Leachability of Immobilize Hydrocarbon Waste in Zeolite Cement

By

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Chemical Engineering Programme
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Approved by,

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(DR ASNA BT MOHD ZAIN)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

MAY 2014

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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MUHAMMAD ZAHID BIN ZAZARIN

ABSTRACT

Currently, over 80% of hazardous wastes are from industrial processes and that includes hydrocarbon waste. Hydrocarbon (HC) wastes are complex substances consisting of free oil, oily sludge, solvents, emulsified oil, solids, and water that can either originate from crude oil stock or processed streams. Hydrocarbon waste is highly volatile and is dangerous to the environment. Therefor the solidification/stabilization (S/S) technology is applied in the treating of hydrocarbon waste to observe its capability in treating hydrocarbon waste. The scope of the research covers waste which consists a high amount of hydrocarbon residue from the petroleum refinery in Kerteh, Terengganu. The case study of this project is to obtain the best mixture ratio of cement to water (C/W) ratio, cement to sludge (C/S) ratio and cement to zeolite binder (C/B) ratio by testing its compressive strength and also study the effects of zeolite on the porosity, permeability, leachability and total oil and grease content of the immobilized hydrocarbon, (HC) waste in the cement. The technology of solidification and stabilization must prevent uncontrolled releasing of bounded harmful components in immobilize hydrocarbon waste into the environment even under conditions of long exposure to the action of possible agents such as atmospheric conditions and other aqueous electrolytes. The quantities of harmful components released into the environment through the rinsing processes must not exceed the quantities allowed by standards and rules on taking care of harmful wastes is discussed in this report. The main results obtain were the compressive strength test of the cement which met USEPA standards for construction purposes. The highest compressive strength with admixture zeolite is 31 MPa with 10% zeolite composition and 40% sludge content in mixture. In addition the metal content and total oil and grease content of the cement mixture after leaching is well below the allowable standard which is the target. For TOG the reduction of concentration is from 36 ppm to 3 ppm, which is an approximate of 90% reduction of TOG content in the leachate compared to raw sludge. Furthermore the metal content in the leachate obtained are all under 1ppm. Zeolite is a natural occurring element and not harmful to the environment, therefor using it in the case study is well in line with the universities target to create a greener alternative in its inventions.

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CHAPTER 1

INTRODUCTION

1.1 Background Study

There are numerous methods and mixtures to form cement for construction used up till today. Not all methods and mixtures can be used for all forms of construction. Therefor it is necessary to study what type of mixture and methods are best use for its construction. This is the same when incorporating hydrocarbon waste into cement. Study and testing must be carried in order to mix hydrocarbon waste in cement for construction.

Before handling with hydrocarbon waste it is essential to understand what hydrocarbons are in general. A hydrocarbon is an organic compound consisting entirely of hydrogen and carbon. The majority of hydrocarbons found on earth naturally occur in crude oil, where decomposed organic matter provides an abundance of carbon and hydrogen which, when bonded, can catenate to form seemingly limitless chains. Hydrocarbons are burnt and the energy released in this way is used to turn water in to steam, which is used to turn a turbine that generates energy.

In an ideal reaction the waste would be only water and carbon dioxide but because the hydrocarbon is not pure or clean there are often many toxic by-products such as mercury and arsenic. Also, incomplete combustion causes the production of carbon monoxide which is toxic because it will bind with haemoglobin more readily than oxygen, so if it is breathed in, oxygen cannot be absorbed, causing suffocation. Incomplete combustion also has a by-product of carbon in the form of soot (Hydrocarbon, 2014). Those are the main reason why hydrocarbon waste must be treated and not be given freedom to be released into the atmosphere. In addition burning of hydrocarbon as a method to dispose of its waste may cause more harm to the environment.

To overcome this, solidification and stabilization technology comes in place. Solidification/stabilization (S/S) is typically a process that involves the mixing of a waste with a binder to reduce the contaminant leachability by both physical and chemical means to convert the hazardous waste into an environmentally acceptable waste form for land disposal or construction use (Malviya and Chaudhary 2006). “Stabilization” refers to techniques that chemically reduce the hazard potential of a waste by converting the contaminants into less soluble, mobile or toxic forms (Roger and Caijun 2005). “Solidification” refers to techniques that encapsulate the waste, forming a solid material, and does not necessarily involve a chemical interaction between the contaminants and the solidifying additives (Jeffery, Lawrence et al. 1995). The technology is mostly applied in segments that immobilizes soils or sludge which contain one or more metal contaminants. High volumes of waste that are difficult to treat using other using existing technologies are recommended to apply this technique.

The technology though is affected by certain factors that have to be taken into consideration before proceeding further into the implementation stage. One of the criteria involved is the presence of admixtures in the cement based matrix. The presence of admixtures may help to improve the immobilization of specific contaminants which in this study case, hydrocarbon waste. The efficiencies of the encapsulation of the waste sometimes can be enhanced with the addition of additives. Certain existing admixtures proved its efficiencies in improving the cement physical or chemical behaviour which results in better outcome. However, the application of admixtures under this technology is still under study. Zeolite is generally applied as a replacement material for binder as it exhibit similar behaviour as a cementing material (Roger and Caijun 2005). Generally, zeolite mixed with Portland cement has many advantages including increase in viscosity, preventing phase separation, acting as pozzolan, binding additional water, decreasing the pore pH, adsorbing metal ions, and sometimes results in retarding the setting time of the cement (Trussell and Spence 1994). The application of zeolite generally results with cement matrix with increased strength and increased durability in tests such as freeze-thaw and wet dry resistance (Shi, C. and A. Fernández-Jiménez , 2006). However, with the combination of hydrocarbon waste in the cement mixture, the properties of the zeolite might be altered which may result in better or underperforming S/S cement matrix.

1.2 Problem Statement

Hydrocarbon waste is a huge concern to the environment and the community as it is a harmful substance if absorbed into the soil and released to the atmosphere. It originates from crude oil refineries and are classified under the nonspecific source wastes. The list of waste it falls under is called F list wastes and is specified under USEPA. For centuries waste and refuse, both nonthreatening and dangerous have been disposed of directly into landfills without significant concern about the environment and this is the same for hydrocarbon waste. Therefor in order to decrease the amount of hydrocarbon being dispose of carelessly into the environment there is a new theory or technology being used where the hydrocarbon waste itself is blended with cement to become a part of the cement mixture itself. This is also known as solidification/ stabilization technology. This is where the challenge begins. Test must be carried out to see if this method is applicable or will it diminish the identity of the cement as a building block material. In order for the cement, zeolite and hydrocarbon waste to bond together and achieve greater results a binder must be used.

1.3 Objective

The objective or aim of this project is:

i. To obtain the best mixture ratio of cement to water (C/W) ratio, cement to sludge (C/S) ratio and cement to zeolite binder (C/B) ratio by testing its compressive strength.
ii. To study the effects of zeolite on the porosity, permeability, leachability and total oil and grease content of the immobilized hydrocarbon, (HC) waste in the cement.

1.4 Scope of Study

To find appropriate literature related to leachability and the immobilization of hydrocarbon waste in zeolite cement. In addition the scope of study for this project covers waste characterization of the samples and also the cement matrix. Characterization of the waste was conducted for the physical and chemical reactivity can be observed. This is done based on the guidelines provided by the United States Environmental Protection Agency (USEPA). Characterization of the cement samples covers a few criteria the cement has to achieve in order to be used for construction. Therefor the author will first carry out study on the laws, regulation and standards required for a cement mixture. When the standards are known, test are carried out for the cement matrix. They are test for its unconfined compressive strength (UCS), leaching capability, metal content and pH value, permeability and porosity. Leaching test for the cement can be carried out using these methods of testing, crush block leaching, whole block leaching and flow through leaching dynamic. Another scope that the author will be covering is the basics of hydraulics cement system and the effect of admixtures on cement formation for solidification and stabilization. Waste characterization can be done by testing total solid, specific gravity, oil/grease content, metal content and moisture content of the waste. Most of the scope of the test that is being carried out is to observe the results with and without the use of Zeolite as a replacement binder inside the cement mixture. By doing so a comparison of the data can be made and a conclusion can be achieved. This is done by comparing the performance of the cement before and after the process of leaching.

CHAPTER 2

LITERATURE REVIEW

In order to obtain the objective we have to first look at the main goal of the project which is to combine hydrocarbon waste with cement and zeolite to make a new formulation of cement. The new cement mixture tested is to be used for construction and would have if not the same but even better quality compared to normal cement mixture. Only then the mixture with hydrocarbon waste be viable for daily usage. An immobilize waste which is acceptable for transportation and storage is prepared (M.W.Dean, 1997).

2.1 Hydrocarbon Waste

There are several waste treatment techniques already being carried out at this moment and they are pasty and liquid slurry waste treatment, soils decontamination, sludge dewatering, sludge drying, thermal desorption and hydrocarbon vapour oxidation. The benefits on carrying out these treatment are substantial volume and waste reduction, generally greater than 80%, recover oil and water for reuse, substantially reduces disposal and incineration costs, detoxification of solids to inert materials for safe disposal, recovery of valuable resources for recycle and finally flexible process capability for waste streams and throughput capacity (ART Engineering, LLC, 2014).

There has been no systematic attempt at characterizing the chemical composition of all the hydrocarbon waste types from a refinery operation. Codified in regulation at 40 CFR 261.31, the nonspecific source wastes which are also known as the F list waste consist of seven groups. One of the groups is known as the petroleum refinery wastewater treatment sludge. Waste classified under this group is from the gravitational and physical/chemical separations of oil/water/solids/ during the storage or treatment of process wastewaters and oily cooling wastewaters from petroleum refineries. This group can be further subdivided into 2 which are coded by EPA as F037 and F038 based on the sludge stage of separation which is either primary or secondary. Therefor the new cement mixture waste disposal method actually is trying to reduce waste and waste disposal cost both at the same time. If achieved the cement waste management is one that uses less energy and man power to be carried making it more preferable.

2.2 Cement Mix Design For The Study Of S/S Technology

Cement consists of three types of materials: limestone (CaCO3), aluminium silicate (clay or shale), and inert materials such as sand and gravel.

Pozzolans are "siliceous or siliceous and aluminous material, which in itself possesses little or no cementations valve but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementations properties" (ASTM, 2014).

In order to obtain the best cement mixture to study the properties of cement when mixed with waste the solidification and stabilization technology is used. The stabilization and solidification technology is a waste management technology which involves the process of mixing the waste with a binder to reduce the contaminant leachability both physically and chemically. Doing so it will indirectly convert the hazardous waste into an environmentally acceptable waste form, which goes to a landfill or used in construction (Bone, Barnard et al. 2004). Both terms carry different function towards the contribution in this technology. By changing its chemical state or by physical entrapment, stabilization attempts to reduce the solubility or chemical reactivity of a waste. The physical nature and handling characteristics of the waste are not necessarily changed by stabilization (Conner and Hoeffner 1998). On the other hand, converting the waste into an easily handled solid with reduced hazards from volatilization, leaching, or spillage is what solidification is about.

S/S technology was originally developed for treatment of nuclear waste in 1950s and later on different types of hazardous wastes. From around 1980s the technology also was applied for treatment of contaminated soil and sediments (Laugesen 2007). The development in the solidification was mainly originated from the low-level radioactive waste disposal. The regulations derived from this technology was slowly begun to be applied to other waste provided certain standards are met. The standards are achieved by applying few pre-treatments to prevent contaminant leaching, such as neutralization, oxidation/reduction, physical entrapment, chemical stabilization and binding of the stabilized solid into a monolith.

2.3 Characteristic of Zeolite Cement

Zeolites are naturally-occurring porous aluminosilicate minerals that impart improved hydraulic cementing properties when added to cement and concrete, giving them improved strength.

Natural zeolite can be used to prepare lightweight concrete for construction. Its porous silicate structure makes zeolite much lighter than sand and provides increased volume per tonne with similar hardness and strength. Zeolite is free from clay (clay reduces overall strength of concrete) and zeolites' porous structure holds moisture thus facilitating more rapid curing of the concrete (Zeolite and Concrete, 2014).

Zeolite also prevents alkali-silica reaction by decreasing the alkaline ion concentration in the pore solution in concrete via ion exchange, adsorption and pozzolanic reaction, therefore the formation of alkali silicate gel is eliminated and the interface is improved.

Zeolite can be heat treated to form lightweight cement. When heated it loses moisture. When mixed into a cement mixture it rehydrates and releases air. The 'foam' or 'air-entrainment' increases strength and decreases weight. This is why zeolite is used for this project.

2.3.1 Ion Exchange

The subsequent substitution of Si4+ by Al3+ leaves a net negative charge on the zeolite framework - known as Isomorphous Substitution. These areas of negative charge are therefore ideal sites for adsorption of exchangeable cations in solution (Stead K., Ouki S.K., Ward N.I., 2001). If there is no suitable site in the structure, or if it is already filled, the cations occupy the sites of water molecules upon ion exchange (Alpha-Omega, 2014).

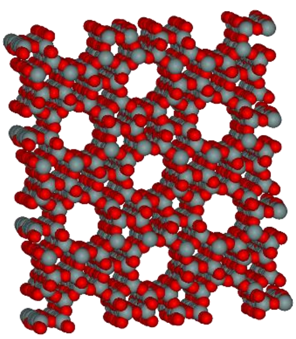


Figure 2.1 Microporous molecular structure of a zeolite (Zeolite, 2014)

2.3.2 Molecular Sieves

Zeolites also have the ability to exclude certain cations depending on their size; i.e. the size of the microporous channels and cavities within the zeolite structure can act to 'sieve' cations. Those cations that are bigger than the internal cavities are excluded from all or part of the internal surface of the zeolite, whereas, cations that can 'fit' into the internal structure can be exchanged (through isomorphous substitution or ion-exchange) onto the structure and become part of the zeolite framework. Hence, natural zeolites are renowned for their 'molecular sieve' properties. Ion exclusion phenomena are frequently observed in zeolites in which a particular ion is excluded from the exchanger because of its size (Cejka J., Van Bekkum H., Corma A., Schueth F., 2007). Ions can be partially exchanged because the volume the ion occupies may be too great, therefore occupying the intracrystalline space in the channels before complete exchange can be attained. Stead K. et al., (2001) detailed that zeolitic water molecules act as bridges for framework ions and exchangeable ions in large framework cavities. This shows the mobility of these cations within the framework.

CHAPTER 3

METHODOLOGY

3.1 Project Flow Chart

Figure 3.1 below explains the overall flow of the project. It consists of four main components which are done in a chronology starting with the literature review followed by experimenting and data collection and finally conclusion of the project. Each components have to be completed fully and clearly so that the project can run smoothly without any problems.

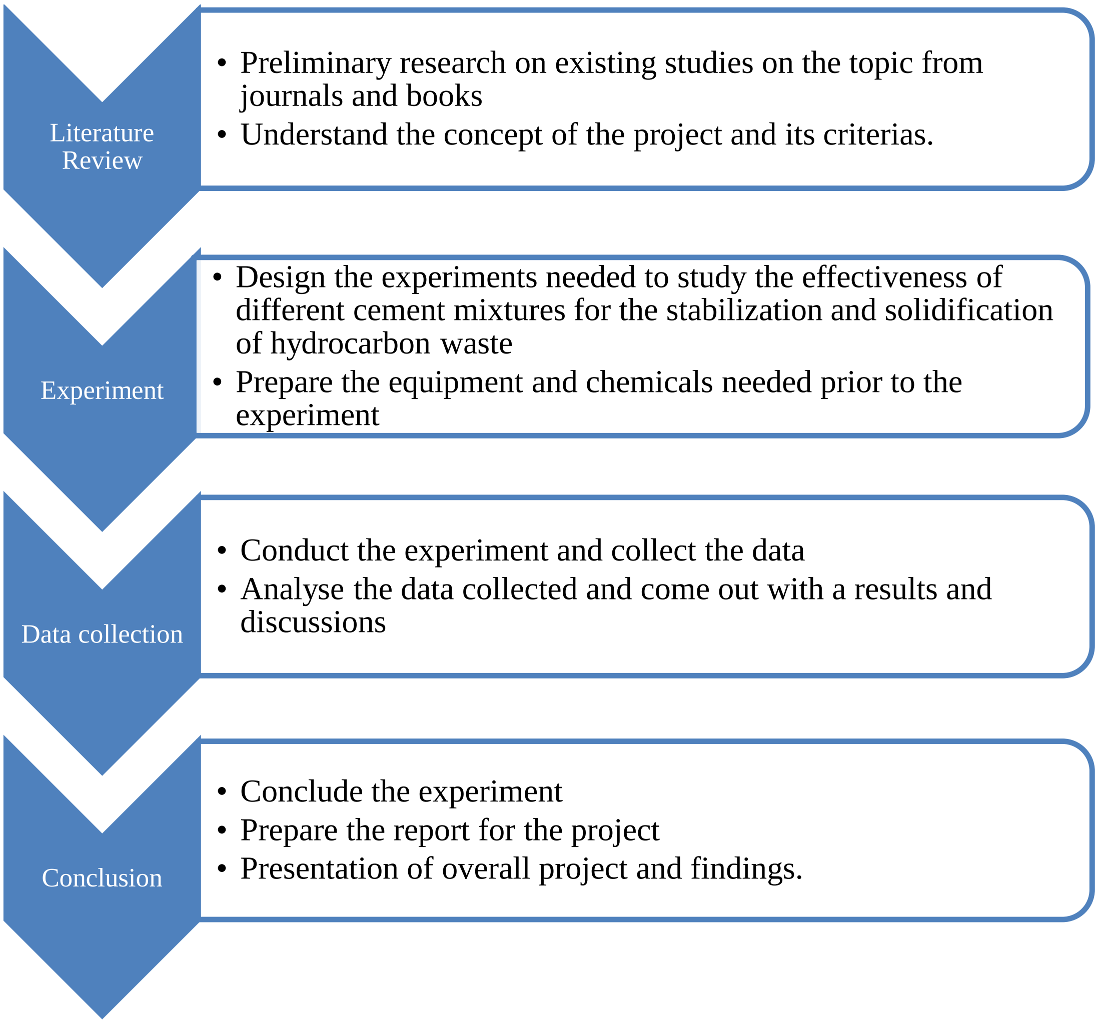


Figure 3.1 Project Flow Chart

3.2 Gantt Chart and Key Milestone

3.2.1 Final Year Project I Gantt Chart

Progress of the authors Final Year Project I is scheduled based on the given table below. It contains seven elements that has to be completed in the given time frame of 14 weeks. As shown in Table 1 all matters have been completed and all the milestones that was listed in the proposal was able to be achieved based on the dates assigned. For number 2 and number 5 from Table 1 the research work that has to be completed is based on finding pass literature material which is related to the author's project. In addition it is also to research the standards and types of test needed to observe and study the sample mixtures. Furthermore the research work that needed to be completed is making sure all the needed equipment and chemicals are available so that tests can be carried accordingly. Lastly a few sample of the cement mixture is to be prepared for the test listed in the experimental methodology (section 3.3) of the report.

Table 3.1: Final Year Project I Gantt Chart

No.	Detail Work/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1.	Selection of Project Topic														
2.	Preliminary Research Work														
3.	Submission of Extended Proposal														
4.	Proposal Defence														
5.	Continuation of Project Work														
6.	Submission of Interim Draft														
7.	Submission of Interim Report														

Completed Research Activities Based on Week Number

3.2.2 Final Year Project II Gantt Chart and Key Milestone

Table 2 describes the schedule that has been set for the second section of the authors Final Year Project. In Final Year Project II more studies will be carried out and all the tests planned out in Final Year Project I will be done based on the time frame given.

Table 3.2 Final Year Project II Gantt Chart and Key Milestone

No.	Detail Work/ Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1.	Experimentation Continuation and Analysis	■	■	■	■	■	■	■	■	■	■				
	i. Cement Based Matrix Test	■	■	■	■	■	■	■							
	ii. Compressive Strength Test	■	■	■	■	■	■	■							
	iii. Permeability & Porosity Test						■	■	■	■					
	iv. Leaching and Leachate Analysis						■	■	■	■					
	v. Characterization of hydrocarbon waste						■	■	■	■					
2.	Submission of Progress Report								●						
3.	Results and Discussion Summarization								■	■	■				
4.	Pre-SEDEX											●			
5.	Draft Report Submission												●		
6.	First Dissertation Submission (Softbound)												●		
7.	Technical Paper Submission													●	
8.	Oral Presentation														●

■ Ongoing Process ● Suggested Milestone

3.3 Experimental Methodology

For this project to move forward a series of test must be carried out in order to see the overall results in a more systematic manner. This is because all the test are interrelated to the objective of this project which is to obtain the best mixture ratio of cement for the immobilization of hydrocarbon waste in zeolite cement. Next carry out characterization of the hydrocarbon waste based on the series of test stated in Section 3.4 of the report. Then carry out solidification and stabilization tests on the best cement mix matrix ratio. Finally study the composition of the best cement mix matrix ratio and also the composition of the hydrocarbon waste. This will determine if the cement is able to immobilize the hydrocarbon waste and convert the hazardous waste into an environmentally acceptable waste form for land disposal or construction use (Malviya and Chaudhary 2006).

3.3.1 Cement Mixing Ratio Planned For Testing

The planned test for the 'Cement Mix Matrix' is based on the mixtures of all the mixing ratio from Table: 1. Cement matrix will test mix ratio of water and cement for Test 1. From Test 1 the cement to water ratio that has the highest compressive strength will be used for test two. The ratio between cement, water and sludge is as based on percentage of sludge to be added to the mix matrix ratio. For Test 2 only 5-20% of Sludge (Hydrocarbon Waste) is used because sludge has a binding limit with the cement. This is because at a certain point the amount of sludge may be in access and the cement will be too watery and will have a low compressive strength. When this occurs the prepared cement block for testing would not meet standards for a cement block to be tested. Again the highest compressive strength ratio from Test 2 will be used for Test 3. In Test 3 the binder or the admixture 'zeolite' is added to mix matrix ratio using percentage between 5-20%. There is also a limit for the amount of zeolite used as it will cause the cement to be to dry and have a low compressive strength. By completing each test the author will be able to observe the best cement mix matrix ratio and from there series of test can be carried out to observe the effectiveness of the cement in immobilizing hydrocarbon waste in zeolite cement.

Table 3.3 Cement to Water (C/W) Ratio, Cement to Sludge (C/S) Ratio and Cement to Zeolite Binder (C/B) Ratio Based On Test Numbering

Test 1: Cement and Water	Test 2: Cement, Water, Sludge (Hydrocarbon Waste)	Test 3: Cement, Water, Sludge (Hydrocarbon Waste) and Zeolite
Ratio	**Ratio**	**Percentage Zeolite%**
W/C	**S/C**	**-**
0.30		
0.35	40, 50 and 60	
0.40	40, 50 and 60	
0.45	40, 50 and 60	5, 10, 15 and 20

Additional data needed to justify Table 3.3 is located in Appendix III to Appendix VI in the appendices section.

3.3.2 Cement Mixing Procedure

Obtain properties needed for mix calculation (sludge density, solid content & water content). Calculate the ratio needed for the mixing based on mix calculation template prepared. Apply thin layer of oil (engine oil) onto the mould. This is to avoid the dried cement block sticking on the mould & to make it easier to be removed from the frame. Mix the cement, sludge, water and binder accordingly to type of sample being prepared. Carry out slump test using k-slump tester & pH test by using pH paper for the cement mixture sample. Place the mix evenly into the mould (layer by layer). Let the mixing (mould) dry in ambient conditions for approximately 24hours. Open the moulds after 24 hours and weigh mass and measure its dimensions for each block for day one for weight and dimension analysis. Place the blocks in a curing cabinet until desired testing period (day 1, 3, 7, 14 and 28) of the blocks compressive strength. Clean the mould by rinsing it with water first. Then soak the mould in 2.5% decontamination solution and 5% nitric acid, HNO_3 solution and leave over night when soaking in each solution. By doing so the dried cement on the mould will deteriorate making cleaning process easier.

3.4 Waste Characterization

Before test on cement matrix can be done characterization of the hydrocarbon waste (sludge) must be carried out so we can observe the results of before and after carrying out the test on cement matrix for different ratio of cement.

Methodology of test on hydrocarbon sludge sample for characterization are as follows:

Figure 3.2 Methodology of Waste Characterization

3.4.1 Total Dissolved Solid (TDS)

Total dissolved solids (TDS) is a measure of the combined content of all inorganic and organic substances contained in a liquid in molecular, ionized or micro-granular (colloidal sol) suspended form (Total dissolved solids, 2014). This analysis is applied to examine the total amount of solid material dissolved in solution. The standard applicable for this test is U.S. EPA Method 209B. The TDS level in leaching solution is applicable to track the degradation of S/S-treated waste solid or leaching of constituents from the sample.

3.4.2 Specific Gravity

Specific gravity is the ratio of the density of a substance to the density (mass of the same unit volume) of a reference substance. The reference substance is usually water. The measurement of specific gravity is for the purpose of the mixing calculation for the cement to sludge ratio. To obtain the specific gravity first the density of the hydrocarbon waste must be known. This can be measured using standard hands on method or the pycnometer. Certain pycnometer can even provide the specific gravity of a substance directly. Calculation for the specific gravity for both procedures mentioned above can be done using the formulas below respectively:

$$Specific\ Gravity\ at\ 4^{\circ}C\ for\ Procedure\ 1 = \frac{weight\ of\ sample}{weight\ of\ equal\ volume\ of\ water\ at\ 4^{\circ}C} = \frac{S - W}{R - W} \times F$$

$$Specific\ Gravity\ at\ 4^{\circ}C\ for\ Procedure\ 2 = \frac{weight\ of\ sample}{weight\ of\ equal\ volume\ of\ water\ at\ 4^{\circ}C} = \frac{(P - W)}{(R - W) - (Q - P)} \times F$$

Based on the temperature, T measured, derived the value of F from the tabulated temperature correction factor below:

Table 3.4 Temperature Correction Factor, F

Temperature (^{o}C)	Temperature Correction Factor
15	0.9991
20	0.9982
25	0.9975
30	0.9957
35	0.9941
40	0.9922
45	0.9903

3.4.3 Oil/Grease Analysis

The analysis is to measure certain constituents that may influence leachate. Aerobic and anaerobic biological processes might be disrupted with the presence of excessive amount of waste thus reducing the efficiency of the wastewater treatment itself. “Oil and grease” is a conventional pollutant under 40 CFR 401.16 and generally refers to substances, including biological lipids and mineral hydrocarbons that have similar physical characteristics and common solubility in an organic extracting solvent. According to U.S. EP SW-846 Method 9071b, this procedure helps to examine the total content of oil and grease in a sample. Method 9071 employs n-hexane as the extraction solvent with Soxhlet extraction and the results of this method are appropriately termed “n-hexane extractable material (HEM).” This analysis is crucial as oil and grease interfere with cement or pozzolan-based S/S treatment. This test must be conducted on the hydrocarbon waste as well as the leachate for ensuring the S/S technology does aids in stabilizing and immobilizing the hydrocarbon waste in the cement based matrix.

3.4.4 Metal Content

This test is carried out to observe the metal content of hydrocarbon waste. The final metal content after the process of S/S will be carried out to compare the metal content and observe if any metal leach through the cement after leaching process. The leachate obtained after 18 hours undergoes this test to examine the concentration of metals leached from the S/S treated waste. The metal content can be determined in accordance with U.S. E.P.A SW-846 Methods 6100, which is analysis by inductively coupled plasma atomic emission spectroscopy (ICP). Prior to testing, the leachate must be treated with appropriate digestion procedure U.S. EPA SW-846 Methods 3005, 3010, 3020, 3040 and 3050 which is done by treating with nitric acid.

3.4.5 Moisture Content

Moisture content express the amount of free water present in a moist sample. Under the S/S technology, it is necessary to run this procedure to determine the material handling properties and to determine whether pre-treatment is needed. Based on the amount of moisture content in the waste sample, the amount of additional water required for the S/S binder can be calculated.

3.5 Solidification/ Stabilization Of Cement Mixture

A series of test are proposed and carried out for solidification and stabilization of cement mixture. The presence of admixture in the mixture must be specialized to recognize its general properties and applications to justify its purpose or function in the cement based matrix. The physical nature and handling characteristics of the waste are not necessarily changed by stabilization. On the other hand, converting the waste into an easily handled solid with reduced hazards from volatilization, leaching, or spillage is what solidification is about. Test are carried out according to the series of characterizing method below for the cement mixture.

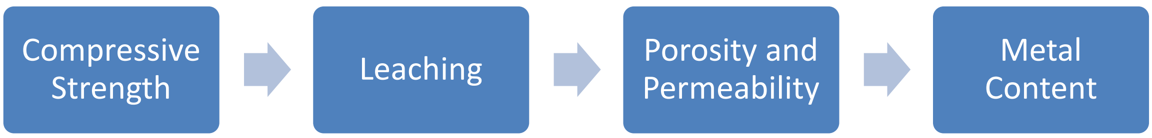


Figure 3.3 Methodology of Solidification/ Stabilization of Cement Mixture Test

3.5.1 Compressive Strength

This test measures the shear strength of a material without lateral confinement. Compressive strength of the cement that has been moulded is tested based on the days that it had been prepared. The standard applicable for this test would be according to ASTM C109. The test is carried out using an unconfined compression strength (UCS) testing machine. Settings for the particular type of cement blocks is first set. The block of cement is then place on the platform of the UCS according to the grid lines on the surface of the platform. Raise the platform up to desired height and lock the platform so it would not move during the testing period. The compressive strength value is determined by compressing the sample until it is deformed or broken. The compressive strength value can be observed from the display meter of the equipment. Average reading must be taken by repeating the procedures with 3 samples of the same mixture component.

The unconvinced compressive strength varies according to a number of factor such as x matrix ratio, surface structure of the cement block, aging period of sample block and also the positioning of the cement sample on the centre of the platform.

3.5.2 Leaching

Leaching is used to evaluate the leaching ability of metals, volatile and semi volatile organic compounds, and pesticides in waste categorized under RCRA. Based on the context of this project TCLP 1311 procedure are used to carry out the leaching process. Crush block leaching (CBL) is selected to simulate the leaching behaviour of the solidified waste. The simulation of the leaching behaviour is done in 2 different environments which is acidic and neutral. Crushed sample recovered from the compressive strength test will be used in this procedure. Samples crushed during the compressive strength test need to be recovered in a sealable sample bag to preserve its condition prior to the leaching test. Usually Day-28 sample is used for the leaching test as it had the most time for the cement mixture to bind together all of its components.

The crushed sample is then mixed with acetic acid as a leachant. About 80 g of solidified sample is weighed and placed into a number of plastic bottles depending on the number of sample tested. About 800mL of acetic acid, at a solid to leachant ratio 1:10, was added. The samples were mixed at 30±2 rpm in a rotary agitator at room temperature for 18 h. At the end of the extraction, the solid sample was let to settle overnight (V.A., Karamalidis A.K. and Voudrias, 2006).

3.5.3 pH and Metal Content using AAS

The crushed sample was extracted using an acetic acid solution (pH 2.88) in a volume with a weight equal to 20 times the weight of the sample. The extraction vessel must be rotated in an end-over-end manner at 30 rpm for 18 h. The leachate must be filtered through a 0.45-lm membrane filter or Whatsman no.41 filter paper to remove suspended solids and then divided it into two portions. One portion is for a pH measurement, and the other is for the determination of the metals present in the leachate by ICP-AAS. This test is carried out with accordance with U.S. E.P.A SW-846 Methods 6100, which is analysis by inductively coupled plasma atomic absorption spectroscopy (AAS). Each extraction was performed in triplicate, and the average value was reported to ensure the reproducibility of the data (Asavapisit, S., Naksrichum, S., Harnwajanawong, N.,, 2001).

Sample are prepared using a standard solution at 3 ppm, 5ppm and 10ppm. The AAS equipment is used to determine the zinc, copper, iron, nickel, chromium, manganese and lead content. Data obtained is used to compare the metal content of sludge and crushed cement blocks. The sample is efficient if the metal content from the leaching process is less than the sludge metal content.

3.5.4 Permeability

Permeability is the rate of flow of a liquid or gas through a porous material. It measures the rate at which water can flow though a material. In the oil and gas industry, this property is defined as the ability of porous material to allow fluid to pass through it. This property is crucial in determining the possible movement of the immobilized waste. Although encapsulated with cement, the presence of pores and its interconnection with other pores may increase the permeability of the matrix which easily enable leaching medium to leach away the improperly immobilized hydrocarbon waste. Therefore, the lower the permeability of the matrix, the better quality it is to act as a waste management method. The standard test procedures applicable for this segment would be classified under ASTM D5084 – 10.

3.5.5 Porosity

Porosity is the ratio of the volume of all the pores in a material to the volume of the whole. Porosity is also defined as the void space or pore spaces in solid structures which might be or not able to retain fluids. The porosity of a material, is the fraction of the volume of pore spaces over the total volume of the solid. Porosity of a specific material plays an important role in determining whether the immobilized waste can be leached when it comes in contact with any other external fluids. Based on this research case study, if the waste is not completely immobilized, then the chances of the waste being expelled by the cement is high due to its high porosity and large number of interconnected pores within the cement based matrix. The standard applicable for this segment would be according to the ASTM D4404-10 test standards. This test method covers the determination of the pore volume and the pore volume distributions of soil and rock by the mercury intrusion porosimeter method. The range of apparent diameters of pores for which this test method is applicable is fixed by the operating pressure range of the testing instrument.

CHAPTER 4

RESULTS AND DISCUSSION

This chapter discusses the results gathered from cement block samples prepared and tested for its unconfined compressive strength (UCS). Section 4.1 will be discussing on results for ‘Test’ 1. Test 1 is to study the compressive strength of the water to cement ratio. As explained earlier unconfined compressive strength test is used to determine whether the cement mixture is suitable to be used for construction material. As like all construction material the most important factor is of course its strength.

Results are obtained from research assistant Mr.Anas Khalid whom results are incorporated with the author’s project as the author’s project is one of the subsection of an even larger project that observes the use of different binders (admixture) in the S/S technology to overcome sludge hydrocarbon waste. All parameters are the same only the binding material are the different. Therefor the first test which is the control for the cement mixture can be taken and incorporated in the author’s project.

4.1 Mixing Calculation

The first results obtained from this research is calculation carried out to find the mixing calculation of the cement mixture. The density of the materials used were obtained from a device called a pycnometer. In addition moisture content analysis was carried out on the sludge samples to calculate the amount of water present in the sludge. Moisture content is crucial for mixing calculation for the determination of amount of water required to be added to the cement mixture to prevent dehydration of the mixture during curing in room temperature. Insufficient water in the mixing may lead to difficulties to handle and equipment malfunction as well as brittle properties of the cement block. The dry mass or total solid of the sludge must also be measured to estimate the amount of dry sludge required to mix with cement and binder to estimate the additional amount of water required. The test to determine the total amount of solid and moisture content in hydrocarbon waste (sludge) used was carried at once and the results obtained are as below.

Table 4.1: Total solid content and moisture content in hydrocarbon waste sample tested.

Test Sample No.	Empty Dish	Dish + Sample	Dish + Dried Sample	Total Solid	Moisture Content
1	2.2727	28.5413	3.6245	0.051461	0.948539
2	2.2669	28.3992	3.6965	0.054706	0.945294
3	2.2441	28.9845	3.3421	0.041061	0.958939
Average				0.049076	0.950924

Based on test carried out the total amount of solid observed to be in hydrocarbon sludge is approximately 5% of its total weight and a moisture content of approximately 95%.

Once all information is gathered for the total solid content and moisture content, the number of samples required and their dimension are determined for the volumetric estimation of the cement mixture required to be placed in the mould for the curing and testing procedures. Steps of calculation are included in the appendices section of the report (Appendix IX).

Overall, the mass of each component is tabulated as below:

Table 4.2 Mass for C/Sd = 40 and C/W = 0.45

Component	Mass
Cement	2.6042
Raw Sludge	0.8892
Zeolite	0.4596
Water	0.3756

The sample calculation showed can be computed using Microsoft Excel for better accuracy. The experiment will cover a wider range of cement to sludge ratio as well as cement to water ratio. The expected experiment ratios are as shown in the Table, 4.3, 4.4 and 4.5. The complete calculation for all the selected ratios is included in Appendices. The calculation was made by using Microsoft Excel spreadsheet. Once the mixing calculation is completed, the next thing to look into is the mixing procedure for the mixture.

Table 4.3 Proposed Set of Ratios for Cement + Water

Table 4.2 Proposed Set of
0.35
0.40
0.45

Table 4.4 Proposed Set of Ratios for Cement + Water + Waste Sludge

Cement to Sludge Ratio (C/S_d)	Water to Cement Ratio
40	0.45
50	0.45
60	0.45

Table 4.5 Proposed Set of Ratios for Cement + Water + Waste Sludge + Zeolite

Cement to Sludge Ratio (C/S_d)	Zeolite Composition (%F.A.)	Water to Cement Ratio (W/C)
40	5%	0.45
	10%	
	15%	
50	5%	0.45
	10%	
	15%	
60	5%	0.45
	10%	
	15%	

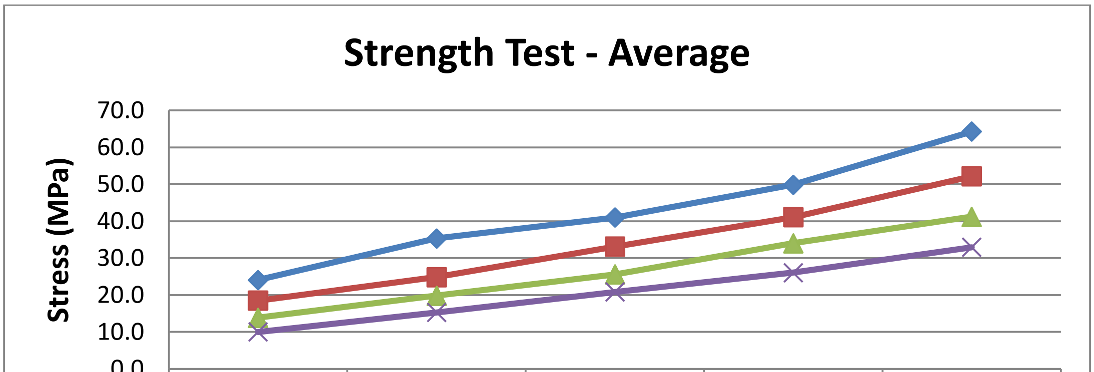
4.2 Mixing

The sludge needs to be homogenized using the electric mixer for approximately 2-3 minutes. During mixing, add cement slowly followed by the addition of the zeolite. Leave the mixture to homogenize for 5 minutes. Slowly add distilled water to the electric mixer to further homogenize the mixture. Once the homogenous slurries can be observed, quickly add the slurries into the 50 x 50 x 50 caste mould for the UCS test. The moulds are then left to harden at room temperature (25 ^{o}C to 33 ^{o}C) with 92% relative humidity for 24 hours. Cover the mould with Perspex cover to prevent further excessive loss of water from evaporation. After 24 hours, the moulded cubes removed from its caste and must be kept in the curing chamber for further dry curing. Based on the unconfined compressive strength test for the entire sample, the optimized ratio will be taken from the data and further tested for other properties such as TCLP, metal content, porosity and permeability. Based on these properties, the research will be able to deduce the effect of addition of zeolite to the S/S cement matrix for waste management purpose. If proven successful, this technique can be certified as one of the promising waste management method rather than incinerating the hydrocarbon waste which results in consumption of energy and natural resources.

4.3 Unconfined Compressive Strength (UCS) Test For W: C Ratio

The objective of this test to observe the development of cement strength with different ratios of water to cement, cement to sludge ratio as well as cement to binder ratio. The optimized ratio can be determined from the strength growth curve to further study the characteristics of the stabilized and solidified cement matrix.

Section 4.4 for chapter 4 is the results and discussion carried out for the first round of testing. Which is to obtain the control cement mixture ratio for water and cement only. The procedure in preparing the cement was done accordingly and tests for the compressive strength of the cement was done based on its given procedure. The results are as shown in Figure 5 below.



	Day 1	Day 3	Day 7	Day 14	Day 28
0.35	24.1	35.4	41.0	49.9	64.3
0.40	18.5	24.9	33.1	41.1	52.2
0.45	13.9	19.9	25.6	34.0	41.2
0.50	10.0	15.3	20.8	26.1	33.0

Figure 4.1 Compressive Strength of Water: Cement Ratio

Results obtained from figure 4.1 is based on Test 1 explained in section 3.3.1. Results from Figure 4.2 shows that water to cement ratio of 0.35 has the highest compressive strength from day 1 up till day 28. This shows that water to cement ratio 0.35 is best choice of cement to water ratio mixture to be used for Test 2 in section 3.3.1. Minimum stress that the load needed to withstand is approximately 17.2-20.7 MPa (Lamond, J.F. and Pielert, J.H., 2006) on day

28. This is the standard compressive strength that the cement mixture needs for construction use. Based on Figure 5 after day 7 are all the samples are cement mixture are acceptable by the S/S standard for cement. Due to the standard set by (Lamond, J.F. and Pielert, J.H., 2006) which states all values must be compared for its compressive strength on day 28 therefor the main comparison for the cement mixture compressive strength should be based on day 28. For day 28 cement mix ratio of 0.35 is the best choice as the compressive strength test showed that on day 28 the sample could withstand the highest amount of pressure, 64.3MPa. The worst cement to water mixture ratio is 0.50 on day 28 as it has the lowest compressive strength of 33.0MPa. Based on data obtained it shows that even at cement to water ratio 0.50 the compressive strength is accepted by the standard. But for this project the best compressive strength is considered and taken for further testing as the goal is to obtain the best cement mixture at the end of the project that can be used in the solidification and stabilization of hydrocarbon waste in cement. In the next section of the results the water to cement ratio of 0.50 is not considered as its compressive strength test shows the lowest strength and considered irrelevant if wanted to be further tested with the sludge to cement mixture ratio and also the cement to binder mixture ratio. Other than that at 0.50 water to cement ratio the mixture was to wet. This also being a factor in its compressive strength. Furthermore the hydrocarbon waste (sludge) is in a liquid form and may cause the cement mixture to be more watery and further losing the strength of cement block itself.

4.4 Unconfined Compressive Strength (UCS) Test for C: S Ratio

In section 4.4 the results obtain is also related to the compressive strength of the mix cement mixture. The variable manipulated that causes the variation in the data obtained is due to the difference in sludge to cement to cement ratio of the cement mixture. The sample mixture also included a manipulated variable of the water to cement ratio. This is to observe if there is a significant difference in the compressive strength of the cement mixture if there are more or less water mixed with the cement and sludge mixture. Other than that since the graph was indecisive in section 4.3 of the results, it was decided to carry out all 3 cement to sludge ratios together with the zeolite, to get a better picture on the difference on unconfined compressive when added together with an additive which is zeolite. The data was collected over a period of 28 days with an interval for data collection at day 1, day 3, day 7, day 14 and finally day 28. The results obtain are as shown below in Figures 4.2 to 4.4.

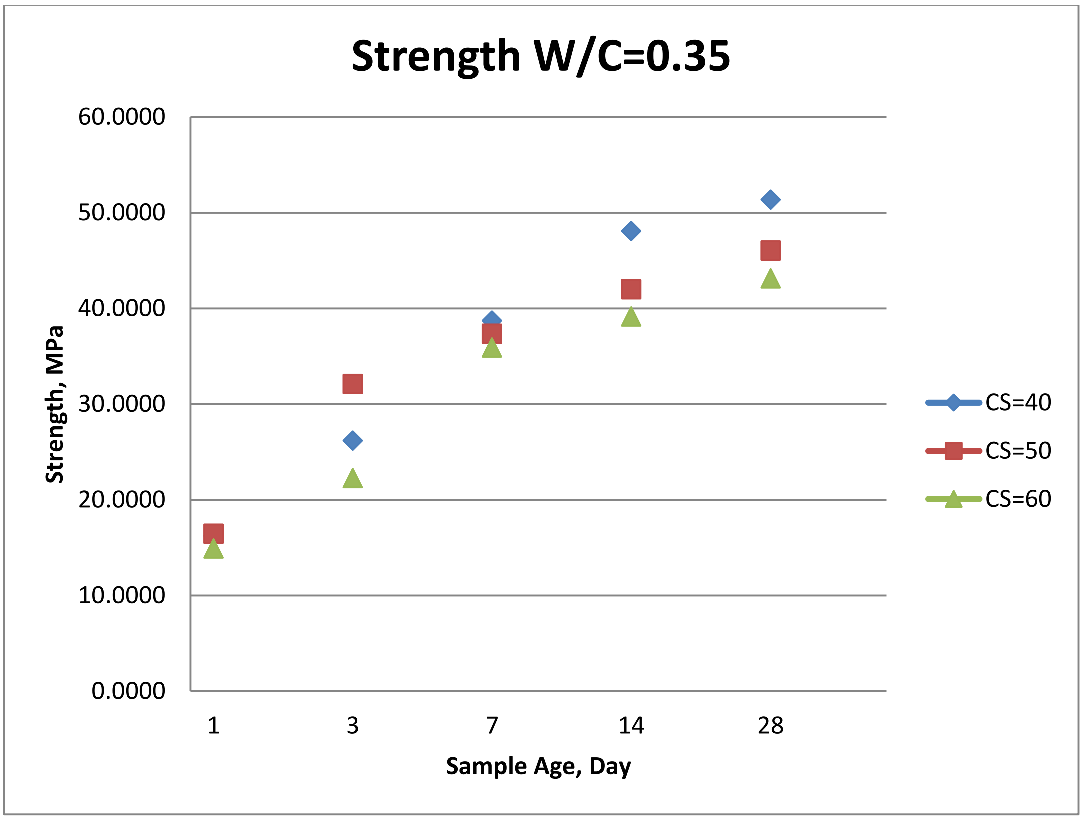


Figure 4.2 Compressive Strength of Cement: Sludge at W/C Ratio of 0.35

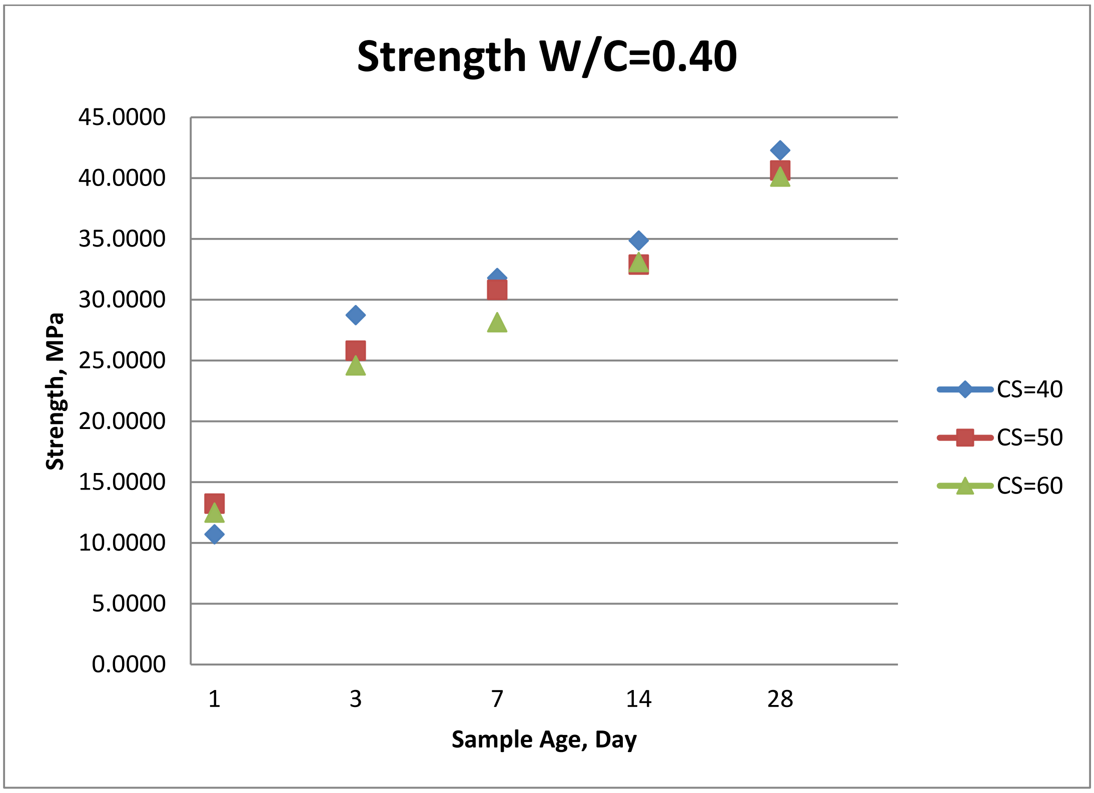


Figure 4.3 Compressive Strength of Cement: Sludge at W/C Ratio of 0.40

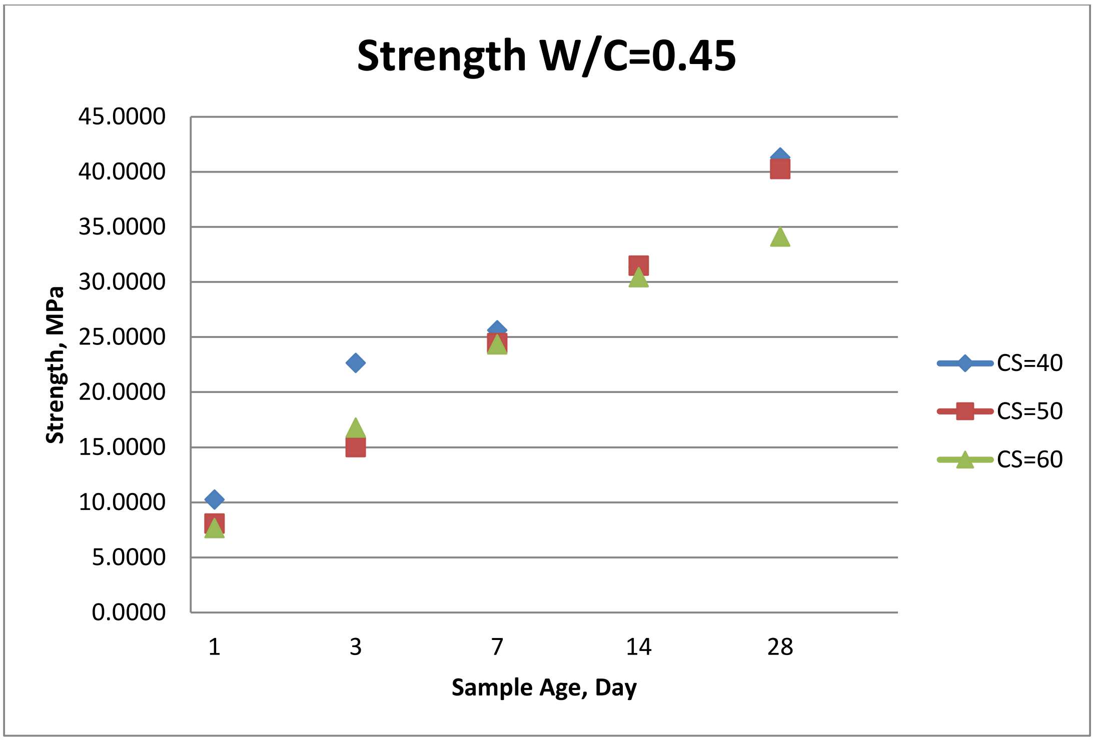


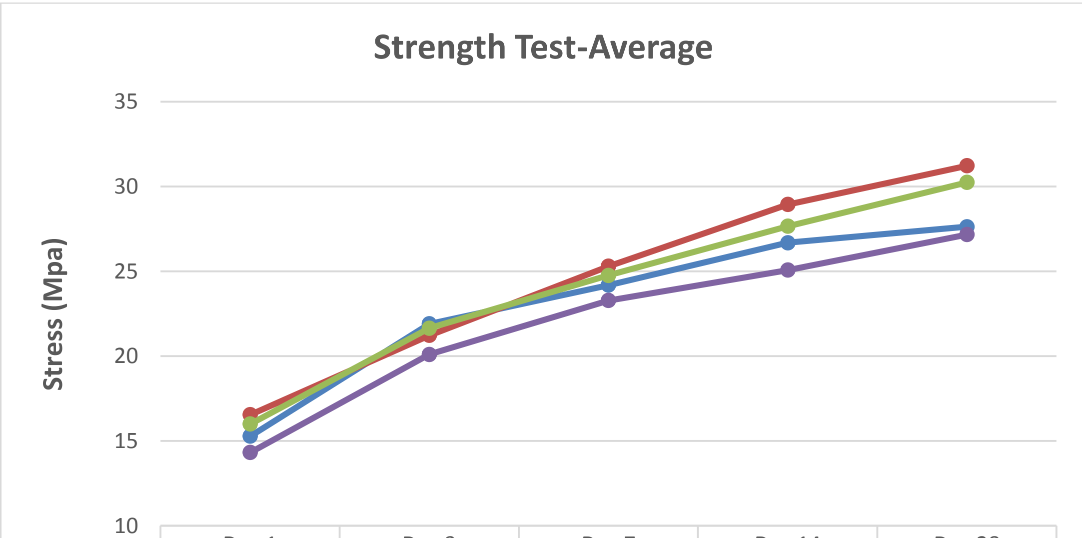
Figure 4.4 Compressive Strength of Cement: Sludge Ratio at W/C Ratio of 0.45

Based on Figures 4.2 to 4.4 it is observed that the unconfined compressive strength of the cement mixture is not effected greatly by the cement to sludge ratio in the cement mixture. The only visible difference is for W/C ratio of 0.35 shows a greater compressive strength compared to the 0.40 and 0.45 W/C ratio. This is due to a higher cement content in W/C ratio of 0.35 which makes the mixture stronger indeffinetly. In addition if observed carefully the lowest compressive strength is achieved by cement to sludge (C/S) ratio of 0.60. This shows that the larger the amount of hydrocarbon waste (sludge) the weaker the cement mixture becomes. This also means that there is a limit at how much sludge can be added in the mixture before the mixture loses its credibility to be used in construction. If more than the allowed sludge is added into the cement mixture the cement and zeolite binders will not fulfil its task in becoming a stabilization and solidification agent for hydrocarbon waste.

From this observation we can minimize the testing range for the cement to binder mixture ratio for samples needed to be tested for its unconfined compressive strength.

4.5 Unconfined Compressive Strength (UCS) Test For C: B Ratio

For this section of the results we are observing the results of the cement mixture unconfined compressive strength when zeolite which is the binder is added into the mixture. Zeolite is added to observe not only the changes on its compressive strength but also other characteristics that will be tested after all the compressive strength of the cement mixture has been obtained. These test were explained in the methodology (Chapter 3) section of this report. The samples tested were maintained at 0.45 W/C ratio and 0.40 C/S ratio. The fixed variables are chosen due to previous compressive strength tests results obtain from section 4.3 and 4.4 W/C content of 0.45 is chosen instead of 0.35 because of the property of the binder (Zeolite) which is a dry powder made the cement mixture too dry and unworkable if there is insufficient water available to mix all the component of the mixture itself. This information was obtained through initial tests carried out in section 4.1 and 4.2 of the results. The manipulated variable of this test is the percentage of binder (zeolite) added to cement mixture which varied from 5% to 20% with a difference of 5% in between each test. Samples were tested for its unconfined compressive strength test on day 1, 3, 7, 14 and 28. The results obtained are as shown in Figure 4.5 below:



	Day 1	Day 3	Day 7	Day 14	Day 28
0.05	15.28508326	21.90518082	24.18457953	26.68636402	27.62360954
0.1	16.54064139	21.22566294	25.29553132	28.93945543	31.21979904
0.15	15.99577066	21.64599752	24.75566722	27.65545449	30.23638096
0.2	14.31781869	20.10041008	23.27804825	25.0680891	27.16474662

Figure 4.5 Compressive Strength of C:B Mixture.

Based on **Figure 4.5** the author is able to observe there is a consistent rise in the average compressive strength of the cement mixture from day 1 to day 28 for all percentage of zeolite added into the cement mixture. Therefor the samples were reacting appropriately towards its aging process which was supposed to make the cement mixture stronger over a period of time. The minimum stress that the concrete block needed to withstand is approximately 17.2-20.7 MPa (Lamond, J.F. and Pielert, J.H., 2006) on day 28 and beyond it. This is the standard compressive strength that the cement mixture needs for construction use. All data shows a compressive strength of more than 20.7 MPa after day 28. From the average compressive strength test that was carried out on the samples it showed that sample with 10% of zeolite in it has the highest compressive strength test of 31.22 MPa after 28 days and at 20% zeolite concentration the lowest which is only 27.16 MPa. Due to the small amount of data the author is unable to determine whether the difference is due to analytical or systematic error or due to cement mixture property which again has its limits when considering all its cement mixing ratios. From data obtain the cement mixture compressive strength showed that it can still be used for construction even though its average unconfined compressive strength is lower than Test 1(64.33MPa) and Test 2 (51.99MPa) of the cement mixture test. The significant difference between the strength of the cement mixture for Test 1, Test 2 and Test 3 was caused by the number of ingredients used in each of the Test. This causes the cement which is the primary binder to lose its ability to solidify and maintain a high compressive strength rate.

After the completion of the compressive strength test all other tests on the characterization of the cement mixture can be carried out as planned.

4.6 Porosity and Permeability

For the porosity and permeability test, samples mixed with the waste petroleum sludge and zeolite will be analyzed for porosity and permeability. Matured crushed samples, with size not less than 4 mm in diameter was taken and measured for its weight before being tested using Mercury Porosimeter. The tabulated data for the samples taken for porosity and permeability test is as tabulated below.

Table 4.6 Porosity and Permeability Sample Data

Sample No.	Cement to Sludge Ratio (C/Sd)	Zeolite Composition (%F.A.)	Water to Cement Ratio (W/C)	Mass (g)	Density (g/cm3)
1	40	5%	0.45	0.76	2.426
2		10%		0.80	2.418
3		15%		0.82	2.405
4		20%		0.74	2.412

Permeability and porosity obtained is one of the most important key to the success of this study as it shows the capability and credibility of this method in encapsulating hydrocarbon waste. Both permeability rate and porosity of the sample should be low as hydrocarbon waste is considered to be a highly harmful substance if released into the environment without proper pre-treatment. The results are shown in Table 4.7 below.

Table 4.7 Porosity and Permeability Data

Sample	Without Compressibility Correction (CC)		With Compressibility Correction (CC)	
	Accessible Porosity	Inaccessible Porosity	Accessible Porosity	Inaccessible Porosity
1	15.43	1.82	11.48	6.21
2	17.15	3.34	12.97	8.44
3	19.94	6.82	14.82	11.94
4	22.46	9.12	18.35	15.43

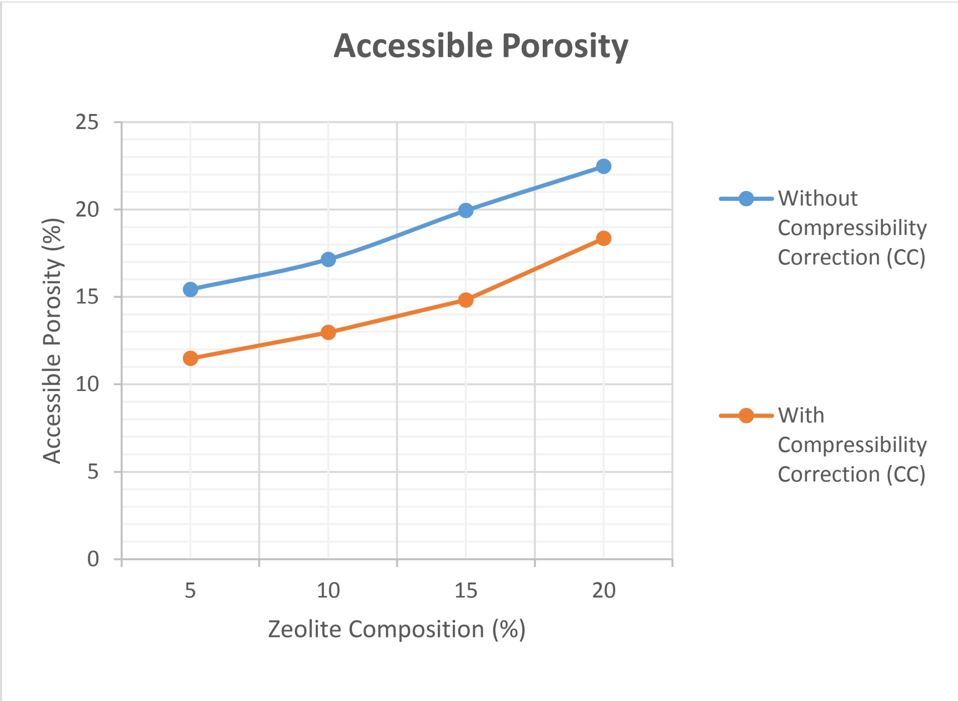


Figure 4.6 Comparison of Accessible Porosity and Zeolite Composition

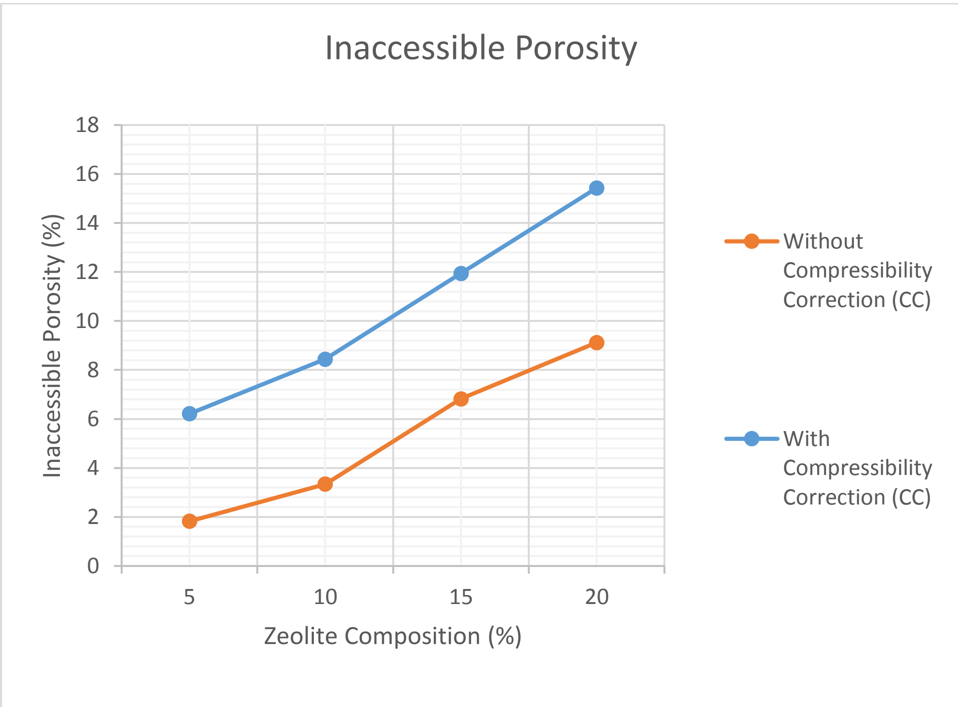


Figure 4.7 Comparison of Inaccessible Porosity and Zeolite Composition

It is observed by both results shown in the table and graph above the porosity of the cement mixture samples with zeolite of 5-20% compositions. It is observed that the accessible porosity of the cement samples at 5% zeolite concentration is the lowest with an accessible porosity of 15.43% without the compressibility correction and 11.48% with compressibility correction. The accessible porosity of the samples increases as the composition of zeolite concentration also increases. In addition the results shown in figure 4.6 for the inaccessible porosity shows that at low composition the inaccessible percentage is low and again rises as the zeolite composition rises. Therefor we can conclude that even though the inaccessible and accessible rates are co-reacting to the composition of zeolite content composition the percentage of accessible porosity is still significantly high for a cement mixture to be used for the purpose of construction. As a higher porosity will lead to a high permeability rate. This may cause the hydrocarbon waste immobilize in the cement mixture to escape or leach out of its mould over a period of time and this will be dangerous to the environment as hydrocarbon waste is highly volatile and dangerous to the environment.

4.7 Toxicity Characteristic Leaching Procedure (TCLP)

This test aims to observe the credibility of zeolite cement towards the solidification and stabilization theory through leaching process. Before carrying out this part of the tests the samples were tested before undergoing the leaching procedure. Test that were carried beforehand as a control were AAS and TOG test for the hydrocarbon. TCLP 1311 procedure were followed as a standard outlined by USEPA. Refer to Appendix V for the flowchart of the whole process. For the current week, samples were already leached as and waiting to be tested with Atomic Absorption Spectrometry for metal content. Standard solutions are already in place for the metal content test and the data and result obtained will be compiled and presented in the final report, together with the data from the porosity and permeability. The extraction fluid used in this set of experiment would be acetic acid with pH within 2.88 ± 0.05. The extraction fluid was selected based on the preliminary test done for the selection of extraction under the TCLP 1311 procedures. The leachates obtained were filtered using the Watman filter paper and kept in refrigeration in order to maintain the physical and chemical content of the solution. If not the results of tests carried out for AAS and TOG would be disturbed and inconclusive.

4.8 Atomic Absorption Spectroscopy (AAS)

Atomic Absorption Spectroscopy (AAS) test is used to observe the metal content available inside the solution after the process of leaching from section 4.7. By observing the metal content in the acetic acid and cement solution the author was able to observe whether or not any type of metal has leaked out of the cement mixture. The AAS tests are done using controlled solution that the specific metal content is known. From there the test is carried out as a comparison to the absorbance of the known sample to obtain concentration of that specific metal in the solution. This test is carried out for the four different concentration of zeolite additive to the cement mixture ranging from 5% to 20%.

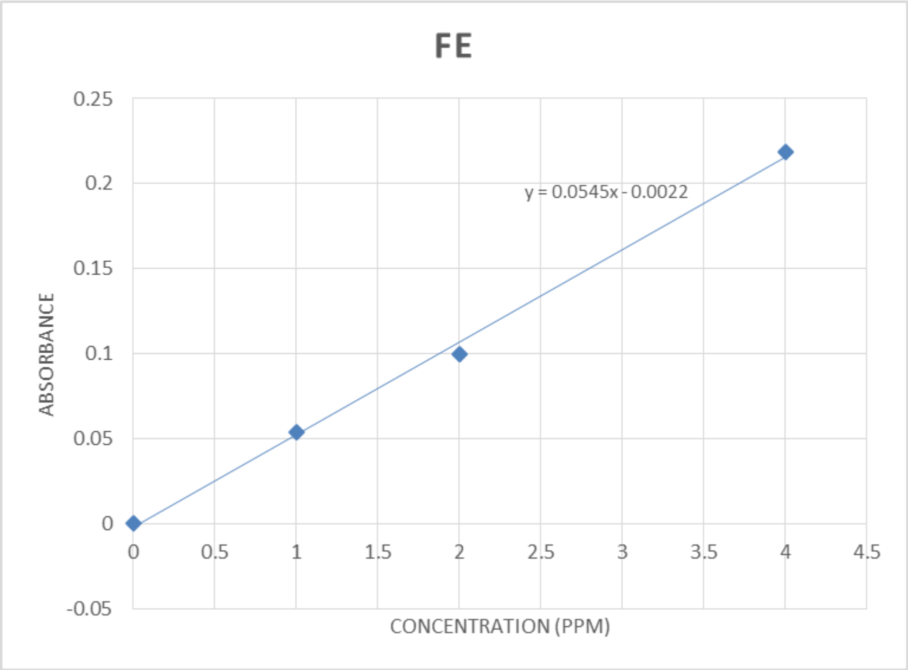


Figure 4.8 Controlled Iron, Fe concentration

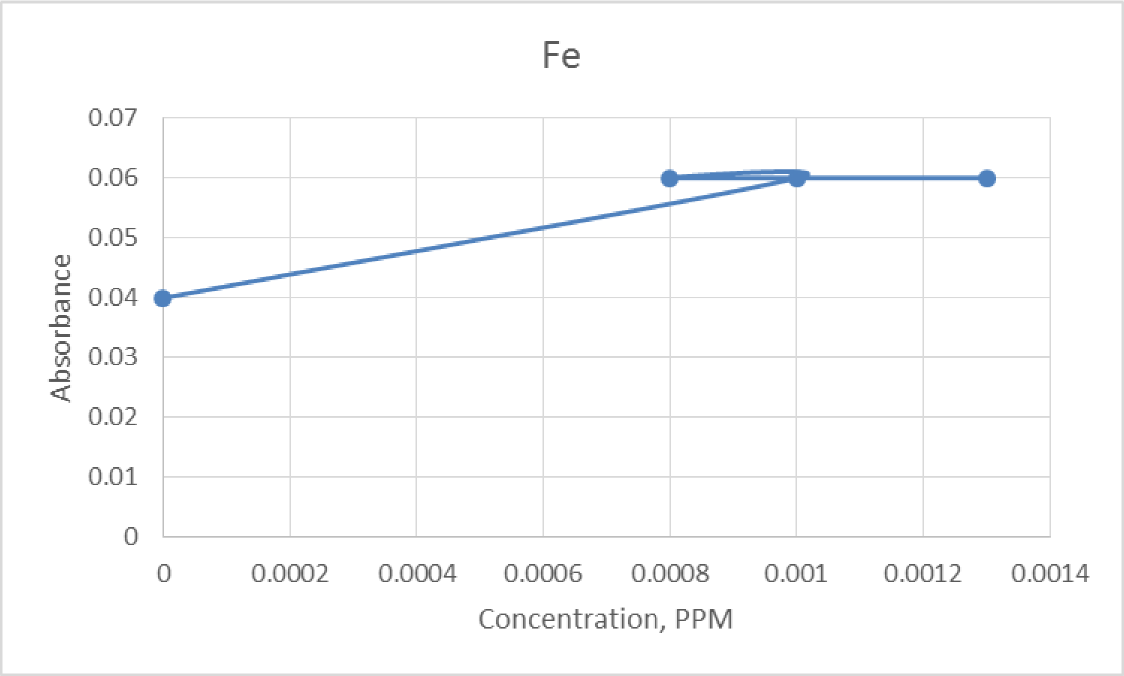


Figure 4.9 Sample solution Iron, Fe concentration

Based on Figure 4.8 and 4.9 it shows that the controlled substance has a higher Iron, Fe content compared to the sample solutions tested. Its absorbency is also considerably lower to that of the control.

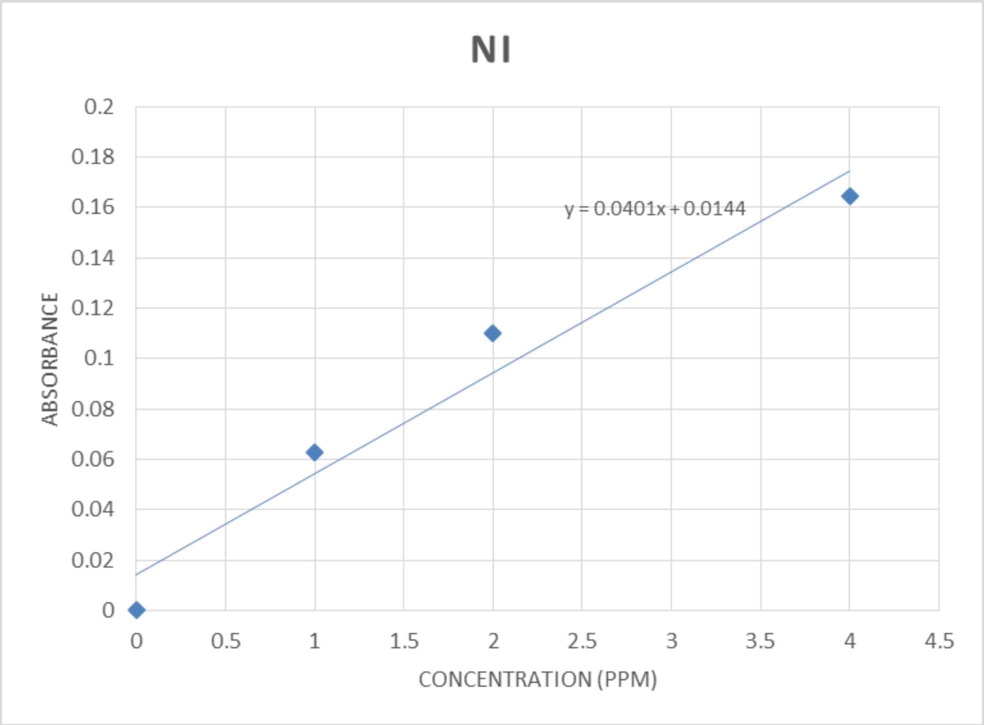


Figure 4.10 Controlled Nickel, Ni concentration

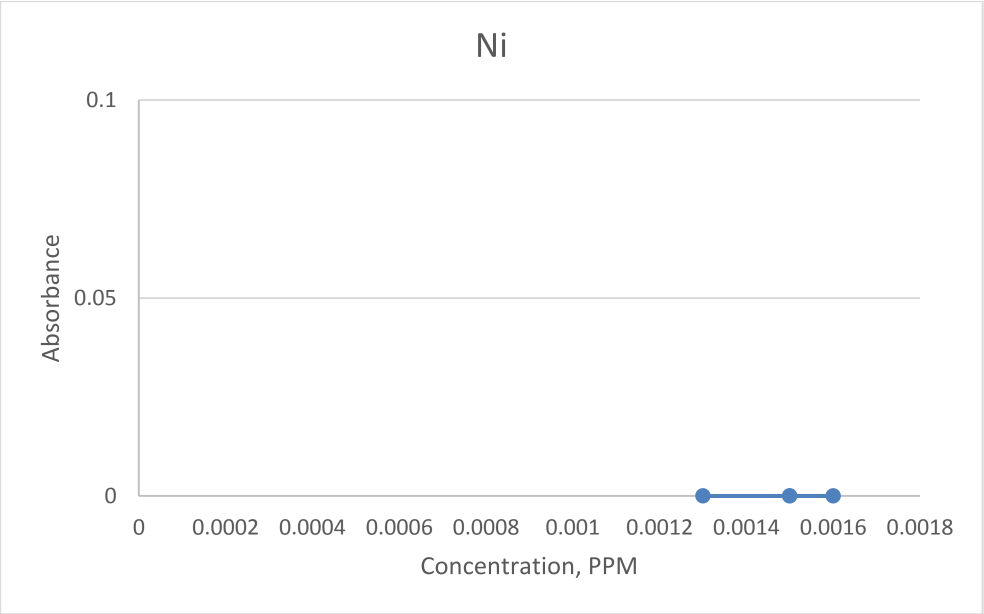


Figure 4.11 Sample solution Nickel, Ni concentration

Based on Figure 4.10 and 4.11 it shows that the controlled substance has a higher Iron, Fe concentration compared to the sample solutions tested. It also has a lower absorbance compared to the controlled solution.

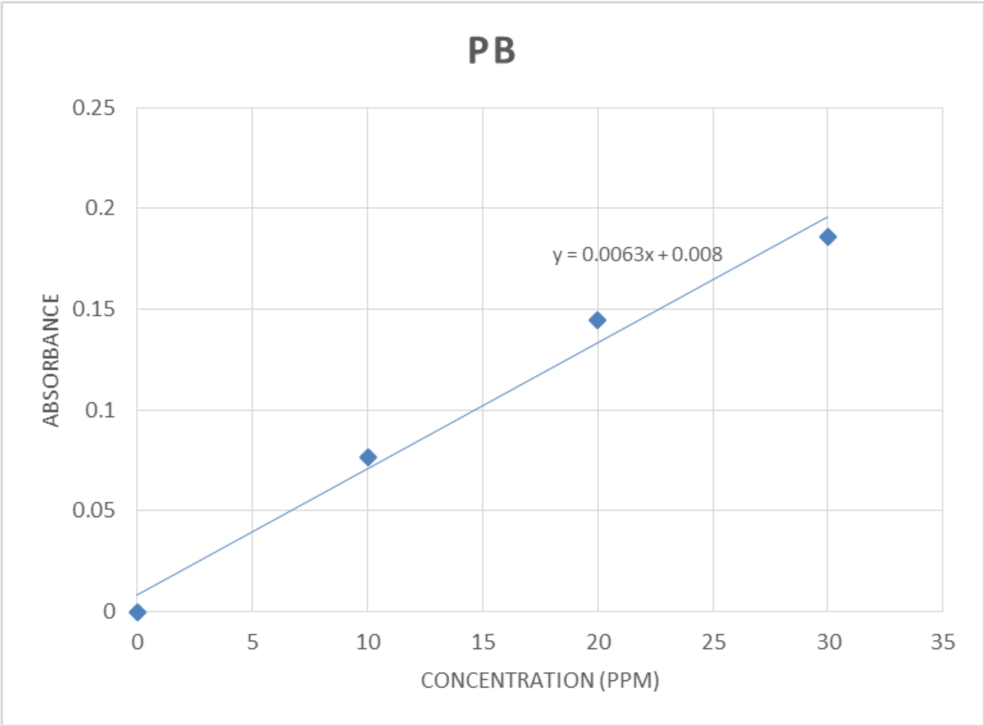


Figure 4.12 Controlled Lead, Pb concentration

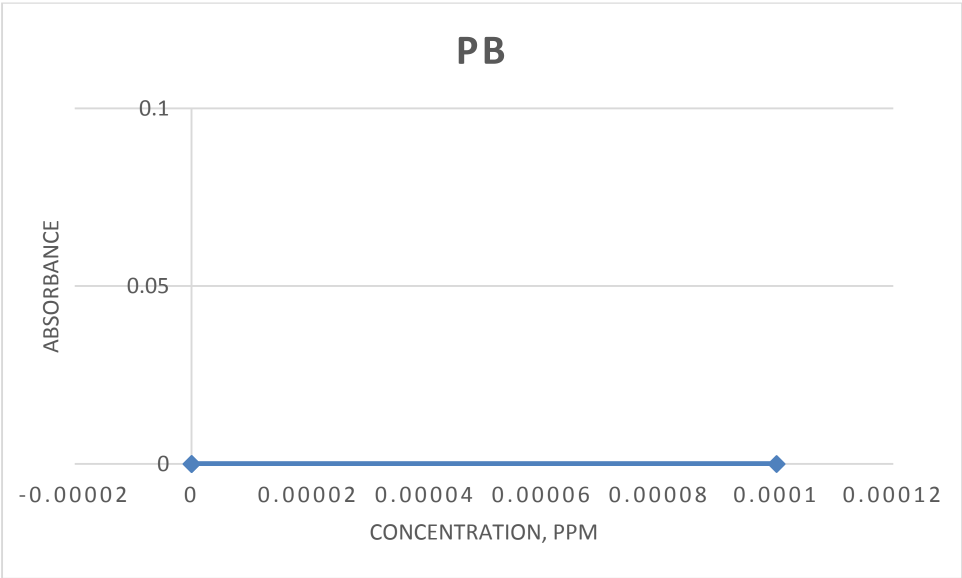


Figure 4.13 Sample solution Lead, Pb concentration

Based on Figure 4.12 and 4.13 it shows that the controlled substance has a higher Lead. Pb concentration compared to the sample solutions tested. It also has a lower absorbance compared to the controlled solution.

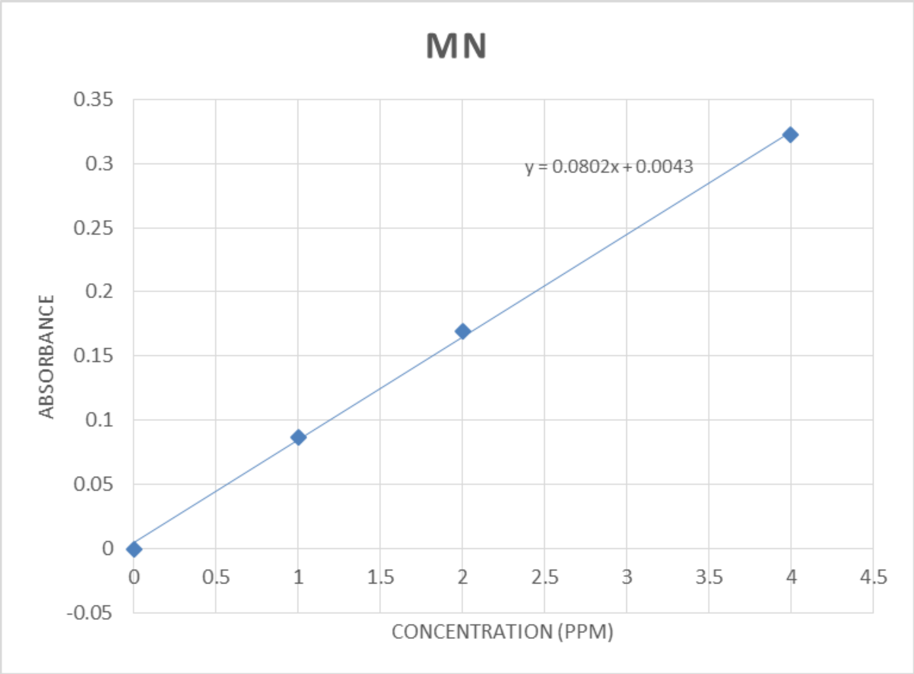


Figure 4.14 Controlled Manganese, Mn concentration

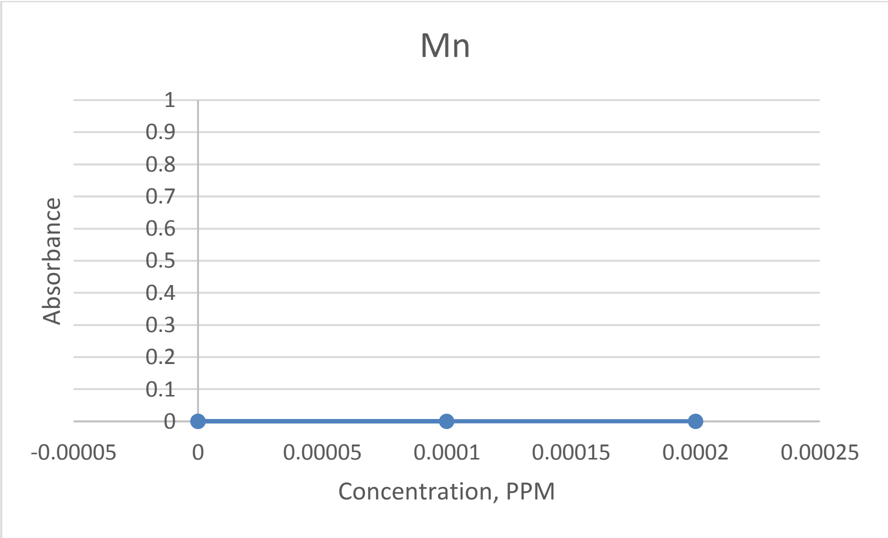


Figure 4.15 Sample solution Manganese, Mn concentration

Based on Figure 4.14 and 4.15 it shows that the controlled substance has a higher Mangenese, Mn concentration compared to the sample solutions tested. It also has a lower absorbance compared to the controlled solution.

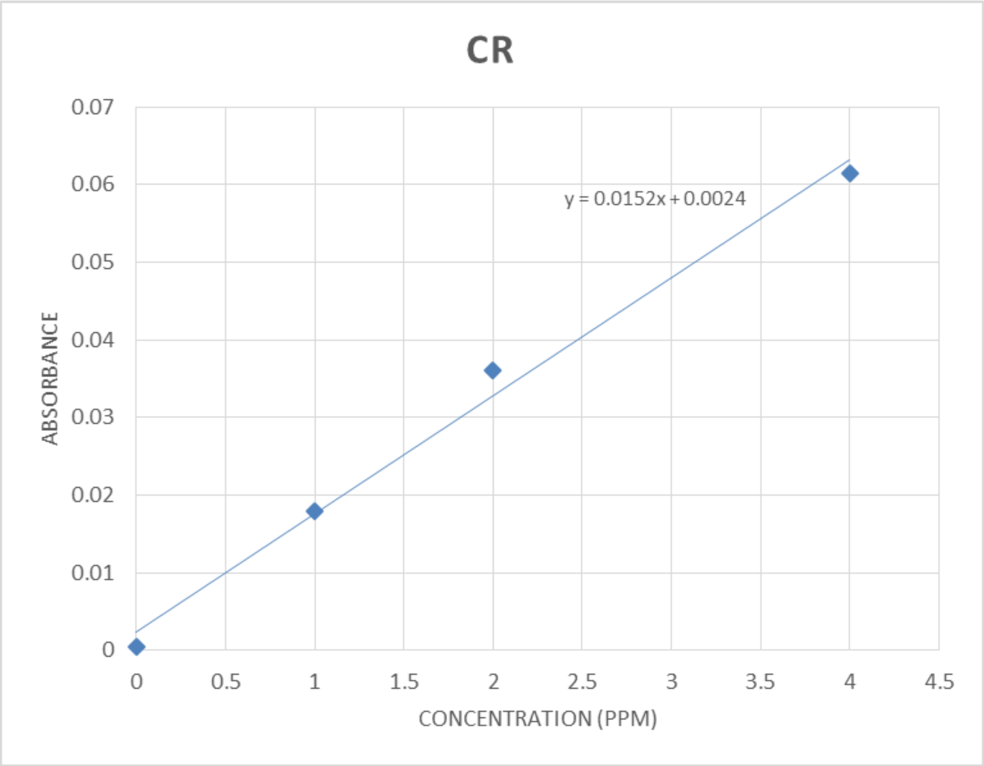


Figure 4.16 Controlled Chromium, Cr concentration

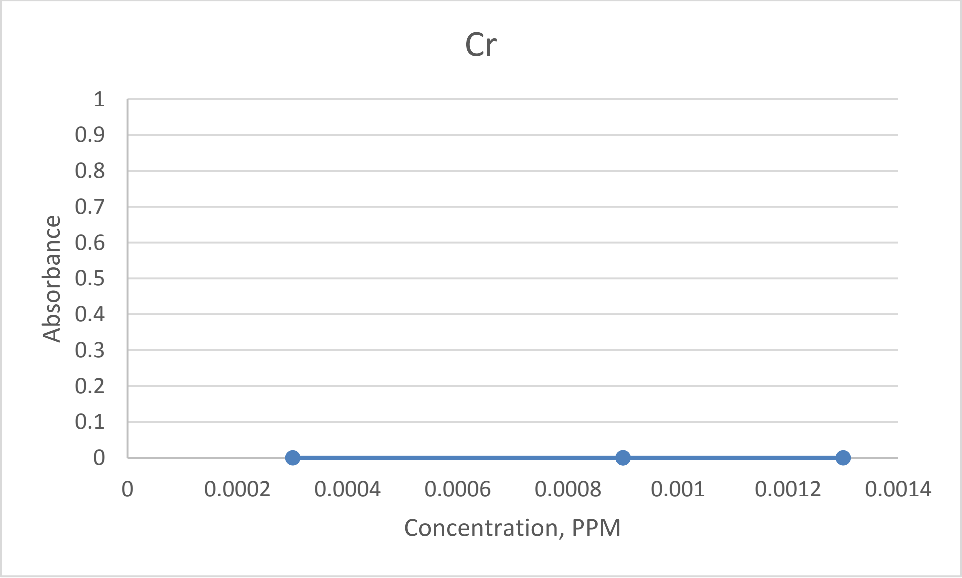


Figure 4.17 Sample solution Chromium, Concentration

Based on Figure 4.16 and 4.17 it shows that the controlled substance has a higher Chromium, Cr concentration compared to the sample solutions tested. It also has a lower absorbance compared to the controlled solution.

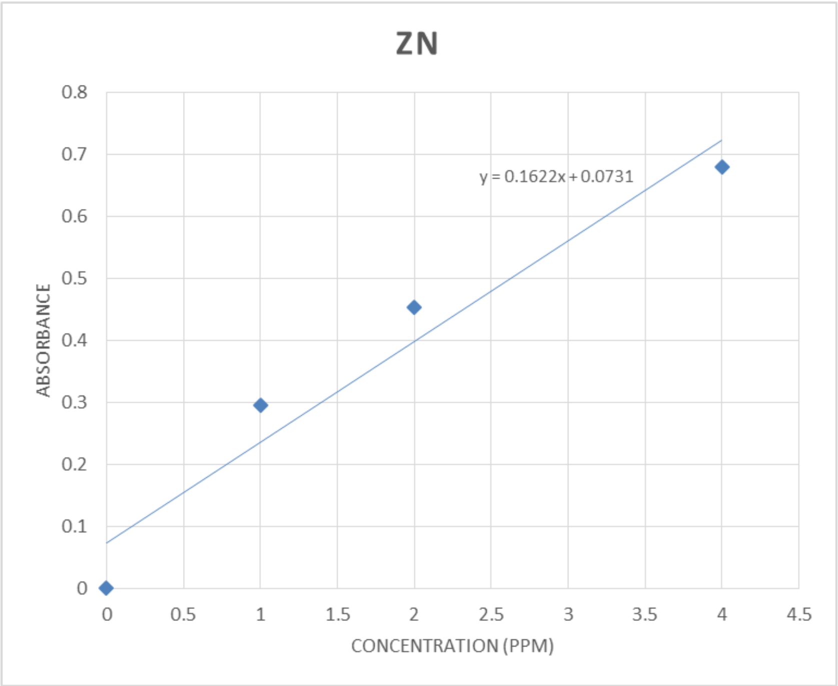


Figure 4.18 Controlled Zinc, Zn concentration

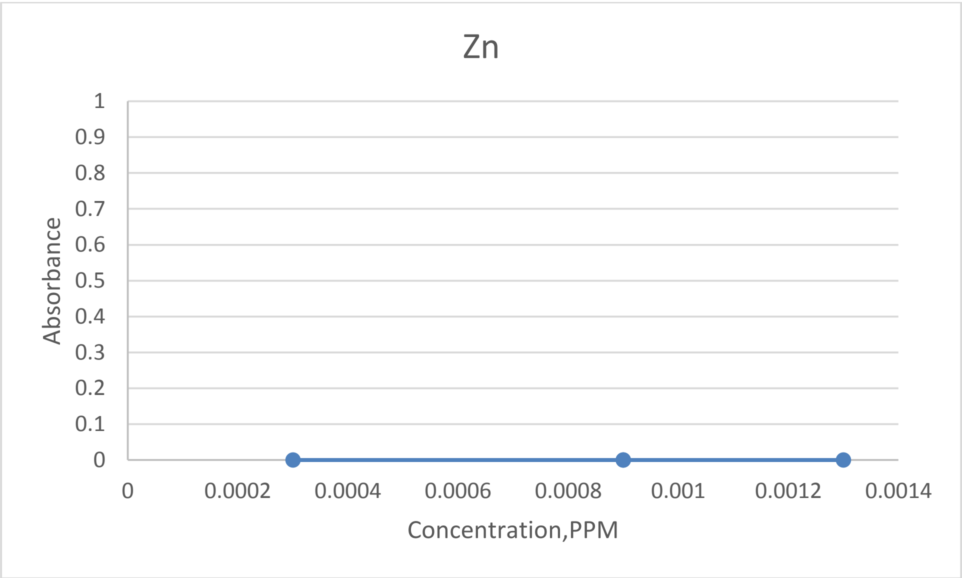


Figure 4.19 Sample solution Zinc, Zn concentration

Based on Figure 4.18 and 4.19 it shows that the controlled substance has a higher Zinc, Zn concentration compared to the sample solutions tested. It also has a lower absorbance compared to the controlled solution.

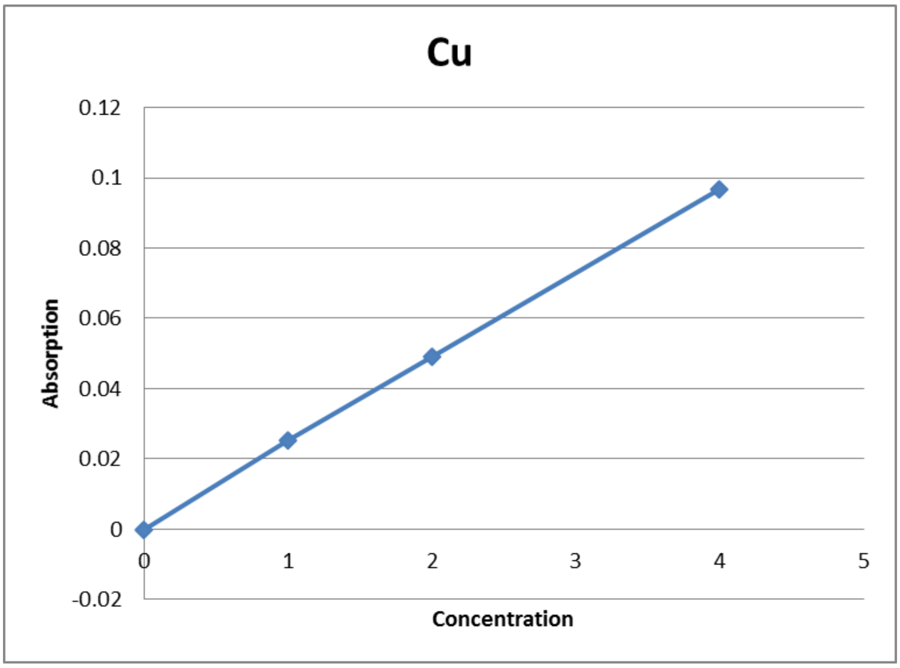


Figure 4.20 Controlled Copper, Cu concentration

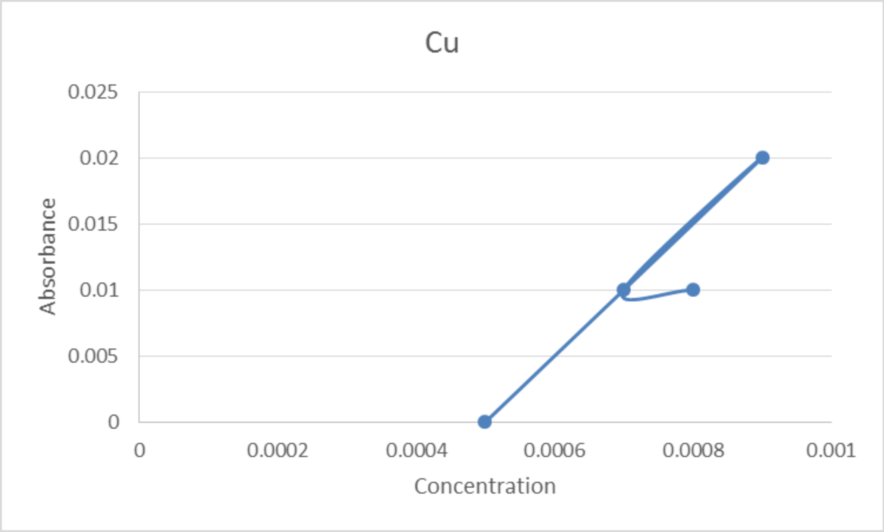
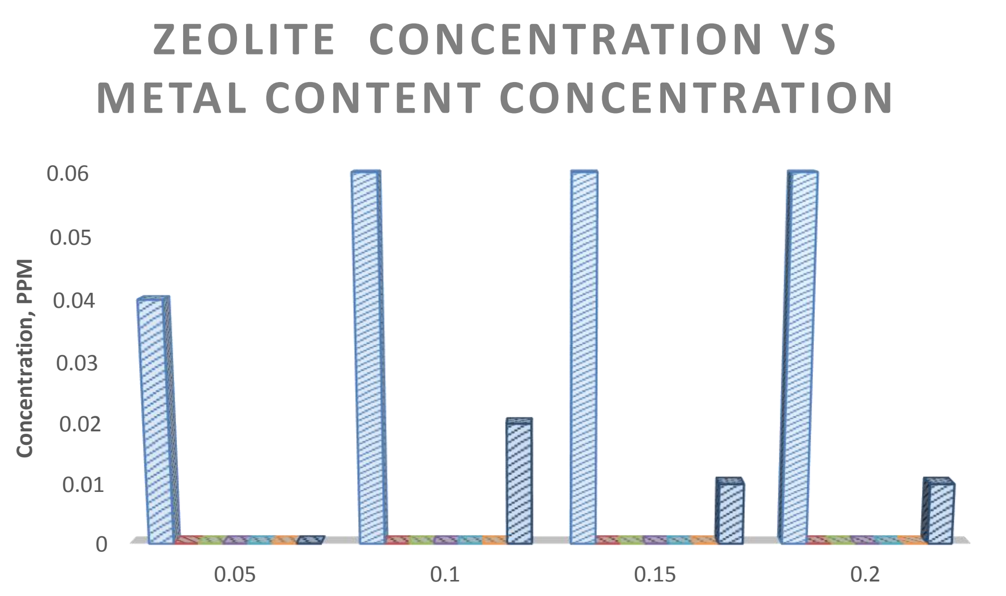


Figure 4.21 Sample solution Copper, Cu concentration

Based on Figure 4.20 and 4.21 it shows that the controlled substance has a higher Copper, Cu concentration compared to the sample solutions tested. It also has a lower absorbance compared to the controlled solution.

4.7.1 Overall AAS Results

It is observed that all test that has been carried out showed positive results based on the metal concentration. All the metal concentration of the sample solution tested for all 6 metals tested were lower compared to the concentration of metal in the known controlled solution. The absorbance of the sample solution were also considerably low for all 6 metals it tested for. It also showed the author that there is a low possibility of metal being leached out of the cement mixture. By observing the data it is applicable that the zeolite cement is able in immobilizing the hydrocarbon waste within its structure. This test was also carried because if metal were able to seep out of the cement moulding and enter the soil and water supply system which can be harmful if consumed by any living organisms. Therefor it was essential that metal content in the sample solution to is low. AAS results shows no leaching of waste occurred.



	0.05	0.1	0.15	0.2
Fe	0.04	0.06	0.06	0.06
Ni	0	0	0	0
Pb	0	0	0	0
Mn	0	0	0	0
Cr	0	0	0	0
Zn	0	0	0	0
Cu	0	0.02	0.01	0.01

Figure 4.22 AAS results at different concentration of Zeolite

Based on Figure 4.22 it shows that all metal content are less than 0.2 PPM. The highest metal content obtain is Iron, Fe and the lowest metal content are lead. Based on the invention (USA, New York Patent No. 4113504, 1977) it shows that the average metal content in hydrocarbon waste is 2.7 ppm for a sample size of less than 100ml and this no reduces as the volume gets larger. It is taken into consideration that the sample was carried out using sample sizes of less than 100ml but the value of metal present is still low. Based on (USA, New York Patent No. 4113504, 1977) it explains as volume increase the metal content decreases per volume. It is observed that even at low volumes that there is a low metal content therefor it further supports the data claiming that zeolite is able to solidify and stabilize hydrocarbon waste in cement.

4.8 Total Oil and Grease (TOG)

The Total oil and Grease (TOG) content was measured using the InfraCal TOG/TPH Analyzer. Based on the mentioned procedure, a sample size of 10 mL was taken, combining a mixture of n-hexane and the sample. The sample was vigorously shaken for 2 minutes before the clear top layer of the mixture was extracted to measure the TOG.

The sample testing was repeated 3 times before taking an average value for the sample TOG content. This was done for the raw hydrocarbon waste sludge and also the four leachate comprising of 5% to 20% of zeolite admixture. Each composition are then calculated for its percentage reduction of total oil and grease concentration. A greater reduction in the TOG content means a larger amount of hydrocarbon waste was able to be immobilized in the cement mixture even after the process of leaching. In addition the percentage of total oil and grease content allowable in waste water is less than 5.0mg/L which is equivalent to 5 ppm based on EPA1988 (Method 9070). If we were to observe the concentration of treated sludge and sediment the allowable oil and grease content is less than 10 ppm based on EPA1988a (Method 90701A).

Table 4.8 Total Oil Grease of Hydrocarbon Waste

Total Oil Grease (TOG)	**Hydrocarbon Waste**		
	1	**2**	**3**
Concentration (ppm)	36.4	36.8	35.9
Average TOG (ppm)	**36.67**		

Table 4.9 Total Oil Grease of Leachate (5% Composition)

Total Oil Grease (TOG)	**Leachate (5% composition)**		
	1	**2**	**3**
Concentration (ppm)	3.6	4.2	3.9
Average TOG (ppm)	**3.83**		
Percentage reduction (%)	**89.56**		

Table 4.10 Total Oil Grease of Leachate (10% Composition)

Total Oil Grease (TOG)	**Leachate (10% composition)**		
	1	**2**	**3**
Concentration (ppm)	3.7	3.8	3.5
Average TOG (ppm)	**3.67**		
Percentage reduction (%)	**89.99**		

Table 4.11 Total Oil Grease of Leachate (15% Composition)

Total Oil Grease (TOG)	**Leachate (15% composition)**		
	1	**2**	**3**
Concentration (ppm)	3.4	4.2	3.9
Average TOG (ppm)	**3.73**		
Percentage reduction (%)	**89.83**		

Table 4.12 Total Oil Grease of Leachate (20% Composition)

Total Oil Grease (TOG)	**Leachate (20% composition)**		
	1	**2**	**3**
Concentration (ppm)	3.5	3.8	4.0
Average TOG (ppm)	**3.77**		
Percentage reduction (%)	**89.72**		

Based on all four composition of cement to binder above which was tested for its total oil and grease content all had a similar reduction of approximately 90% from the original oil and grease content. From all the data collected it is observe that all the concentration of oil and grease is under the standard allowable in waste water making the method of solidification and stabilization of hydrocarbon waste in zeolite cement a viable one.

CHAPTER 5

5.1 CONCLUSION

As a conclusion, this project is important as it deals with an alternative way of treating hydrocarbon waste from industries. Zeolite cement and other cement admixture is believed to be one of the effective ways to encounter the current problem with the conventional ways of treating hydrocarbon waste emitted into the environment. The study on zeolite and add mixtures of component in cement is essential to find the correct ratio and combination of mixtures to immobilize hydrocarbon waste completely in the cement and rendering it completely from leaching. The project is within capability of a final year student to be executed with the help and guidance from the supervisor and the coordinator. The time frame is also feasible and the project can be completed within the time allocated. It is hoped that the acquiring of equipment and materials needed for the experiment runs smoothly for the accomplishment of this project at the end. The outcome of the project needs to be proven acceptable not by the university evaluation board itself but also the team of global expertise. That is the reason why every single test conducted are according to the standard outlined by certain body of standard enforcer such as ASTM or EPA.

From the tests carried out it has come to the conclusion of the author that the two main objectives of this case study was able to be achieved which is to obtain the best mixture ratio of cement to water (C/W) ratio, cement to sludge (C/S) ratio and cement to zeolite binder (C/B) ratio by testing its compressive strength and to study the effects of zeolite on the porosity, permeability, leachability and total oil and grease content of the immobilized hydrocarbon, (HC) waste in the cement. The compressive strength was up to the USEPA standard. In addition after the process of leaching the total oil and grease contend was lower than the standard making it an acceptable results. The metal content was also under the allowable concentration making it an acceptable result. Results from porosity on the other hand showed high porosity values which reduces the credibility of the zeolite cement as a binder. Therefor from these number of tests the author is able observe the credibility of zeolite cement in the solidification and stabilization technology. That is zeolite cement may overcome the chemical aspects of hydrocarbon waste but it loses some of its credibility to become a material for construction due to its porosity and permeability.

5.2 RECOMMENDATIONS

Recommendations from the supervisor and the student will be done and included from time to time as part of the cement mix matrix technology evaluation. The project itself covers many aspects of environmental concerns, therefor to obtain a better result or outcome undergoing multiple types of test and experimentation is important to further clarify the findings from this project. Recommendations done based on certain test that has already been carried out is the preparation of samples for the S/S technology testing should be done in a more tedious manner so that reading obtain would be more consistent. Furthermore carrying out a larger sample size for testing will give a clearer data collection of the S/S technology. Other than that all tests must be carried out based on standards already available to us. Accurate and precise measuring equipment's will give better results. It is also important to calibrate all instrumentation as this will also effect data.

If given more time, more ratios can be researched on, and more tests can be conducted on the sample produced. Characterization of the samples can come from many angles, but due to the time constraint, the research ended with only few tests that is feasible within the time limit as well as provided budget. Another recommendation for this research is for it to be carried out not only for hydrocarbon waste but also a variety of solid and liquid waste. This technique may not be the best in the deposal of hydrocarbon waste but is an alternative and may be an alternative for other waste as well.

To sum up, the technology itself covers many aspects of environmental concerns, which carries the burden of undergoing multiple sets of tests and experimentation to further clarify or standardize the finding from this project.

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APPENDICES

APPENDIX I

ASTM C109/C109M-13 for UCS Test

Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens)

1.1 This test method covers determination of the compressive strength of hydraulic cement mortars, using 2-in. or [50-mm] cube specimens.

Note 1—Test Method C349 provides an alternative procedure for this determination (not to be used for acceptance tests).

1.2 This test method covers the application of the test using either inch-pound or SI units. The values stated in either SI units or inch-pound units are to be regarded separately as standard. Within the text, the SI units are shown in brackets. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in nonconformance with the standard.

1.3 Values in SI units shall be obtained by measurement in SI units or by appropriate conversion, using the Rules for Conversion and Rounding given in IEEE/ASTM SI-10, of measurements made in other units.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. (Warning—Fresh hydraulic cementitious mixtures are caustic and may cause chemical burns to skin and tissue upon prolonged exposure.)

APPENDIX II

Characterization of Hydrcarbon Waste Sheet

F037	Petroleum refinery primary oil/water/ solids separation sludge – Any sludge generated from the gravitational separation of oil/water/solids during the storage or treatment of process wastewaters and oily cooling wastewaters from petroleum refineries. Such sludges include, but are not limited to, those generated in: oil/water solids separators; tanks and impoundments; ditches and other conveyances; sumps; and stormwater units receiving dry weather flow. Sludge generated in stormwater units that do not receive dry weather flow, sludges generated from non-contact once-through cooling waters segregated for treatment from other process or oily cooling waters, sludges generated in aggressive biological treatment units as defined in RCRA Section 261.31(b)(2) (including sludges generated in 1 or more additional units after wastewaters have been treated in aggressive biological treatment units) and K051 wastes are not included in this listing.	(T)
F038	Petroleum refinery secondary (emulsified) oil/water/solids separation sludge – Any sludge and/or float generated from the physical and/or chemical separation of oil/water/solids in process wastewaters and oily cooling wastewaters from petroleum refineries. Such wastes include, but are not limited to, all sludges and floats generated in: induced air flotation (IAF) units, tanks and impoundments, and all sludges generated in dissolved air flotation (DAF) units. Sludges generated in storm water units that do not receive dry weather flow, sludges generated from non-contact once-through cooling waters segregated for treatment from other process or oily cooling waters, sludges and floats generated in aggressive biological treatment units as defined in RCRA Section 261.31(b)(2) (including sludges and floats generated in 1 or more additional units after wastewaters have been treated in aggressive biological treatment units) and F037, K048, and K051 wastes are not included in this listing.	

Hazardous Waste Generated By Generic Processes ("F" List) *(continued)*		
EPA Waste No.	**Hazardous waste**	**Hazard code**
F039	Leachate (liquids that have percolated through land disposed wastes) resulting from the disposal of more than 1 restricted waste classified as hazardous under Subpart D (Leachate resulting from the disposal of 1 or more of the following EPA hazardous wastes and no other hazardous wastes retains its EPA hazardous waste number(s): F020, F021, F022, F026, F027, and/or F028).	(T)

Notes:

- (I,T) should be used to specify mixtures containing ignitable and toxic constituents.
- For the purposes of the F037 and F038 listings, oil/water/solids is defined as oil and/or water and/or solids.
- For the purposes of the F037 and F038 listings, aggressive biological treatment units are defined as units that employ 1 of the following 4 treatment methods: activated sludge; trickling filter; rotating biological contactor for the continuous accelerated biological oxidation of wastewaters; or high-rate aeration. High-rate aeration is a system of surface impoundments or tanks in which intense mechanical aeration is used to completely mix the wastes and enhance biological activity. High-rate aeration units employ a minimum of 6 horsepower per million gallons of treatment volume, and either the hydraulic retention time of the unit is no longer than 5 days, or the hydraulic retention time is no longer than 30 days and the unit does not generate a sludge that is a hazardous waste by the toxicity characteristic. Generators and TSD facilities have the burden of proving that their sludges are exempt from listing as F037 and F038 wastes under this definition. Generators and TSD facilities must maintain, in their operating or other onsite records, documents and data sufficient to prove that the unit is an aggressive biological treatment unit and the sludges sought to be exempted were actually generated in the aggressive biological treatment unit.

APPENDIX III

ratio	KG	KG	m^3	m^3	ratio	KG	KG	KG
W/C	C	C used	C volume	total	needed	C real	W real	W add
0.35	1	1	0.0003	0.0003	0.1699	5.8875	2.0606	2.0606
0.40	1	1	0.0003	0.0003	0.1699	5.8875	2.3550	2.3550
0.45	1	1	0.0003	0.0003	0.1699	5.8875	2.6494	2.6494

Appendix III : Mixing Calculation for Different Water to Cement Ratio(W/C)

APPENDIX IV

ratio	ratio	KG	KG	m^3	KG	KG	m^3	m^3	ratio	KG	KG	KG	KG	KG
C/S_d	W/C	S raw	S dry	S volume	C	C used	C volume	total	needed	C real	S real	W real	W in S	W add
40	0.35	11.6089	1	0.0114	40	40	0.0127	0.0241	12.8563	3.1113	0.9030	1.0890	0.8252	0.2638
50	0.35	11.6089	1	0.0114	50	50	0.0159	0.0273	14.5548	3.4353	0.7976	1.2024	0.7289	0.4735
60	0.35	11.6089	1	0.0114	60	60	0.0191	0.0305	16.2533	3.6916	0.7142	1.2920	0.6527	0.6393

Appendix IV : Mixing Calculation for Cement to Sludge Ratio (C/Sd) at Water to Cement (W/C) ratio = 0.35

ratio	ratio	KG	KG	m^3	KG	KG	m^3	m^3	ratio	KG	KG	KG	KG	KG
C/S_d	W/C	S raw	S dry	S volume	C	C used	C volume	total	needed	C real	S real	W real	W in S	W add
40	0.40	11.6089	1	0.011367	40	40	0.0127	0.0241	15.34038	2.6074	0.7567	1.0429	0.6915	0.3514
50	0.40	11.6089	1	0.011367	50	50	0.0159	0.0273	17.36709	2.8790	0.66841	1.1516	0.6108	0.5407
60	0.40	11.6089	1	0.011367	60	60	0.0191	0.0305	19.39379	3.0937	0.5985	1.2375	0.5470	0.6904

Appendix V: Mixing Calculation for Cement to Sludge Ratio (C/Sd) at Water to Cement (W/C) ratio = 0.40

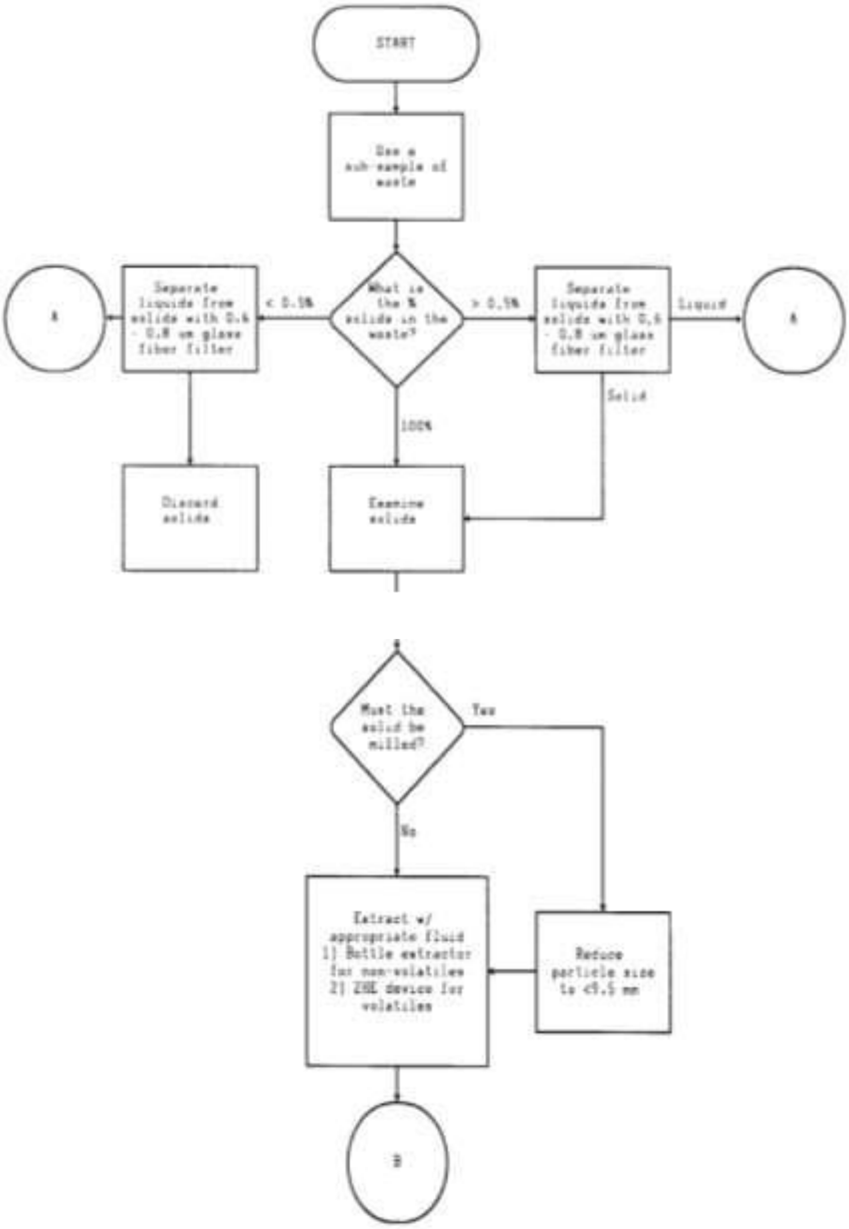
ratio	ratio	KG	KG	m^3	KG	KG	m^3	m^3	ratio	KG	KG	KG	KG	KG
C/S_d	W/C	S raw	S dry	S volume	C	C used	C volume	total	needed	C real	S real	W real	W in S	W add
40	0.45	11.6089	1	0.011367	40	40	0.0127	0.0241	15.34038	2.6074	0.7567	1.17334	0.69155	0.48188
50	0.45	11.6089	1	0.011367	50	50	0.0159	0.0273	17.36709	2.8790	0.66841	1.29554	0.61081	0.68463
60	0.45	11.6089	1	0.011367	60	60	0.0191	0.0305	19.39379	3.0937	0.5985	1.39218	0.54704	0.84514

Appendix VI: Mixing Calculation for Cement to Sludge Ratio (C/Sd) at Water to Cement (W/C) ratio = 0.45

APPENDIX VII Leaching Test

METHOD 1311

TOXICITY CHARACTERISTIC LEACHATE PROCEDURE

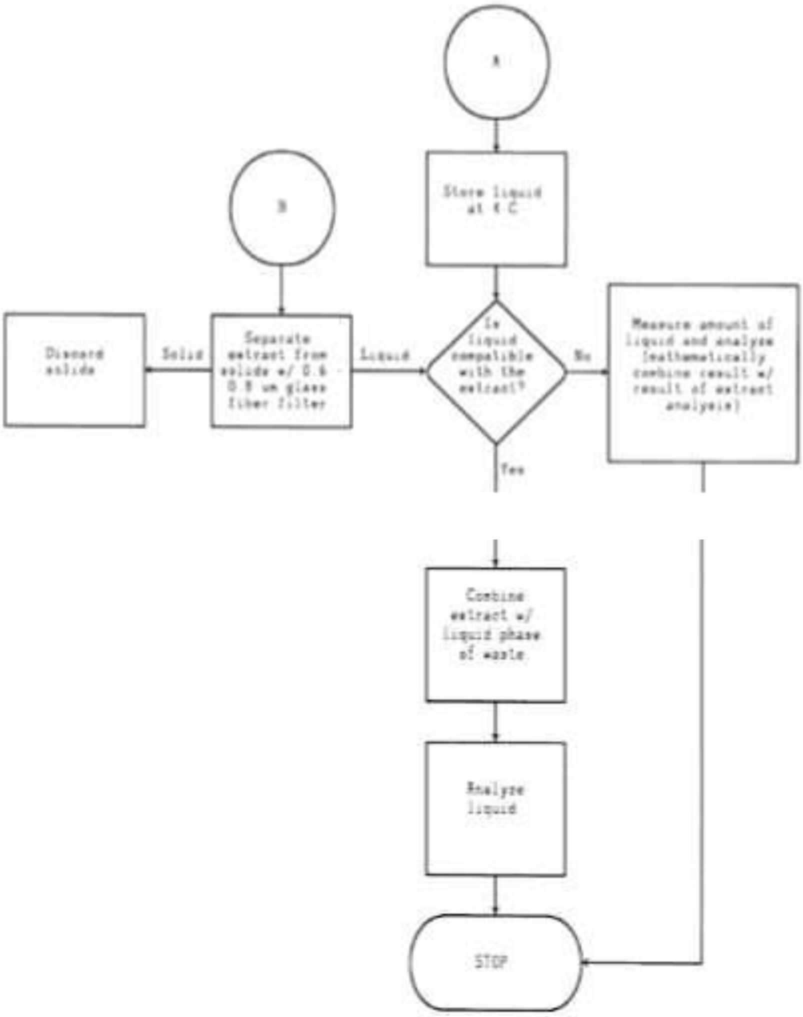


CD-ROM 1311- 34 Revision 0 July 1992

APPENDIX VII (CONTINUED)

METHOD 1311 (CONTINUED)

TOXICITY CHARACTERISTIC LEACHATE PROCEDURE



APPENDIX VIII: Density of Zeolite

QUANTACHROME CORPORATION
Ultrapycnometer 1000 Version 2.2
Analysis Report

Sample & User Parameters

Sample ID: A123
Weight: 5.0235 grams
Analysis Temperature: 32.0 degC

Date: 03-10-14
Time: 11:15:57
User ID: 1013942

Analysis Parameters

Cell Size: Small
V added - Small: 12.4313 cc
V cell: 20.9419 cc
Target Pressure: 19.0 psi
Equilibrium Time: Auto
Flow Purge: 1:00 min.
Maximum Runs: 6
Number of Runs Averaged: 3

Results

Deviation Requested: 0.005 %
Average Volume: 2.0823 cc
Average Density: 2.4124 g/cc
Coefficient of Variation: 0.1964 %

Deviation Achieved: +/- 0.0915
Std. Dev. : 0.0041 cc
Std. Dev. : 0.0047 g/cc

Tabular Data

RUN	VOLUME (cc)	DENSITY (g/cc)
1	2.0704	2.4263
2	2.0779	2.4176
3	2.0823	2.4125
4	2.0802	2.4149
5	2.0787	2.4166
6	2.0880	2.4058

APPENDIX IX: Mixing Calculation

Density of Water = 1000 kg/m^3
Density of Sludge = 1021.31 kg/m^3
Density of Cement = 3140 kg/m^3
Density of Zeolite = 2634.1 kg/m^3
Sludge Moisture Content = 0.913859
Total Solid = 0.086141
Volume of Mould = 15 cubes x (0.05 x 0.05 x 0.05) m^3 for UCS
= 0.001875 m^3

Calculation for Cement to Sludge Ratio (C/S_d) = 40 and Cement to Water Ratio (C/W) = 0.45

Assume
Cement Dry Mass = 40 kg
Sludge Dry Mass = 1 kg
Raw Sludge Mass = 1 kg / Total Solid
= 1 kg / 0.086141
= 11.6089 kg

In the presence of cement replacement material which is the zeolite, the mass of cement reduced according to the percentage of zeolite added. For example:

Percentage of Zeolite: 15 %
Mass of Zeolite based on cement mass = 40 kg x 0.15
= 6 kg

Remaining Amount of Cement in Mixture = 40 kg – 6 kg
= 34 kg

Based on the mass calculated for cement, zeolite as well as raw sludge, the volumes of each component except water was calculated accordingly:

Volume of Cement	=34 kg / 3140 kg/m^3	= 0.01083 m^3
Volume of Zeolite	= 6 kg / 2634.10 kg/m^3	= 0.00228 m^3

Volume of Raw Sludge = 11.6089 kg / 1021.31 kg/m^3 = 0.01137 m^3

Total Volume of Mixture = 0.01083 m^3 + 0.00228 m^3 + 0.01137 m^3

= 0.02448 m^3

Ratio of Calculated Volume/ Ratio of Required Volume

= 0.02448 m^3 / 0.001875 m^3

= 13.056

Based on the ratio calculated above, the real mass of cement, zeolite and raw sludge required for mixing 15 cubic moulds of cement block can be calculated as shown below:

Mass of Cement Required = 34 kg / 13.056 = 2.6042 kg

Mass of Zeolite Required = 6 kg / 13.056 = 0.4596 kg

Mass of Raw Sludge Required = 11.6089 kg / 13.056 = 0.8892 kg

Based on the Cement to Water (C/W) which is 0.45, the amount of water calculated is based on the amount of cement.

Amount of water required = 0.45 x 2.64042 kg = 1.1882 kg

However, water present in the sludge must be considered to prevent too much hydration of the mixture.

Amount of water in sludge = 0.8892 kg x Moisture Content

= 0.8892 kg x 0.913859

= 0.8126 kg of water

Therefore, the real amount of water required is by deducting the amount of water present in the sludge from the amount of water calculated based on cement mass.

Amount of water need to be added: 1.1882 kg – 0.8126 kg = 0.3756 kg