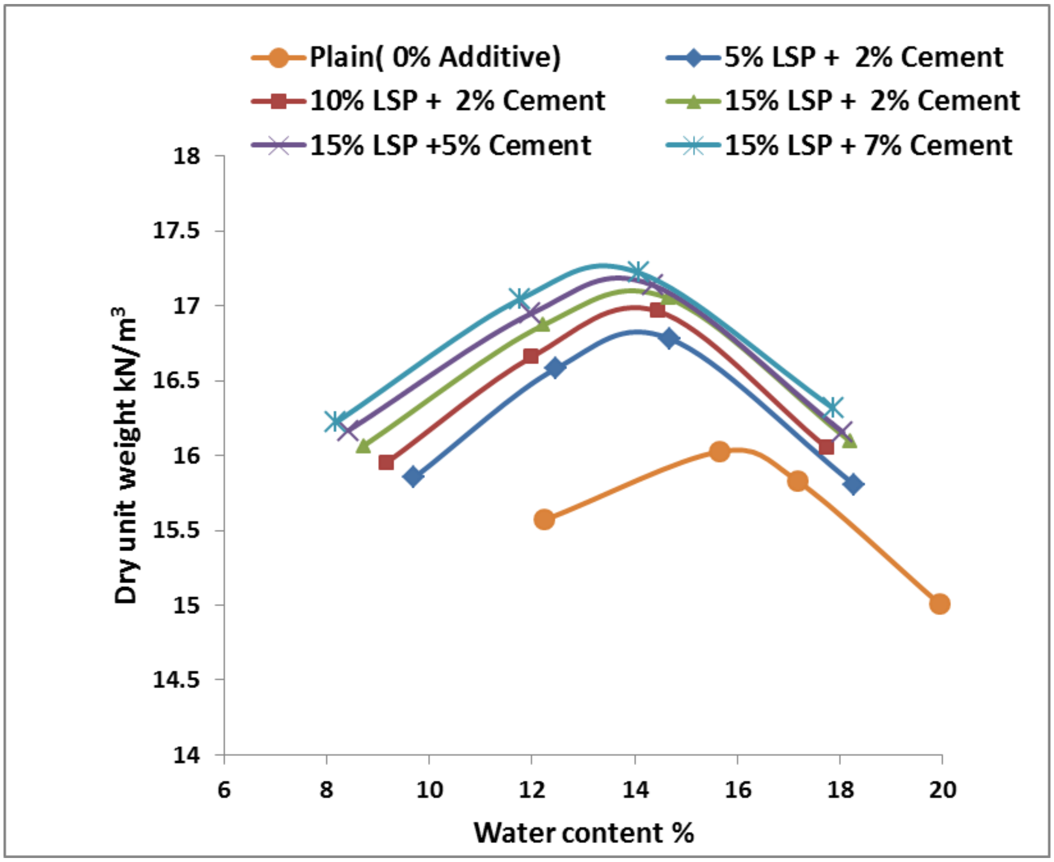


Figure 4.5: Plots of water content and dry density for HOC soil mixed with cement and LSP

### 4.1.2 Compaction Test Results of MOC Soil

Figure 4.6 shows the plots of compaction test results obtained for plain soil (i.e., MOC soil without stabilizer) as well for MOC soil mixed with 7% cement, 30% CKD plus 5% cement and 7% cement plus 15% LSP.

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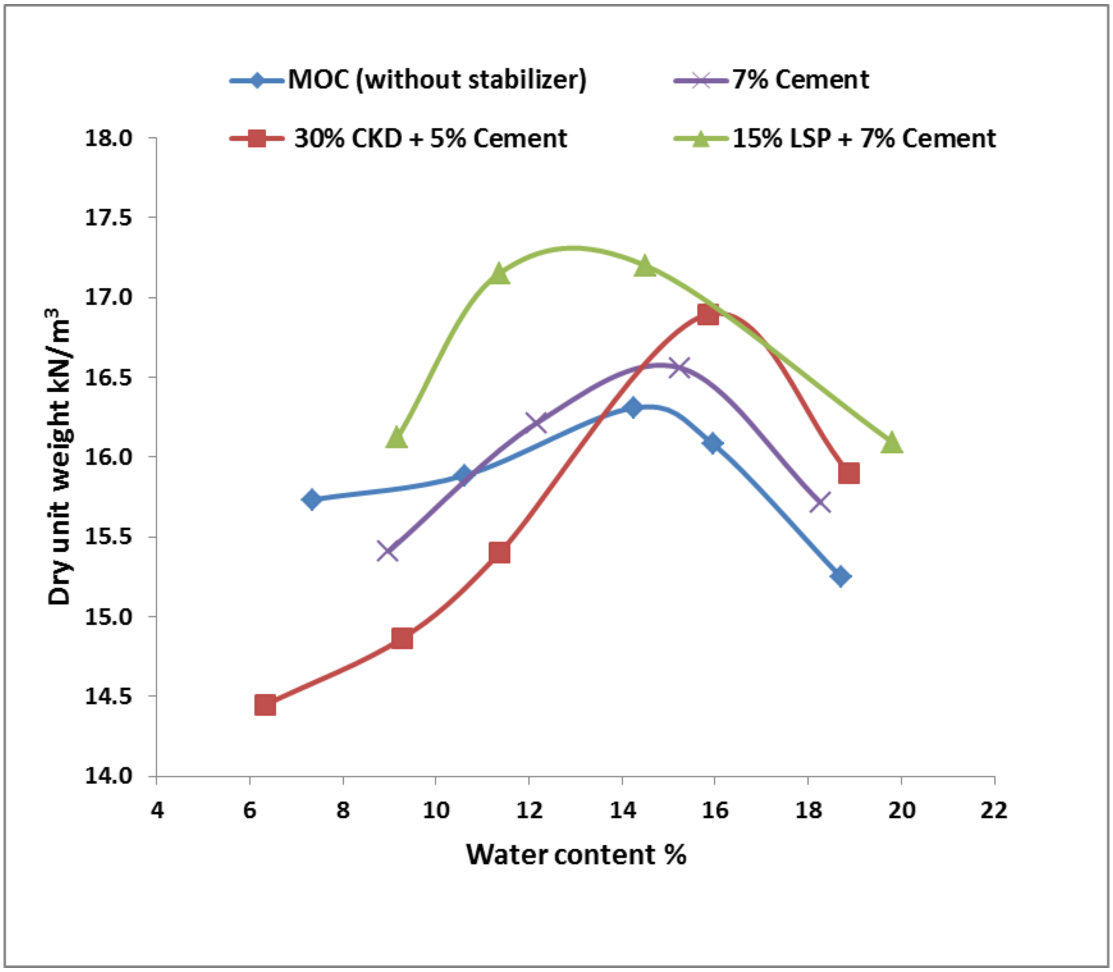


Figure 4.6: Plots of water content and dry density for MOC soil mixed with cement, CKD and LSP

### 4.1.3 Compaction Test Results of LOC Soil

Figure 4.7 shows the plots of compaction test results obtained for plain soil (i.e., LOC soil without stabilizer) as well for LOC soil mixed with 7% cement, 30% CKD plus 5% cement and 7% cement plus 15% LSP.

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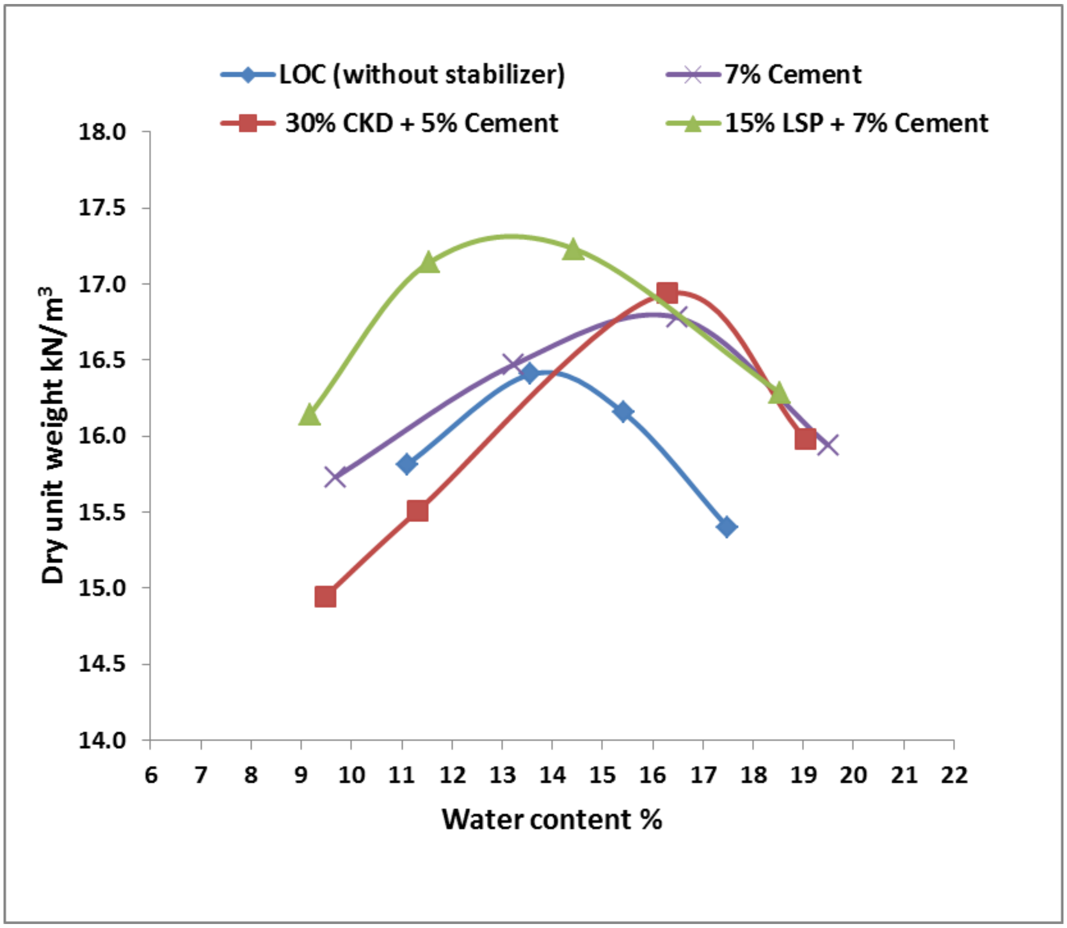


Figure 4.7: Plots of water content and dry density for LOC soil mixed with cement, CKD and LSP

The values of OMC and MDD, obtained from the plots shown in Figures 4.1 through 4.7, for all 31 mixtures of soil and stabilizers, are summarized in Table 4.1.

`

Table 4.1: Summary of OMC and MDD values obtained for all 31 trial mixtures

| Stabilizer Type and Content | HOC | | MOC | | LOC | |
|---|---|---|---|---|---|---|
| | MDD ($kN/m^3$) | OMC (%) | MDD ($kN/m^3$) | OMC (%) | MDD ($kN/m^3$) | OMC (%) |
| Plain (0% Stabilizer) | 16 | 16 | 16.2 | 15.3 | 16.4 | 14.5 |
| 2% Cement | 16.3 | 14.7 | | | | |
| 5% Cement | 16.4 | 15.5 | | | | |
| 7% Cement | 16.4 | 16 | 16.6 | 15.5 | 16.8 | 16 |
| 10% Cement | 16.5 | 16.4 | | | | |
| 30% CKD | 16.7 | 14.2 | | | | |
| 30% CKD + 2% Cement | 16.6 | 16.5 | | | | |
| 30% CKD + 5% Cement | 16.7 | 17 | 16.9 | 16.5 | 16.9 | 16.3 |
| 30% CKD + 7% Cement | 16.8 | 17.5 | | | | |
| 30% CKD + 10%LSP | 17.1 | 15 | | | | |
| 20% EAFD | 17.5 | 13.5 | | | | |
| 20% EAFD + 2% Cement | 16.4 | 15.5 | | | | |
| 20% EAFD + 5% Cement | 16.5 | 14.5 | | | | |
| 20% EAFD + 7% Cement | 16.7 | 14 | | | | |
| 20% EAFD + 10% LSP | 18.3 | 12.5 | | | | |
| 5% LSP | 16.8 | 15 | | | | |
| 10% LSP | 17.1 | 14.4 | | | | |
| 15% LSP | 17.2 | 14.3 | | | | |
| 5% LSP + 2% Cement | 16.8 | 14.2 | | | | |
| 10% LSP + 2% Cement | 17 | 14 | | | | |
| 15% LSP + 2% Cement | 17.1 | 14.1 | | | | |
| 15% LSP + 5% Cement | 17.1 | 13.5 | | | | |
| 15% LSP + 7% Cement | 17.2 | 13.3 | 17.3 | 13 | 17.4 | 13.1 |

`

## 4.2 Unconfined Compressive Strength (UCS) Test Results

The UCS test results of the treated mixtures of HOC soil and air-cured for different periods (7, 28 and 90 days) are presented in Table 4.2.

Table 4.2: UCS of mixtures of HOC soil

| Stabilizer type and content | Unconfined Compressive Strength, UCS (kPa) | | |
|---|---|---|---|
| | 7 days | 28 days | 90 days |
| Plain (0% Stabilizer) | 79 | 87 | 124 |
| 2% Cement | 404 | 453 | 644 |
| 5% Cement | 1124 | 1342 | 1901 |
| 7% Cement | 1728 | 2411 | 3369 |
| 10% Cement | 2551 | 3216 | 4651 |
| 30% CKD | 1117 | 1741 | 2438 |
| 30% CKD + 2% Cement | 1450 | 2218 | 3057 |
| 30% CKD + 5% Cement | 1841 | 3418 | 3953 |
| 30% CKD + 7% Cement | 2098 | 3989 | 4748 |
| 30% CKD + 10%LSP | 1257 | 1893 | 2834 |
| 20% EAFD | 184 | 194 | 221 |
| 20% EAFD + 2% Cement | 446 | 550 | 775 |
| 20% EAFD + 5% Cement | 492 | 1381 | 1871 |
| 20% EAFD + 7% Cement | 625 | 1714 | 2556 |
| 20% EAFD + 10% LSP | 388 | 401 | 499 |
| 5% LSP | 87 | 98 | 132 |
| 10% LSP | 106 | 116 | 160 |
| 15% LSP | 131 | 146 | 224 |
| 5% LSP + 2% Cement | 453 | 691 | 841 |
| 10% LSP + 2% Cement | 731 | 843 | 1053 |
| 15% LSP + 2% Cement | 765 | 1412 | 1898 |
| 15% LSP + 5% Cement | 1430 | 2653 | 3765 |
| 15% LSP + 7% Cement | 1990 | 3546 | 4394 |

`

### 4.2.1 Variation of UCS of HOC Soil with Curing Period

The plots of UCS versus curing time results obtained for treated HOC soil are shown in Figures 4.8 through 4.11. It can be cleared observed from these plots that there is significant increase in UCS with increase in the curing time. It is interesting to note that the curing effect on UCS is more at higher percentages of cement. This is because of completeness of hydration with more curing time which results into more cementing materials at more cement content.

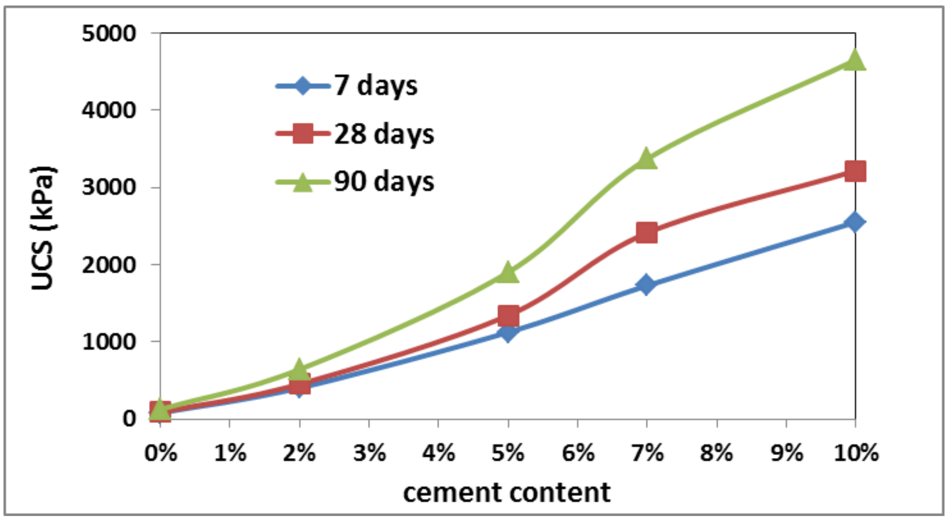


Figure 4.8: UCS of HOC soil mixtures using cement alone as binder

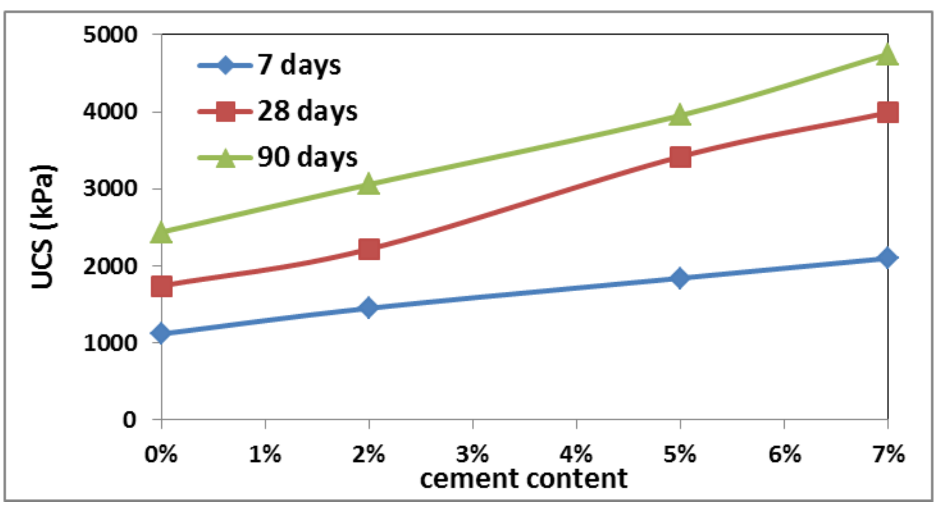


Figure 4.9: UCS of HOC soil mixtures using cement with 30% CKD

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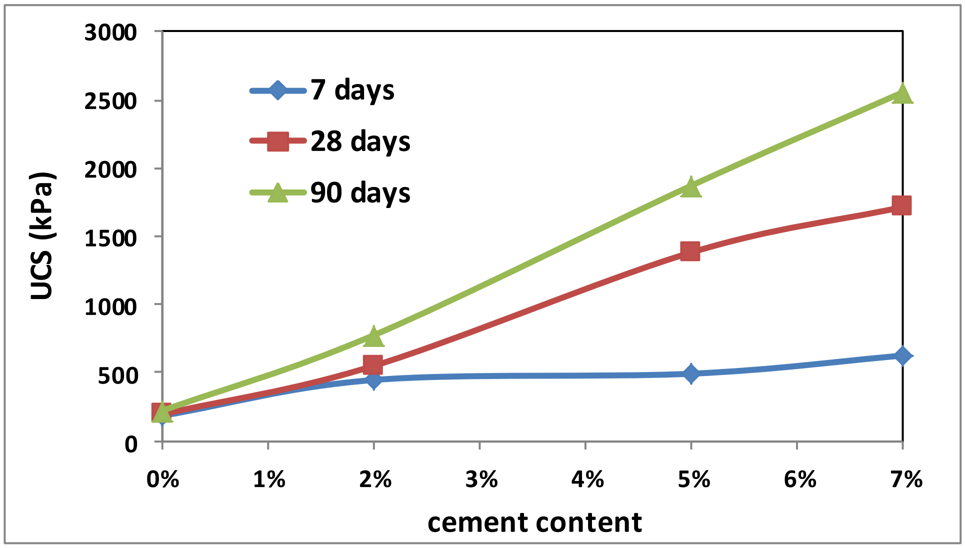


Figure 4.10: UCS of HOC soil mixtures using cement with 20% EAFD

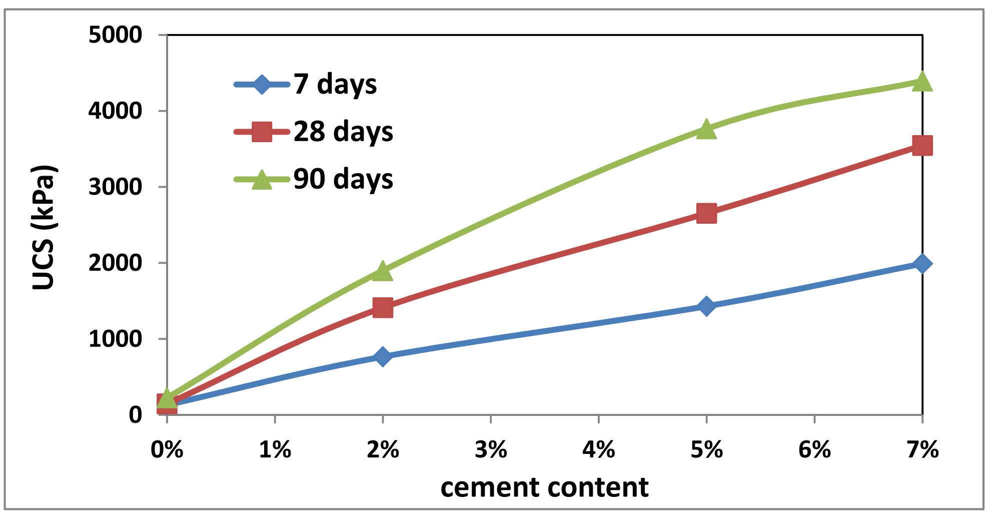


Figure 4.11: UCS of HOC soil mixtures using cement with 15% LSP

### 4.2.2 Exploring Possibility of using S/S treated HOC Soil Mixtures for Road Construction based on 7-day UCS

The 7 day air-cured UCS values obtained for all the treated mixtures of HOC soil were plotted as shown in Figures 4.12 through 4.16. These plots were used to indicate suitability of the stabilized HOC soil mixtures for road construction using the ACI Committee (1990).

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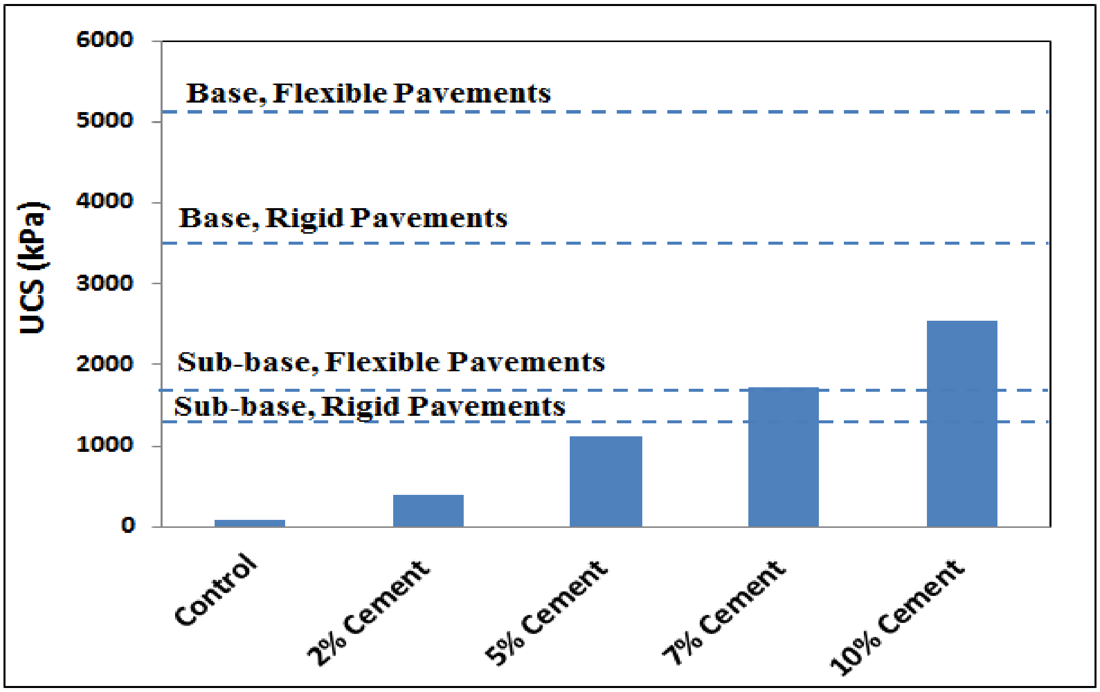


Figure 4.12: Suitability of cement-stabilized HOC soil for road construction

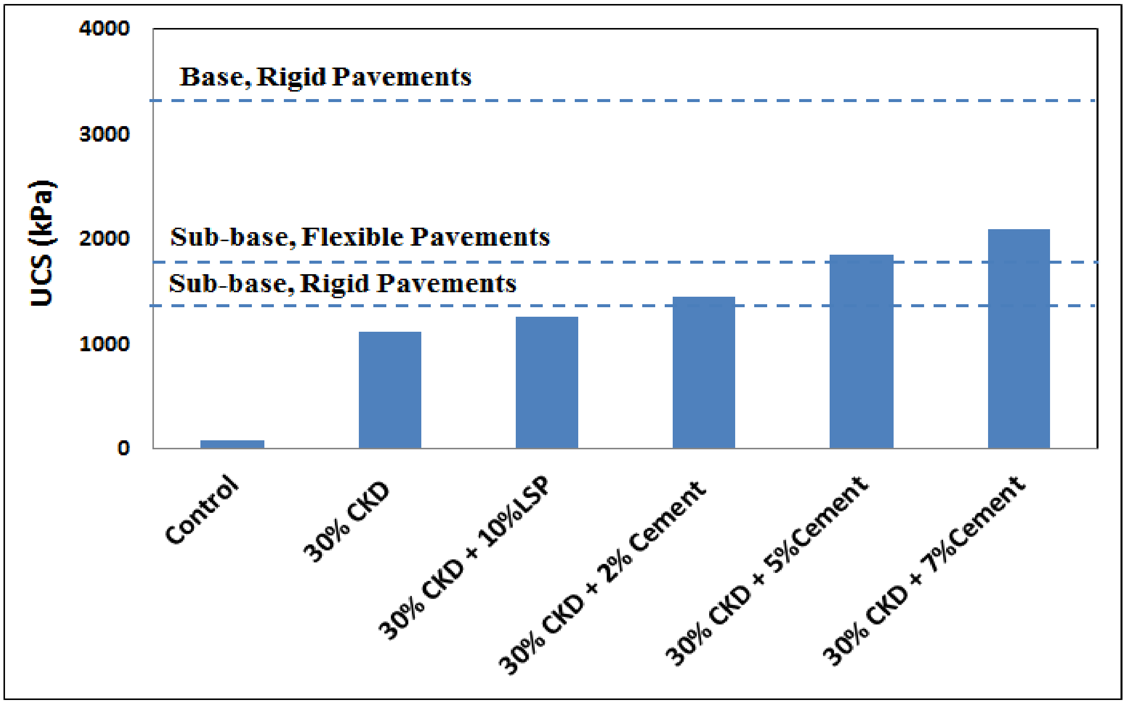


Figure 4.13: Suitability of CKD-Stabilized HOC soil for road construction

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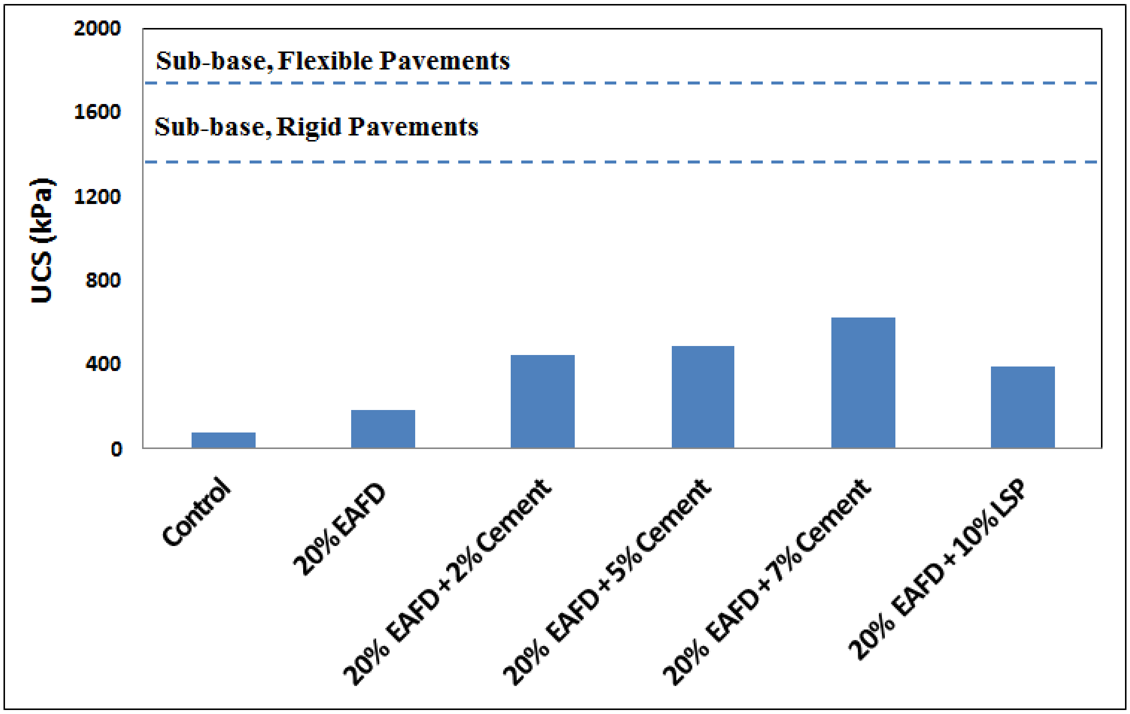


Figure 4.14: Suitability of EAFD-Stabilized HOC soil for road construction

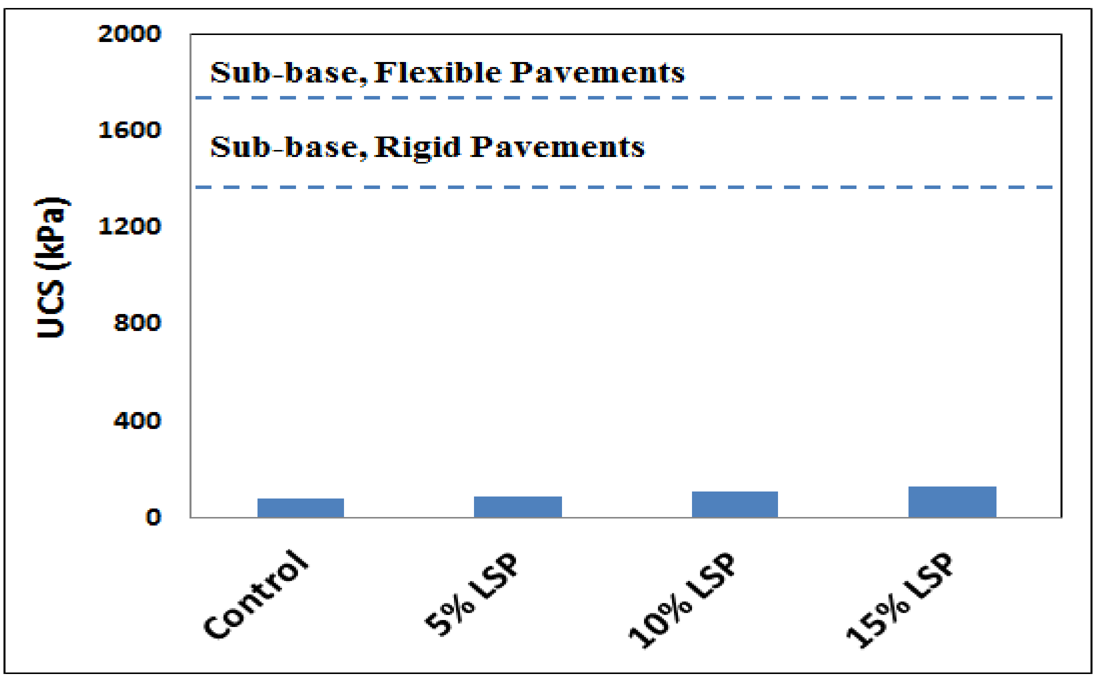


Figure 4.15: Suitability of LSP-Stabilized HOC soil for road construction

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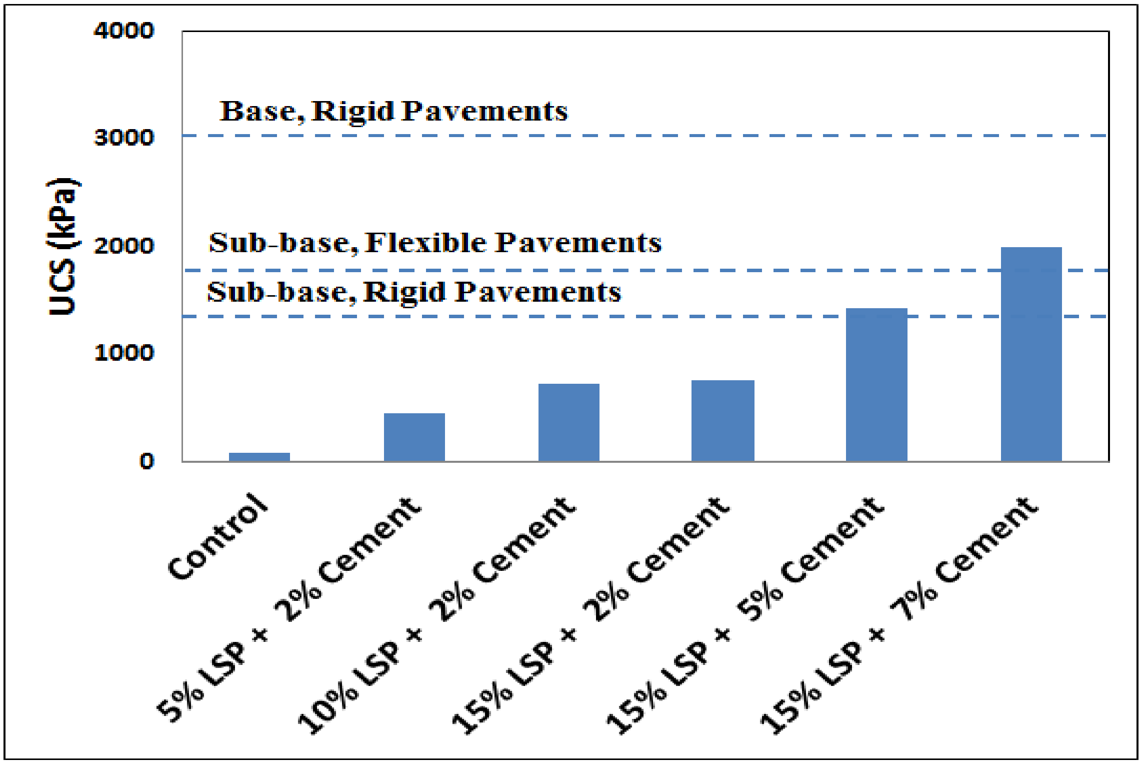


Figure 4.16: Suitability of LSP-Stabilized HOC soil for road construction

Based on the observation of the plots of UCS as shown in Figures 4.12 through 4.16, Table 4.3 summarizes the suitability of selected stabilizers and their dosages that satisfied the minimum UCS requirements for use of treated soil in road construction specified by the ACI Cement Committee 230 Report (1990).

Since 7% cement, 30% CKD + 5% Cement, and 15% LSP + 7% cement satisfied the requirements of 7-day UCS for utilization of S/S-treated HOC soil mixtures, the UCS tests on MOC and LOC soil mixtures were conducted only on those S/S-treated MOC and LOC mixtures corresponding to 7% Cement, 30% CKD + 5% Cement, and 15% LSP + 7% Cement.

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Table 4.3: Stabilizers and their dosages used for S/S treatment of HOC soil which satisfied/not satisfied ACI requirement 7-day UCS

| **Stabilizer Type and Content** | **Rigid Pavement (Sub-Base Course)** | **Flexible Pavement (Sub-Base Course)** | **Rigid Pavement (Base Course)** | **Flexible Pavement (Base Course)** |
|---|---|---|---|---|
| 7% Cement | Yes | Yes | No | No |
| 10% Cement | Yes | Yes | No | No |
| 30% CKD + 2% Cement | Yes | No | No | No |
| 30% CKD + 5% Cement | Yes | Yes | No | No |
| 30% CKD + 7% Cement | Yes | Yes | No | No |
| 15% LSP + 5% Cement | Yes | No | No | No |
| 15% LSP + 7% Cement | Yes | Yes | No | No |

### 4.2.3 UCS Test Results of MOC and LOC Soils

The UCS test results obtained for MOC and LOC soils treated with three sets of stabilizers (7% cement, 30% CKD plus 5% cement, and 15% LSP plus 7% cement) and air-cured for 7, 28 and 90 days, are shown in Figures 4.17 and 4.18. The plots of UCS for all three HOC, MOC and LOC soils, as shown collectively in Figure 4.19, indicates that all three selected sets of stabilizers (7% cement, 30% CKD plus 5% cement, and 15% LSP plus 7% cement) are capable of treating the contaminated soil so that these soils can be utilized as sub-base materials in construction of both flexible as well as rigid pavements.

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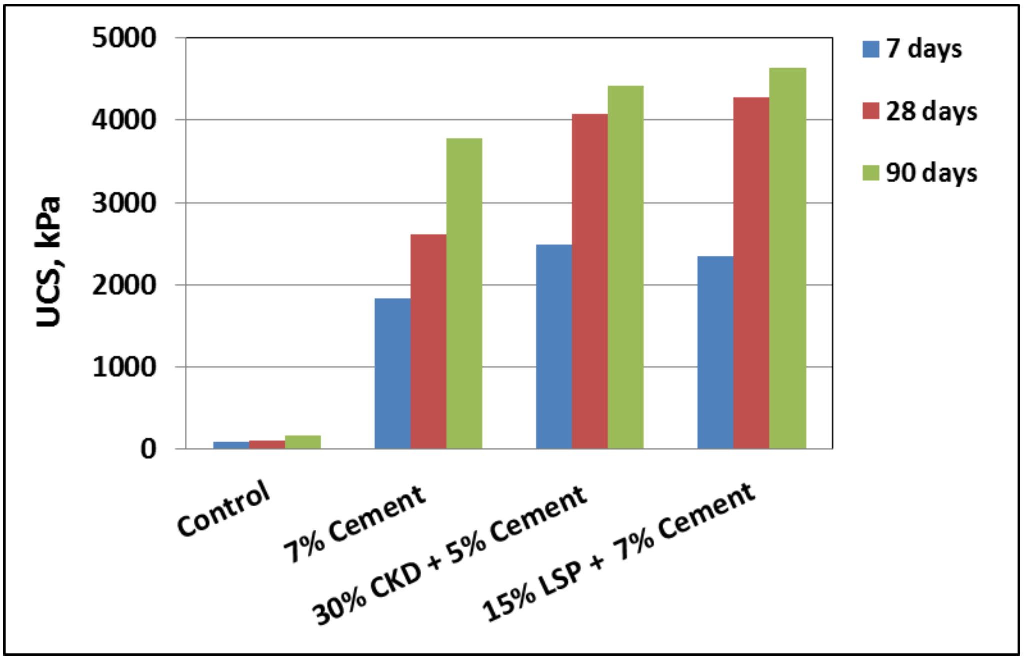


Figure 4.17: Plots of UCS for S/S-treated MOC soil

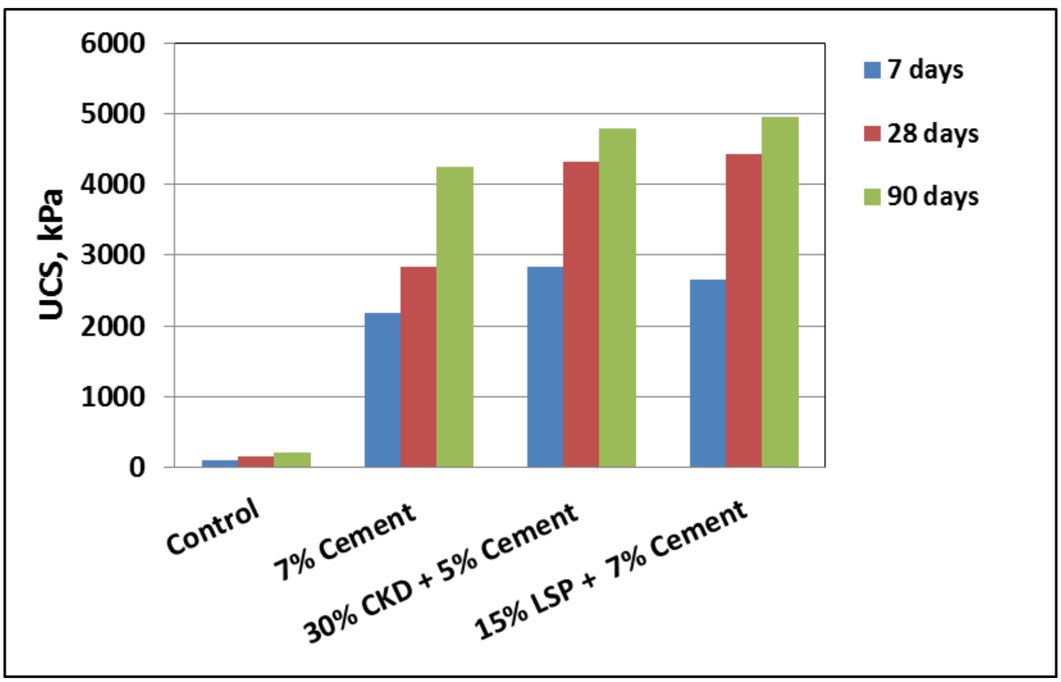


Figure 4.18: Plots of UCS for S/S-treated LOC soil

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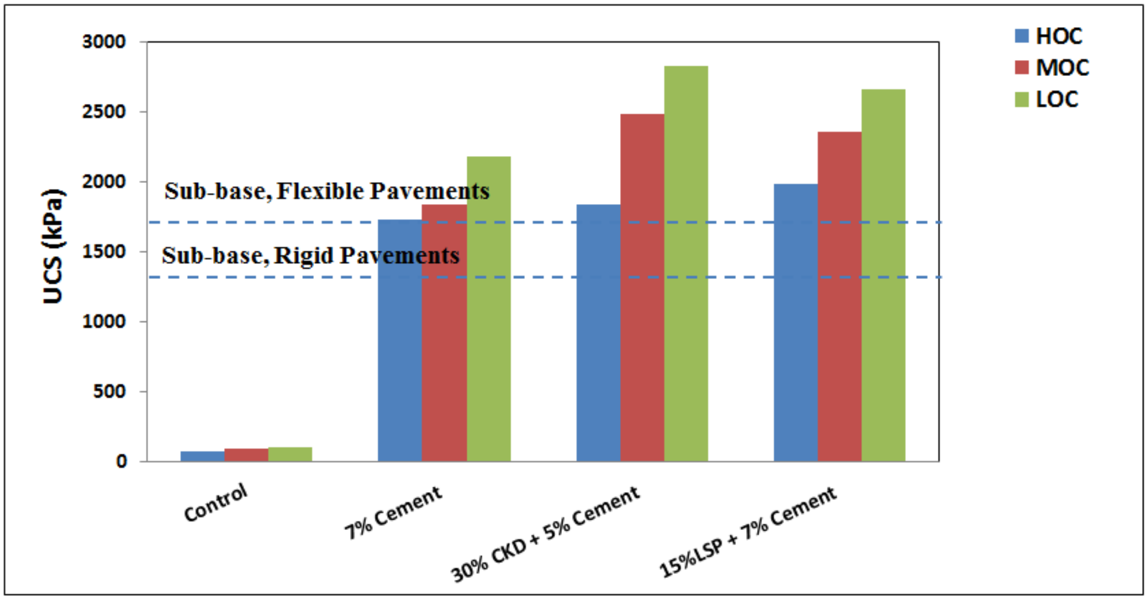


Figure 4.19: Suitability of UCS of S/S-treated HOC, MOC and LOC soils for road construction

## 4.3 Soaked CBR Test Results

Table 4.4 summarizes the test results of soaked CBR of S/S-treated HOC, MOC and LOC soils after 7 day air-curing. It can be observed from Table 4.4 that the minimum CBR requirement (i.e., > 50%) is satisfied only in cases other than the following cases:

i. 2% cement

ii. None of the EAFD mixtures even EAFD with 7% cement

iii. None of LSP mixtures without cement

As can be seen from Figure 4.20, like the case of UCS, all three selected sets of stabilizers (7% cement, 30% CKD plus 5% cement, and 15% LSP plus 7% cement) are capable of maintaining excellent CBR so that they can be utilized as sub-base materials in construction of both flexible as well as rigid pavements.

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Table 4.4: Soaked CBR of S/S-treated HOC, MOC and LOC soils after 7 day air-curing

| **Stabilizer Type and Content** | **CBR (%) for HOC** | **CBR (%) for MOC** | **CBR (%) for LOC** |
|---|---|---|---|
| Plain (0% Stabilizer) | 5 | 5 | 7 |
| 2% Cement | 26 | | |
| 5% Cement | 85 | | |
| 7% Cement | 114 | 121 | 125 |
| 10% Cement | 194 | | |
| 30% CKD | 68 | | |
| 30% CKD + 2% Cement | 80 | | |
| 30% CKD + 5% Cement | 95 | 98 | 100 |
| 30% CKD + 7% Cement | 129 | | |
| 30% CKD + 10%LSP | 73 | | |
| 20% EAFD | 18 | | |
| 20% EAFD + 2% Cement | 35 | | |
| 20% EAFD + 5% Cement | 42 | | |
| 20% EAFD + 7% Cement | 49 | | |
| 20% EAFD + 10% LSP | 24 | | |
| 5% LSP | 11 | | |
| 10% LSP | 14 | | |
| 15% LSP | 19 | | |
| 5% LSP + 2% Cement | 52 | | |
| 10% LSP + 2% Cement | 60 | | |
| 15% LSP + 2% Cement | 66 | | |
| 15% LSP + 5% Cement | 90 | | |
| 15% LSP + 7% Cement | 125 | 129 | 134 |

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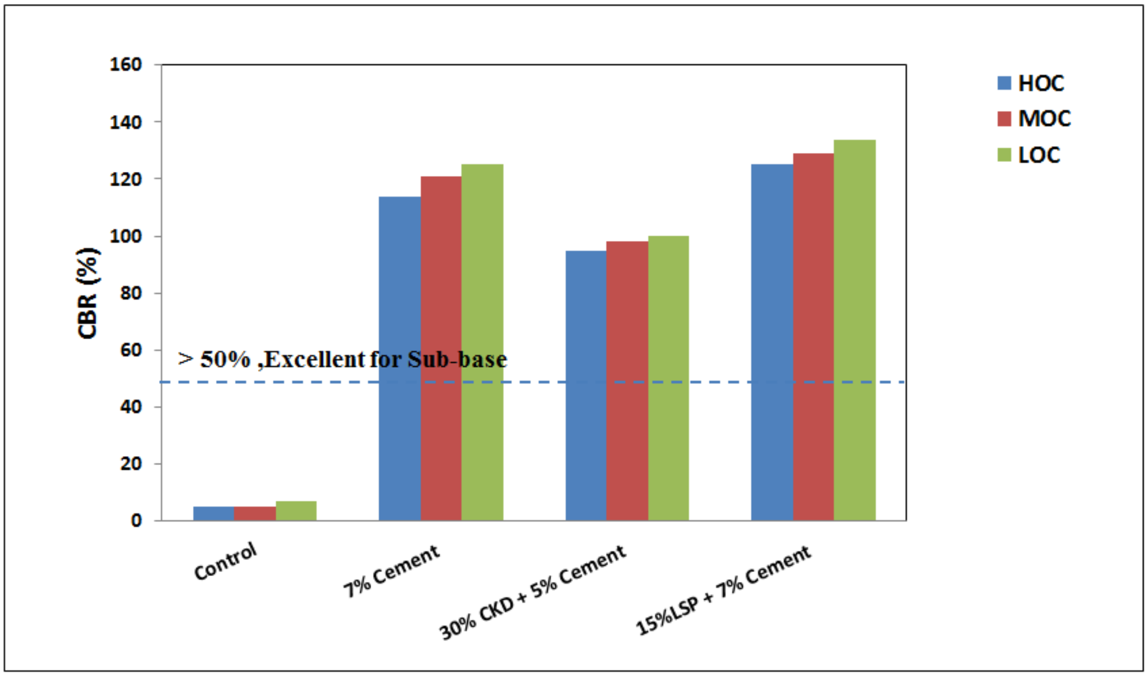


Figure 4.20: Suitability of CBR of S/S-treated HOC, MOC and LOC soils for road construction

## 4.4 TCLP Results

The results of TCLP conducted on HOC, MOC and LOC soils before S/S treatment are presented in Table 4.5 along with the maximum permissible limits of metals and compounds set by EPA. In case of each contaminated soil, the concentrations of metals and compounds are negligible except the concentrations of Barium, Chromium, Nickel and Vanadium. While Barium is within the EPA limit, the concentration of Chromium is found to exceed the permissible limit in case of each contaminated soil. As evident from the TCLP results presented in Tables 4.6 through 4.8, after stabilizing the HOC, MOC and LOC with 7% cement, 30% CKD plus 5% cement, and 15% LSP plus 7% cement and sealed air-curing for 7 days, the concentrations of Barium, Chromium, Nickel and Vanadium were extensively lowered and all chromium was found to be far below the EPA maximum permissible limits.

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Table 4.5: TCLP results for HOC, MOC and LOC soils before S/S treatment

| **Metal and Compound** | **EPA** (mg/l) | TCLP Concentrations (mg/l) | | |
|---|---|---|---|---|
| | | HOC | MOC | LOC |
| Benzene | 0.5 | < 0.00005 | < 0.00005 | < 0.00005 |
| Chlorobenzene | 100 | < 0.00005 | < 0.00005 | < 0.00005 |
| 1,2-Dichloroethane | 0.5 | < 0.00005 | < 0.00005 | < 0.00005 |
| Tetrachloroethene | 0.7 | < 0.00005 | < 0.00005 | < 0.00005 |
| Arsenic (As) | 5 | < 5 | < 5 | < 5 |
| **Barium(Ba)** | **100** | **14.6** | **12.2** | **18.3** |
| Cadmium(Cd) | 1 | < 0.1 | < 0.1 | < 0.1 |
| **Chromium(Cr)** | **5** | **7.1** | **6.9** | **6.2** |
| Lead(Pb) | 5 | < 1 | < 1 | < 1 |
| Mercury(Hg) | 0.2 | < 0.001 | < 0.001 | < 0.001 |
| Selenium(Se) | 1 | < 1 | < 1 | < 1 |
| Silver(Ag) | 5 | < 0.005 | < 0.005 | < 0.005 |
| **Nickel(Ni)** | Not regulated | **9.3** | **8.4** | **8.3** |
| **Vanadium(V)** | Not regulated | **8.2** | **7.8** | **7.6** |

Table 4.6: TCLP results for HOC soil stabilized with 7% cement, 30% CKD + 5% cement and 15% LSP+ 7% cement (tested after sealed air-curing for 7 days)

| **Metal** | **EPA** (mg/l) | TCLP Concentrations (mg/l) | | |
|---|---|---|---|---|
| | | 7% cement | 30% CKD + 5% cement | 15% LSP + 7% cement |
| Arsenic (As) | 5 | < 0.005 | < 0.005 | < 0.005 |
| Barium(Ba) | 100 | **0.36** | **0.256** | **0.425** |
| Cadmium(Cd) | 1 | < 0.015 | < 0.015 | < 0.015 |
| Chromium(Cr) | 5 | **< 0.05** | **< 0.05** | **< 0.05** |
| Lead (Pb) | 5 | < 0.25 | < 0.25 | < 0.25 |
| Mercury(Hg) | 0.2 | < 0.001 | < 0.001 | < 0.001 |
| Selenium(Se) | 1 | < 0.15 | < 0.15 | < 0.15 |
| Silver(Ag) | 5 | < 0.005 | < 0.005 | < 0.005 |
| Nickel(Ni) | Not regulated | **< 0.015** | **0.015** | **< 0.015** |
| Vanadium(V) | Not regulated | **< 0.05** | **< 0.05** | **< 0.05** |

Table 4.7: TCLP results for MOC soil stabilized with 7% cement, 30% CKD + 5% cement and 15% LSP+ 7% cement (tested after sealed air-curing for 7 days)

| Metal | EPA (mg/l) | TCLP Concentrations (mg/l) | | |
|---|---|---|---|---|
| | | 7% cement | 30% CKD + 5% cement | 15% LSP + 7% cement |
| Arsenic (As) | 5 | < 0.005 | < 0.005 | < 0.005 |
| Barium(Ba) | 100 | **0.403** | **0.238** | **0.349** |
| Cadmium(Cd) | 1 | < 0.015 | < 0.015 | < 0.015 |
| Chromium(Cr) | 5 | **< 0.05** | **< 0.05** | **< 0.05** |
| Lead(Pb) | 5 | < 0.25 | < 0.25 | < 0.25 |
| Mercury(Hg) | 0.2 | < 0.001 | < 0.001 | < 0.001 |
| Selenium(Se) | 1 | < 0.15 | < 0.15 | < 0.15 |
| Silver(Ag) | 5 | < 0.005 | < 0.005 | < 0.005 |
| Nickel(Ni) | Not regulated | **< 0.015** | **0.019** | **< 0.015** |
| Vanadium(V) | Not regulated | **< 0.05** | **< 0.05** | **< 0.05** |

Table 4.8: TCLP results for LOC soil stabilized with 7% cement, 30% CKD + 5% cement and 15% LSP+ 7% cement (tested after sealed air-curing for 7 days)

| Metal | EPA (mg/l) | TCLP Concentrations (mg/l) | | |
|---|---|---|---|---|
| | | 7% cement | 30% CKD + 5% cement | 15% LSP + 7% cement |
| Arsenic (As) | 5 | < 0.005 | < 0.005 | < 0.005 |
| Barium(Ba) | 100 | **0.444** | **0.237** | **0.372** |
| Cadmium(Cd) | 1 | < 0.015 | < 0.015 | < 0.015 |
| Chromium(Cr) | 5 | **< 0.05** | **< 0.05** | **< 0.05** |
| Lead(Pb) | 5 | < 0.25 | < 0.25 | < 0.25 |
| Mercury(Hg) | 0.2 | < 0.001 | < 0.001 | < 0.001 |
| Selenium(Se) | 1 | < 0.15 | < 0.15 | < 0.15 |
| Silver(Ag) | 5 | < 0.005 | < 0.005 | < 0.005 |
| Nickel(Ni) | Not regulated | **< 0.015** | **0.018** | **< 0.015** |
| Vanadium(V) | Not regulated | **< 0.05** | **< 0.05** | **< 0.05** |

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## 4.5 Micro-Characterization Results (SEM and EDX IMAGES)

The SEM images of HOC, MOC and LOC soils treated with various stabilizers (7% cement, 30% CKD plus 5% cement, and 15% LSP plus 7% cement) and subjected to sealed air-curing for 7 days are shown in Figures 4.21 through 4.23. The SEM images are showing some differences in the microstructures of different soils treated with different stabilizer indicating the effect of the type of soil and type of stabilizer on the microstructure of S/S-treated soil. However, more expertize is needed for precise information using these SEM micrographs.

The Energy Dispersive X-ray (EDX) images of HOC, MOC and LOC soils treated with various stabilizers (7% cement, 30% CKD plus 5% cement, and 15% LSP plus 7% cement) and subjected to sealed air-curing for 7 days are shown in Figures 4.24 through 4.26. It can be observed from the EDX images that three major elements (Ca, Na, abd Cl) are found more or less in each case.

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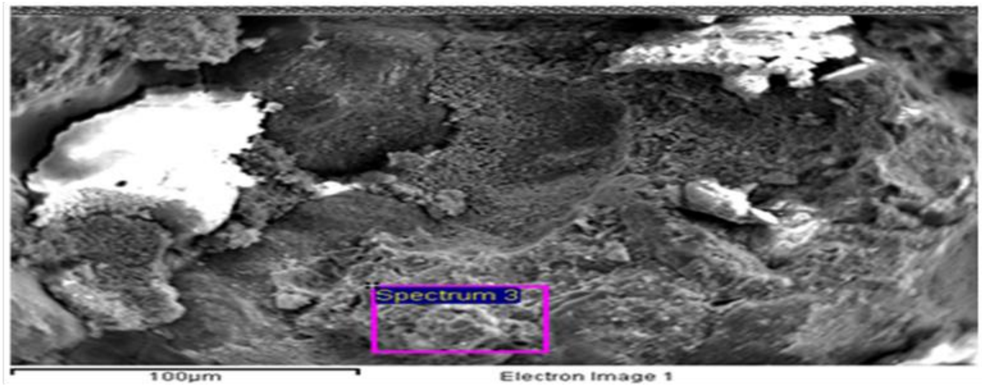
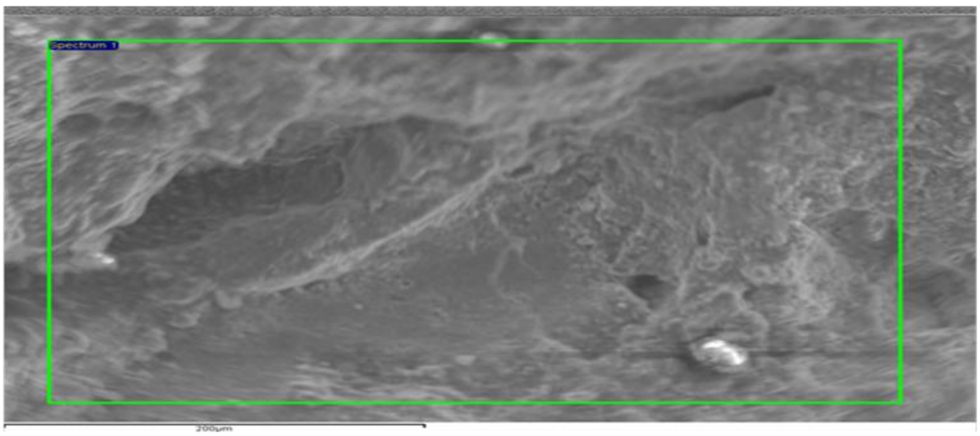
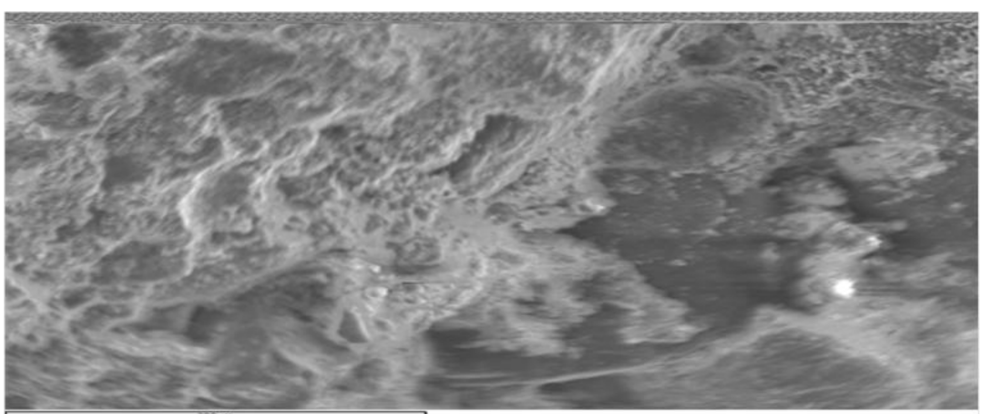



SEM of HOC stabilized with 7% cement (X400)

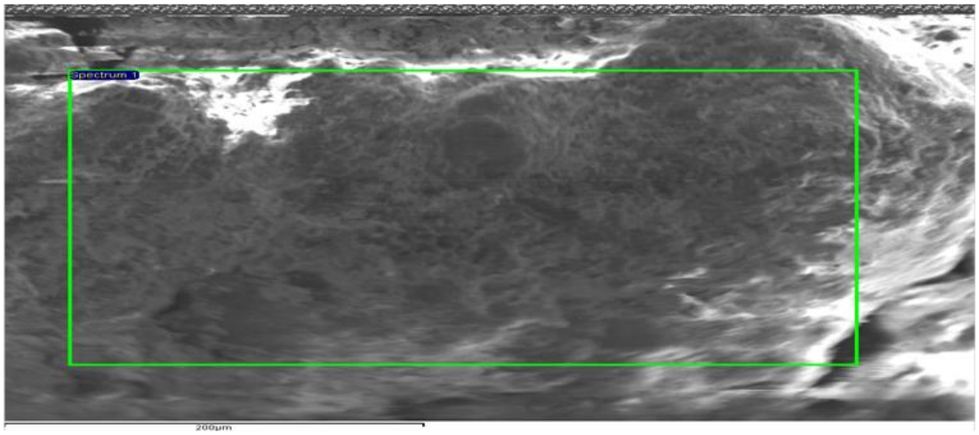



SEM of HOC stabilized with 30% CKD plus 5% cement (X400)

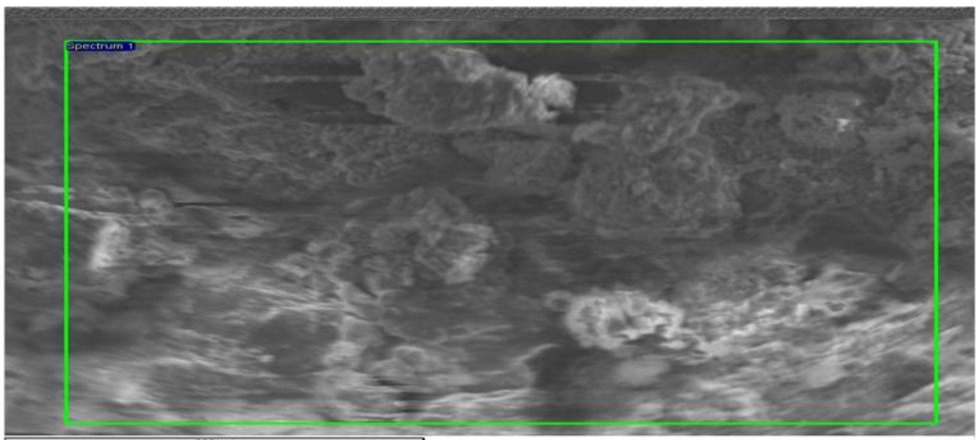


SEM of HOC stabilized with15%LSP plus 7% cement (X400)

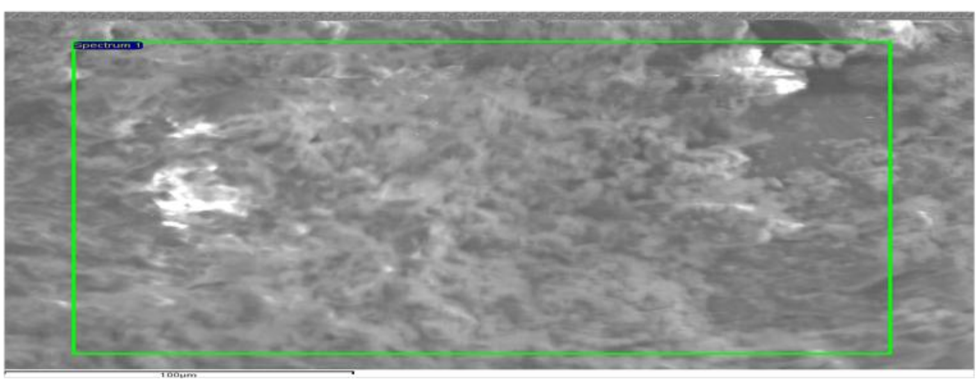
Figure 4.21: SEM images of HOC soil treated with various stabilizers



SEM of MOC stabilized with 7% cement (X400)

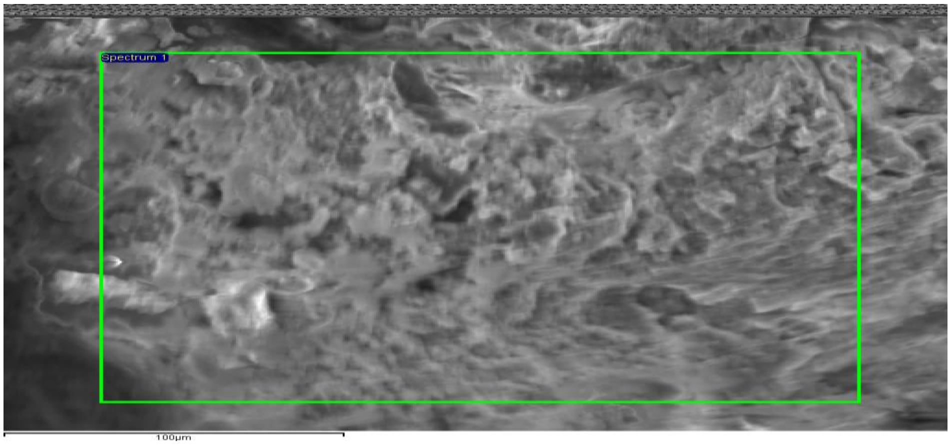


SEM of MOC stabilized with 30%CKD plus 5% cement (X400)

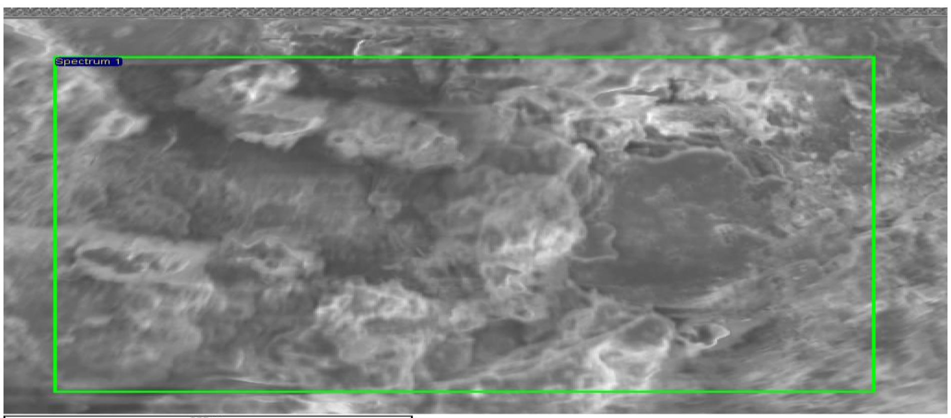


SEM of MOC stabilized with15%LSP plus 7% cement (X400)

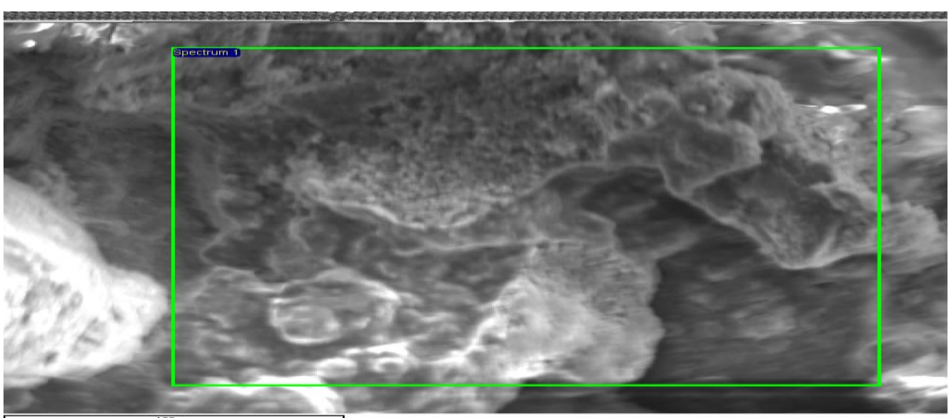
Figure 4.22: SEM images of MOC soil treated with various stabilizers




SEM of LOC stabilized with 7% cement (X400)

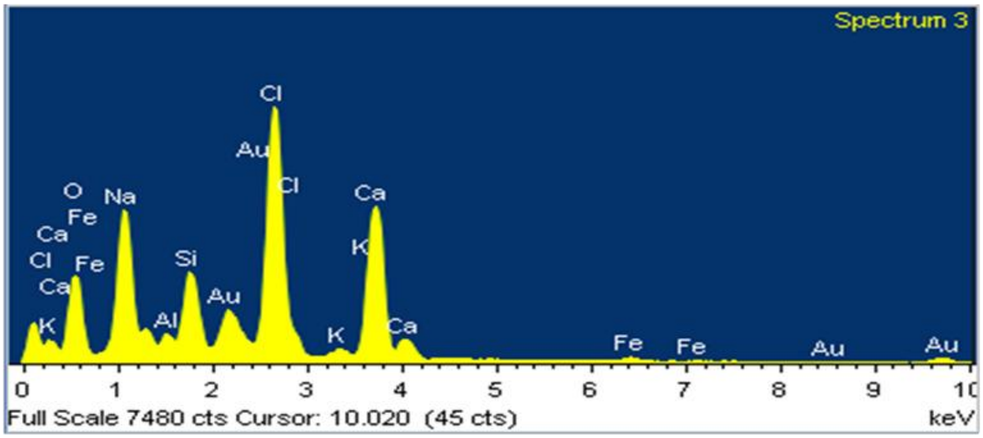



SEM of LOC stabilized with 30%CKD plus 5% cement (X400)

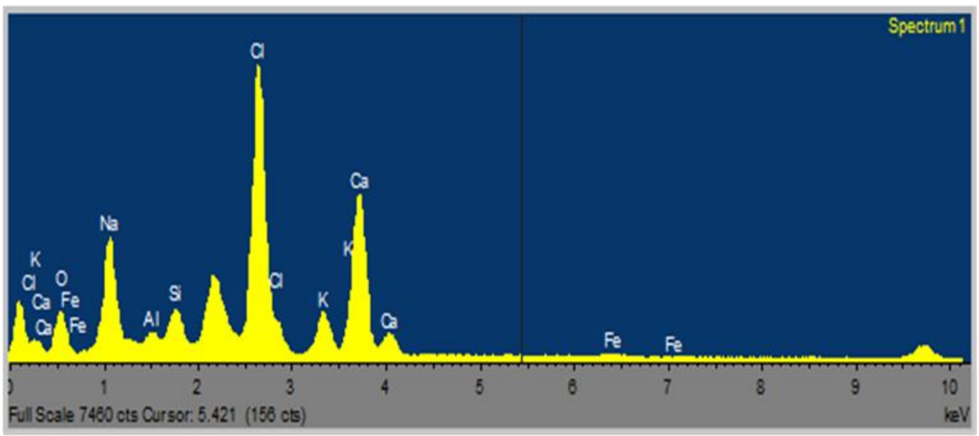



SEM of LOC stabilized with15%LSP plus 7% cement (X400)

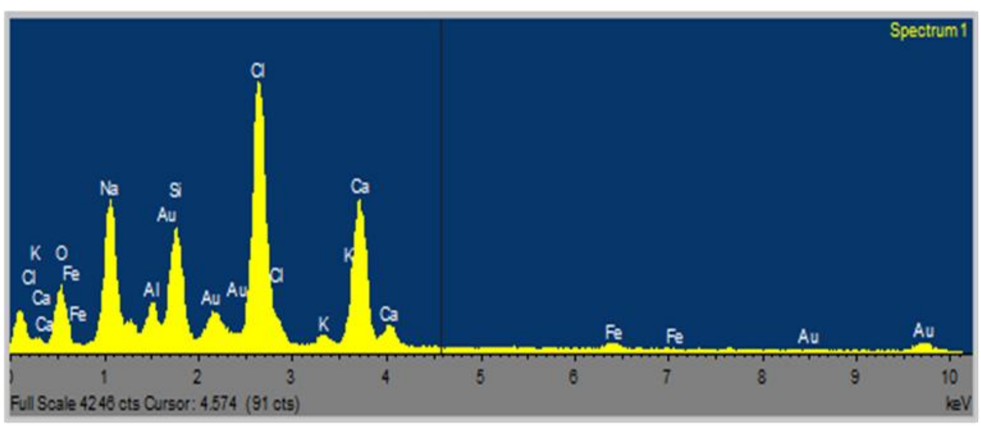
Figure 4.23: SEM images of LOC soil treated with various stabilizers




EDX of HOC stabilized with 7% cement




EDX of HOC stabilized with 30% CKD plus 5% cement




EDX of HOC stabilized with15% LSP plus 7% cement

Figure 4.24: EDX images of HOC soil treated with various stabilizers

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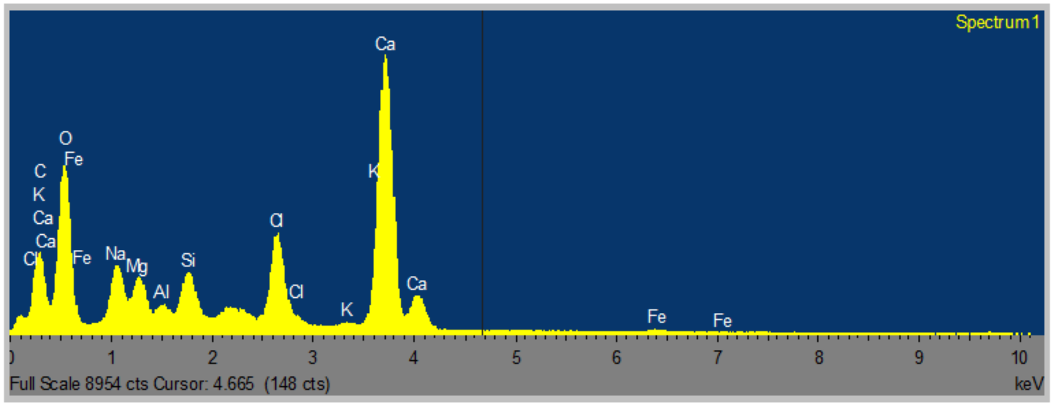
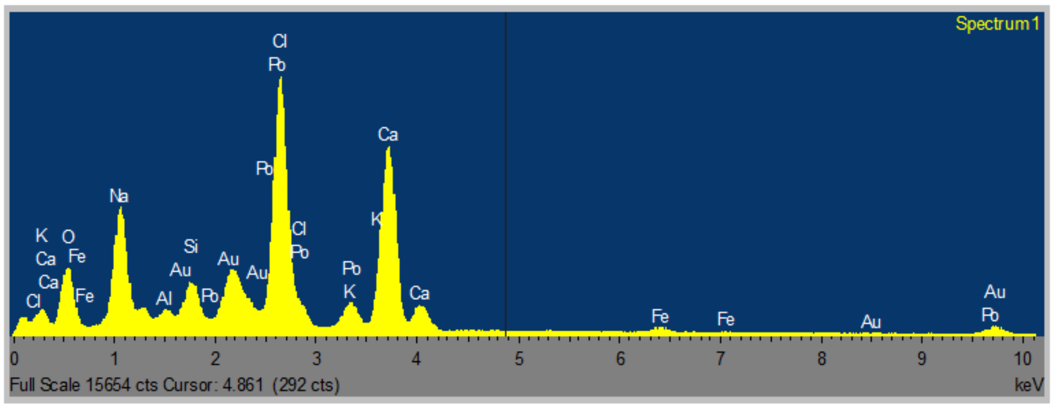
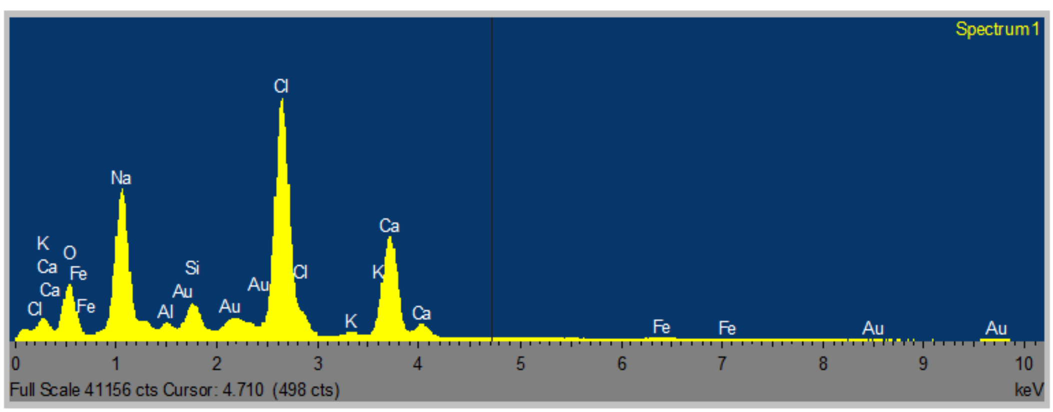



EDX of MOC stabilized with 7% cement

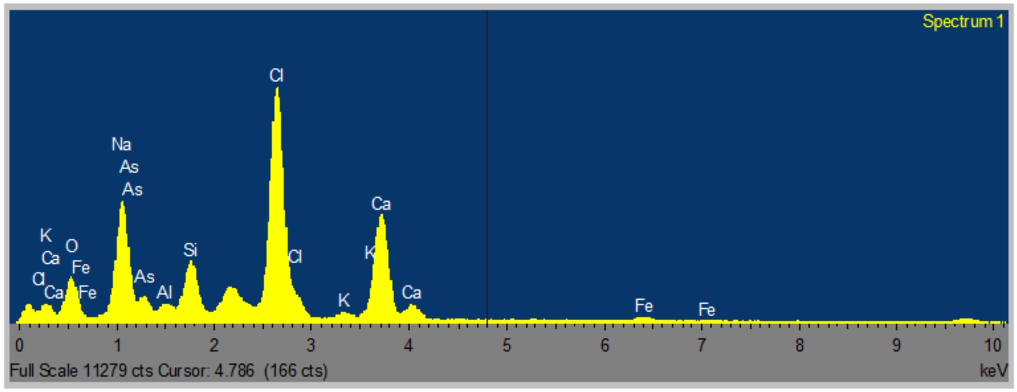



EDX of MOC stabilized with 30% CKD plus 5% cement

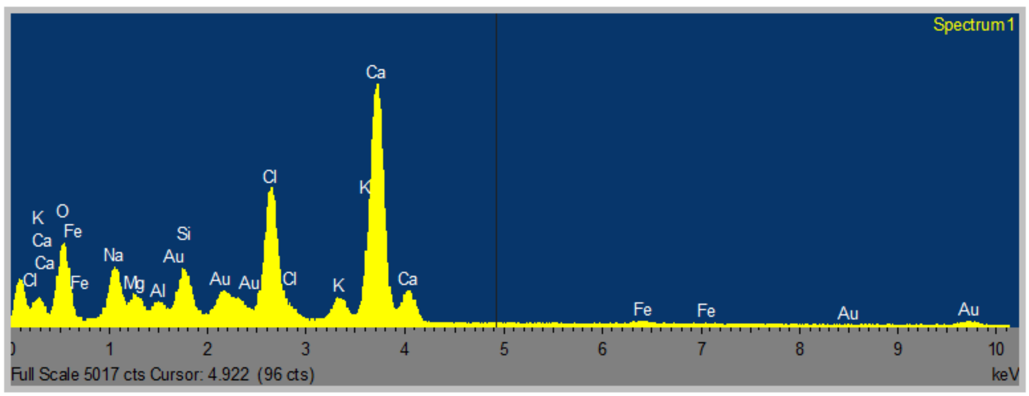



EDX of MOC stabilized with15% LSP plus 7% cement

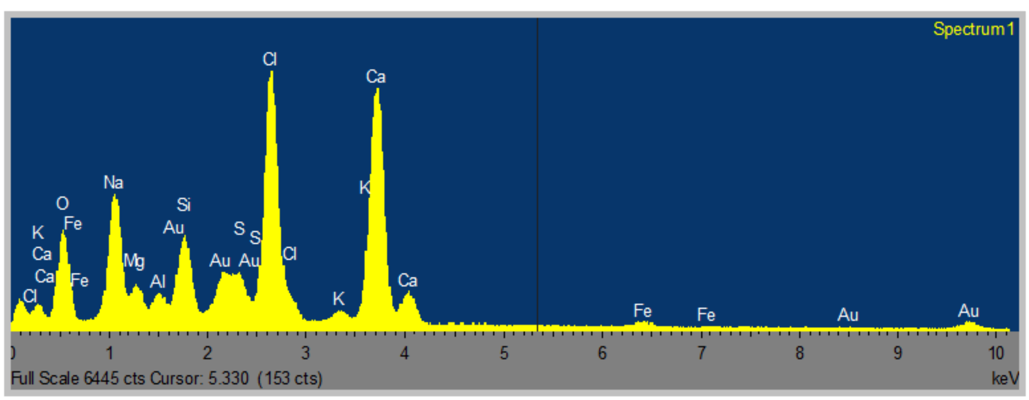
Figure 4.25: EDX images of MOC soil treated with various stabilizers




EDX of LOC stabilized with 7% cement




EDX of LOC stabilized with 30% CKD plus 5% cement




EDX of LOC stabilized with15% LSP plus 7% cement

Figure 4.26: EDX images of LOC soil treated with various stabilizers

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## 4.6 Statistical Analysis of UCS Test Results

The UCS versus cement content test results of S/S-treated HOC soil, air cured for 7, 28 and 90 days, were plotted as shown in Figures 4.27 through 4.29, respectively. The data plotted in Figures 4.27 through 4.29 were best-fitted for obtaining the correlations between cement content and UCS for the three combinations of stabilizers (cement alone, cement plus 30% CKD, and cement plus 15% LSP). A set of 9 best fitted equations of UCS in terms of cement content are presented in Table 4.9.

Using the Equations for 7 day air-curing, the minimum dosages of stabilizers needed for achieving targeted UCS values, as per ACI (1990), for utilization of treated HOC soil in sub-base courses of rigid and flexible pavements, were calculated and tabulated in Table 4.10.

The minimum required dosages of alternative stabilizers worked out and presented in Table 4.10 can be used to select a suitable stabilizer considering availability, economy and environmental concerns.

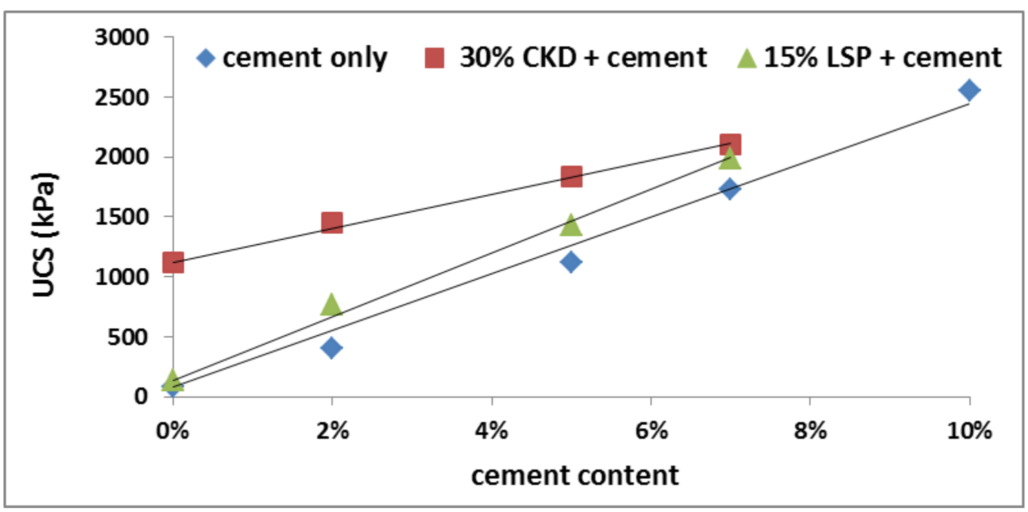


Figure 4.27: Effect of cement content on UCS of S/S treated HOC and air-cured for 7 days

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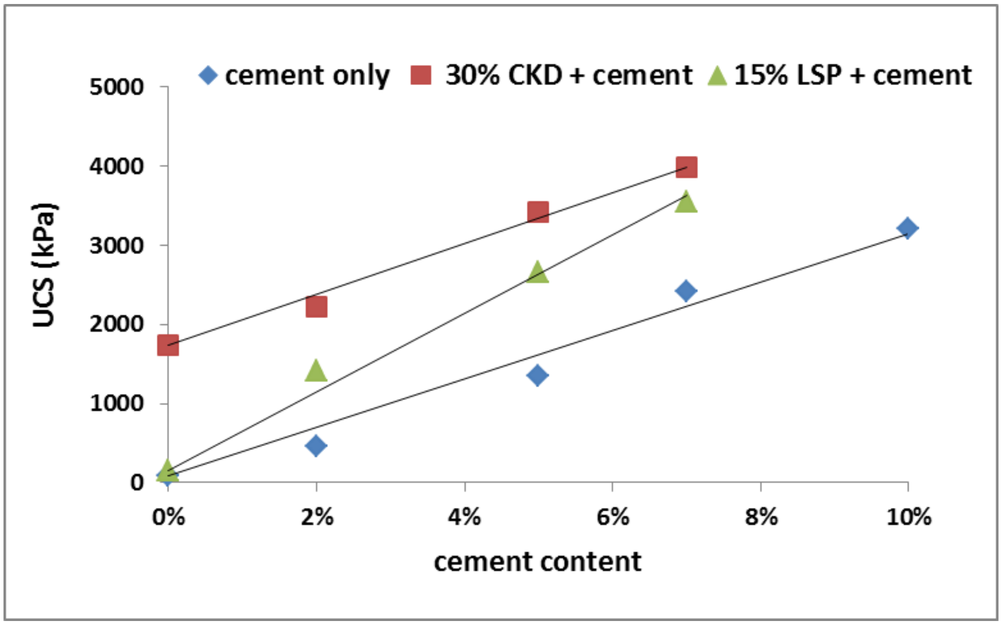


Figure 4.28: Effect of cement content on UCS of S/S treated HOC and air-cured for 28 days

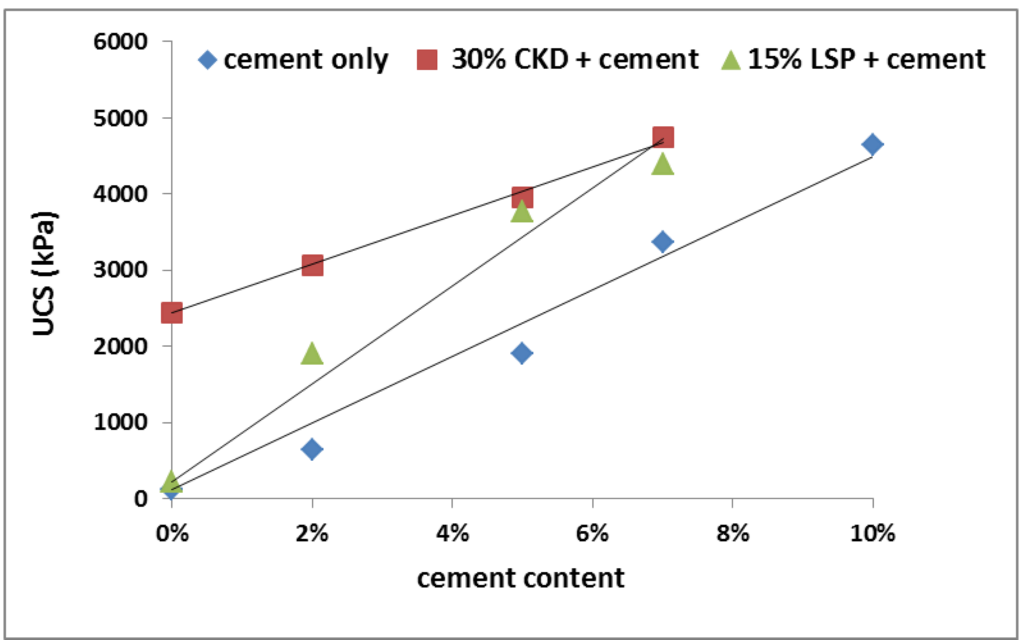


Figure 4.29: Effect of cement content on UCS of S/S treated HOC and air-cured for 90 days

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Table 4.9: Best-fitted UCS equations

| Stabilizer | UCS Equation for 7 day air-curing | UCS Equation for 28 day air-curing | UCS Equation for 90 day air-curing |
|---|---|---|---|
| Cement only | UCS = 23673C+79 [$R^2$ = 0.99] | UCS = 30654C+87 [$R^2$ = 0.97] | UCS = 43770C+124 [$R^2$ = 0.98] |
| Cement + 30% CKD | UCS = 14301C+1117 [$R^2$ = 0.99] | UCS = 32147C+1471 [$R^2$ = 0.99] | UCS =32029C+2438 [$R^2$ = 0.99] |
| Cement + 15% LSP | UCS = 26635C+131 [$R^2$ = 0.99] | UCS = 49829C+146 [$R^2$ = 0.99] | UCS =64414C+224 [$R^2$ = 0.97] |
| Where<br>UCS= Unconfined compressive strength of HOC soil, kPa.<br>C= Cement content (%).<br>$R^2$ = Correlation coefficient | | | |

Table 4.10: Minimum dosages of stabilizers needed for utilization of treated HOC soil in sub-base courses of rigid and flexible pavements

| Stabilizer | For use as sub-base material in rigid pavement (minimum 7-day UCS required: 1380 kPa) | For use as sub-base material in flexible pavement (minimum 7-day UCS required: 1725 kPa) |
|---|---|---|
| Cement only | 5.5% cement | 6.9% cement |
| Cement + 30% CKD | 1.8% cement + 30% CKD | 4.3% cement + 30% CKD |
| Cement + 15% LSP | 4.7% cement + 15% LSP | 6.0% cement + 15% LSP |

`

## 4.7 Statistical Analysis of Soaked CBR Test Results

The 7 day soaked CBR test results for HOC soil treated with three combinations of stabilizers (cement alone, cement plus 30% CKD, and cement plus 15% LSP) are plotted in Figure 4.30 to obtain the best-fitted correlations between soaked CBR and cement content.

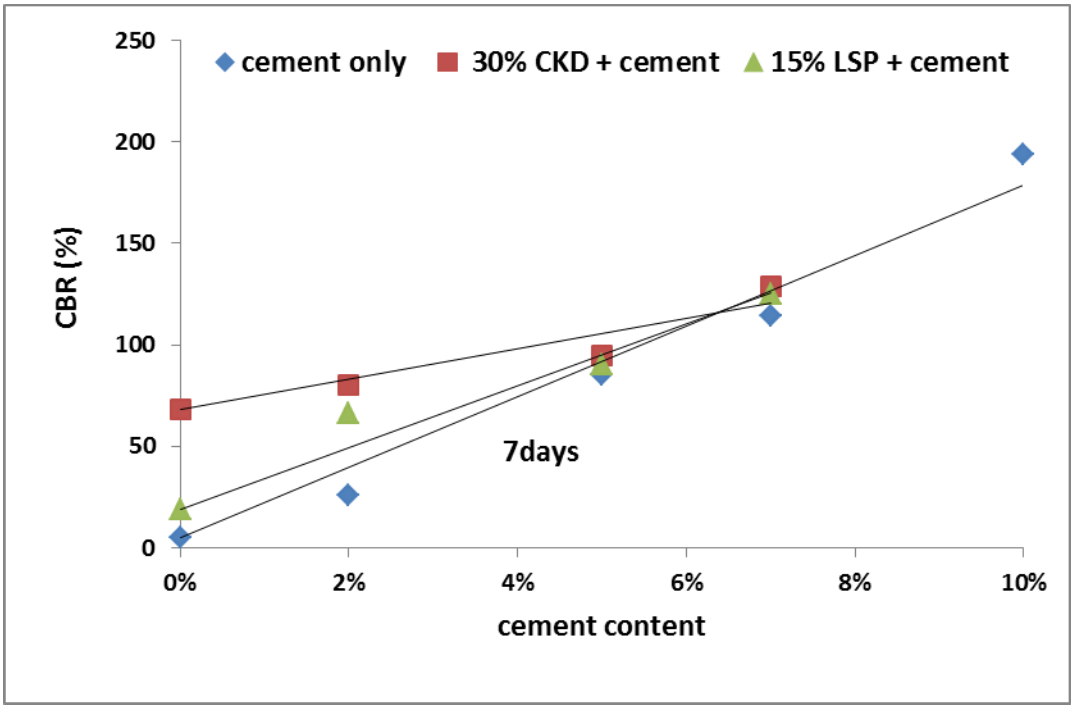


Figure 4.30: Effect of cement content on soaked CBR of S/S treated HOC and air-cured for 7 days

The best-fitted equations showing correlation between soaked CBR and cement content were obtained as follow:

Soaked CBR = 1738.8C+5 $R^2 = 0.97$ (For cement only)

Soaked CBR = 751.28C + 68 $R^2 = 0.91$ (For cement plus 30% CKD)

Soaked CBR = 1526.9C + 19 $R^2 = 0.95$ (For cement plus 15% LSP)

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Where

CBR= California bearing ration of HOC; sealed curing at 7days, (%).

C= Cement content (%).

$R^2$ = Correlation coefficient.

The above correlation of CBR were used to check whether the minimum requirement of soaked CBR for use of treated soil in pavement construction (i.e., > 50%) is satisfied or not for all combinations of stabilizers as listed in Table 4.10. The calculations of soaked CBR using the above equations for each dosage of the stabilizers given in Table 4.10 showed that the CBR requirement is satisfied in each case.

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# CHAPTER 5

# CONCLUSIONS AND RECOMMENDATIONS

This research was conducted for evaluating the performance of S/S treatment of three soils contaminated with three concentrations of oil. Cement, CKD, EAFD and LSP were used as alternative stabilizers. Based on the findings of this research work, following conclusions and recommendations were drawn:

i. The S/S treatment of oil-contaminated soils with cement, CKD, EAFD and LSP stabilizers showed an improvement in the geotechnical properties.

ii. The beneficial effect of the S/S treatment is more with increase in the curing time, especially at higher dosages of cement

iii. 30% CKD plus 2, 5 and 7% cement as binder performed better than the cases of cement alone (2, 5 and 7%). However, the improvement in performance due to CKD is marginal.

iv. 15% LSP plus 2, 5 and 7% cement as binder performed better than the cases of cement alone (2, 5 and 7%). However, the improvement in performance due to LSP is marginal

v. None of the 20% EAFD plus 2, 5 and 7% cement was found to be suitable for the effective stabilization of high oil contaminated soil.

vi. The S/S treated soil satisfied the environmental safety criteria as per the EPA standards.

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vii. Optimum dosages of stabilizers needed for utilization of treated HOC soil in sub-base courses of rigid and flexible pavements are recommended as follows:

| **Stabilizer** | **For use as sub-base material in rigid pavement (minimum 7-day UCS required: 1380 kPa)** | **For use as sub-base material in flexible pavement (minimum 7-day UCS required: 1725 kPa)** |
|---|---|---|
| Cement only | 5.5% cement | 6.9% cement |
| Cement + 30% CKD | 1.8% cement + 30% CKD | 4.3% cement + 30% CKD |
| Cement + 15% LSP | 4.7% cement + 15% LSP | 6.0% cement + 15% LSP |

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