SOLIDIFICATION/ STABILIZATION (S/S) AND IMMOBILIZATION OF ZINC ION-CONTAINING SUDGE USING FLY ASH AND PORTLAND CEMENT

By

NORASHIKIN BINTI NORAINI

FINAL YEAR RESEARCH PROJECT REPORT

Submitted to the Chemical Engineering Programme
in Partial Fulfillment of the Requirements
for the Degree
Bachelor of Engineering (Hons)
(Chemical Engineering)

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CERTIFICATION OF APPROVAL

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Approved:

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Project Supervisor

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK

June 2007

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.

Norashikin binti Noraini

ABSTRACT

Most industrial processes generate wastes in various amounts. The wastes may be hazardous depending on the type of processes and raw materials. Handling all wastes disposal is important to minimize impact to the environment and reduce investment costs. This report intended to point out the outcomes of the project entitle "*Solidification/Stabilization and Immobilization of Zinc ion-containing sludge using Fly Ash and Portland cement*". The main objectives of this project are to obtain the leaching rate and strength of the solidified concrete. The characteristic of the sludge was analyzed by SEM, XRD, SEM and AAS. Then the sludge was bind with the fly ash and Portland cements become concrete and left for curing for 28 days. The concrete then analyzed in term of amount of Zn leached and its strength behavior. The effect of fly ash to the concrete was studied. The optimum ratio which gives the best leaching and strength is at 15% of sludge, 15% fly ash and 70% of cement. The discussion part will explain more about the result including the error occurred during the experiment. The recommendation for improvement also provided at the end of this report. As a conclusion, this experiment is successful to solidify the Zn and producing good strength of concrete which can be further use as a construction material.

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LIST OF ABBREVIATIONS

S/S	Solidification/ Stabilization
Zn	Zinc
TCLP	Toxicity Characteristic Leaching Procedure.
SEM	Scanning Electron Microscopy
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence
AAS	Atomic Absorption Spectrophotometer.

CHAPTER 1
INTRODUCTION

The title of this Final Year Project is Solidification/Stabilization and Immobilization of zinc ion-containing sludge using Fly ash and Portland cement. This chapter will describe the background of study, problem statement, objective and scope of work.

1.1 Background of Study

Stabilization and solidification have been widely applied in the management of hazardous waste. The technologies are being applied to treat the industrial waste prior to secure landfill disposal. In general terms stabilization process is where the zinc is fully or partially bound by addition of supporting binders. Likewise, solidification is a process employing additives, so the physical nature of the waste was altered. Thus, objectives of stabilization and solidification are both to reduce the waste toxicity and mobility as well as improvement in the engineering properties of the stabilized material.

1.2 Problem Statement

The protection of public health and environment from hazardous pollutants has always been an important priority nowadays. With the improvement in petroleum technology, there has also been a rise in the amount of refinery sludge produced. Landfilling such sludge is widely practiced, but there are many limitations. One such limitation is that of heavy metals leaching into the environment at the dumpsite. The accumulation of heavy metals has a significant effect on human and earth. Thus, stabilization of hazardous sludge before disposal is necessary.

Leaching in landfill can occur when the waste is exposed to the stagnant water/ solvent (leachant). This will mobilize the heavy metal and transport it away from the waste. In land disposal, the solvent is usually groundwater. Figure 1 shows some fate of transport processes in the subsurface in landfill [1].

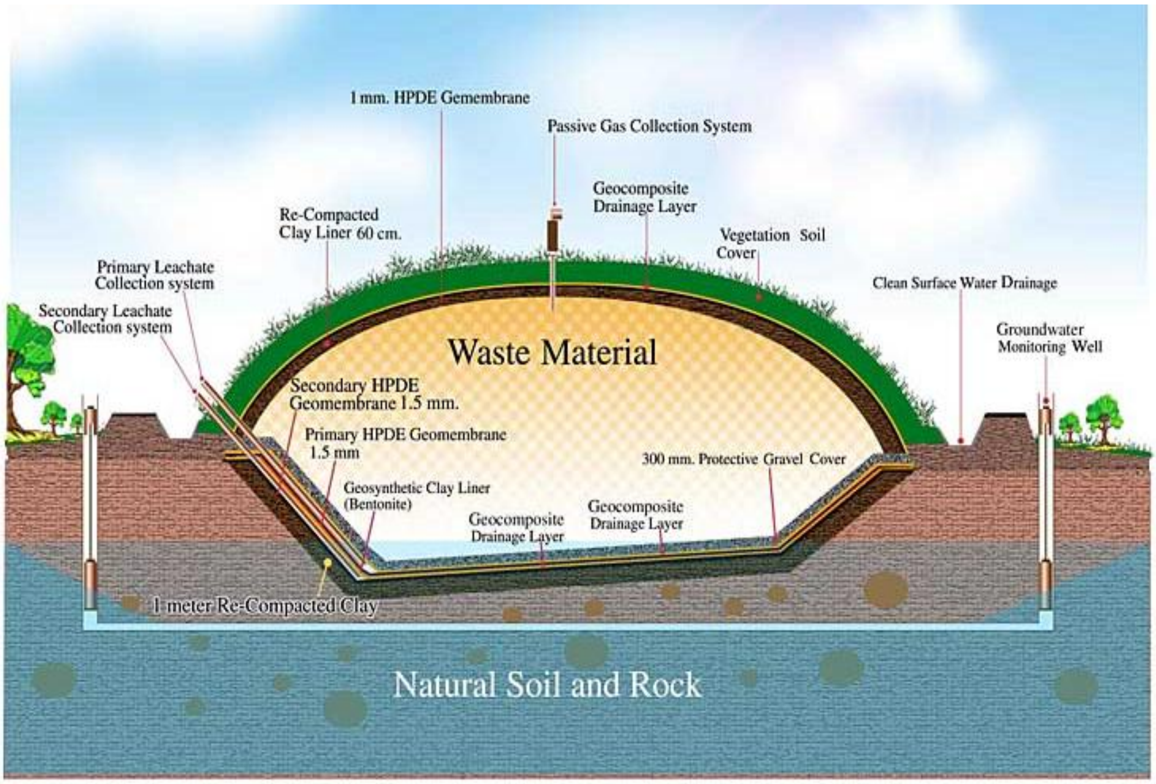

Figure 1 : Some fate of transport processes in the subsurface in landfill.

This study will focus on one of the heavy metal which is zinc. Present of zinc ion in sludge will cause bad impact to people and also environment especially plants. Table 1 shows disadvantages of zinc ion present in groundwater.

Table 1 : Disadvantages of zinc ion present in groundwater.

Effect to people	Effect to Plant
Gastrointestinal	Poor root development
Distress	Growth stunted
Nausea	Chlorosis
Diarrhea	Leaf fall
Anemia	

Kualiti Alam was set up the leaching limit of landfill for zinc ion is 100 mg/l.[2] Any hazardous waste e.g.; sludge that disposed to landfill must satisfied the limit .

1.3 Objectives

This research focused on the formulation of a binding mix of cement and fly ash in solidifying refinery sludge satisfying both compressive strength and leaching requirements for landfill disposal. The optimum amount of fly ash that can replace the cement was studied in other to reduce cost of cement. How the fly ash affects the effectiveness of the project also discussed.

1.4 Scope of Study

The scope of study in this project are first to select the sludge and characterize it, then selection for binders are decided. The concrete then are prepared and their leaching and strength was tested.

1.4.1 Selection of sludge and its characterization

Sludge usually accumulates in refineries because of pump failures, oil draining from tanks and operation units, periodic cleaning of storage tanks and pipeline ruptures.

Due to the physical-chemical processes involved in the treatment, the sludge tends to concentrate heavy metals. Because of this matter, strict standards for the handling, storage and disposal of sludge was established. Sludge used in this studies taken from Petronas Penapisan Melaka Sdn . The characterisation of the sludge was studied by Scanning Electro Magnetic (SEM) , X-Ray Diffraction (XRD) , X-Ray Flourescense (XRF) and Toxicity Characteristic Leaching Procedure (TCLP). This is to analyze weither zinc present or not in the sludge . The leaching amount of zinc before the s/s process also analyzed.

The Scanning Electron Microscope analyses the surface of solid of the sludge. SEM analyze was conducted at several magnification and working distance. In figures 2 and 3, oxides component are represented by the white dots on the images below. It shows that heavy metals exist in the oxide form. The particles are very small and similar sizes thus hard to recognize the zinc [3]. Further test was done using XRD to confirm that the white dots are zinc.

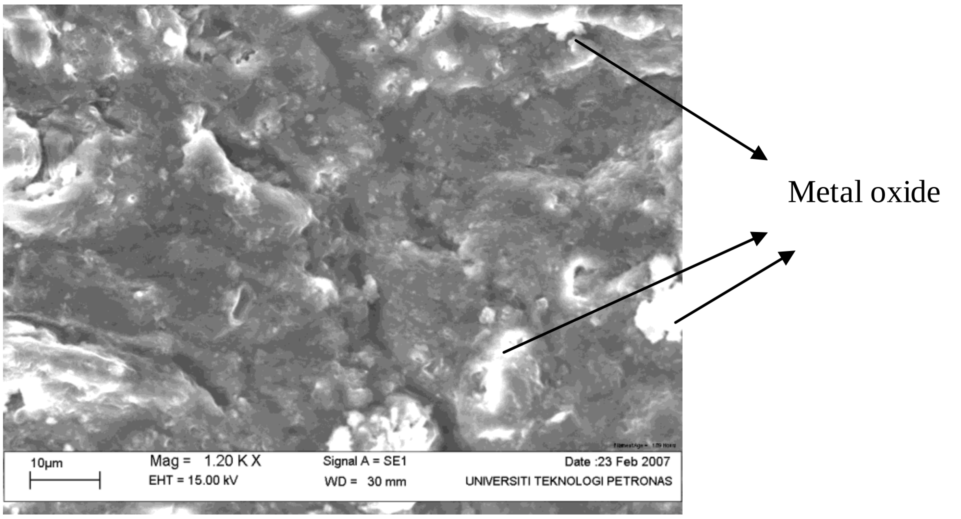

Figure 2 : SEM image of sludge at magnification 1.2 KX

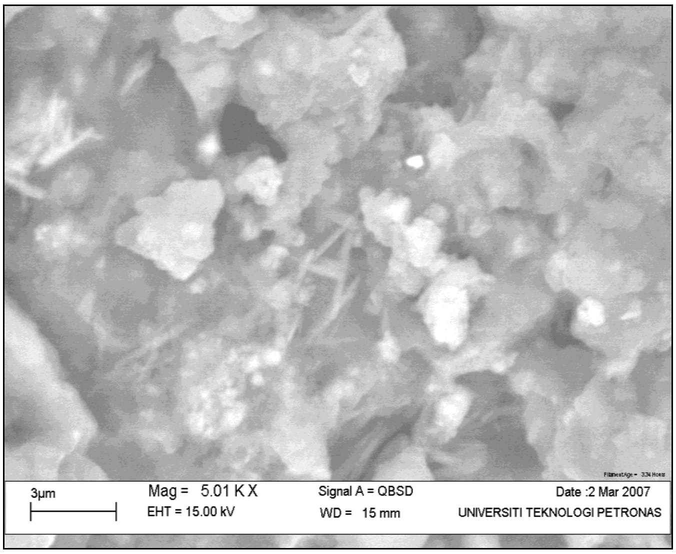

Figure 3 : SEM image of sludge at magnification 5.01KX

Unlike SEM, XRD allows a particular mineral to be identified. Figure 4 shows the graph that tells the present of zinc ion in the sludge. The overlap between black and red peak shows the present of the zinc ion in term of barium zinc sulfate particle in the sludge. But XRD test did not tell the quantity of ion zinc present in the sludge. Only AAS and XRF will tell the quantity of heavy metal present.

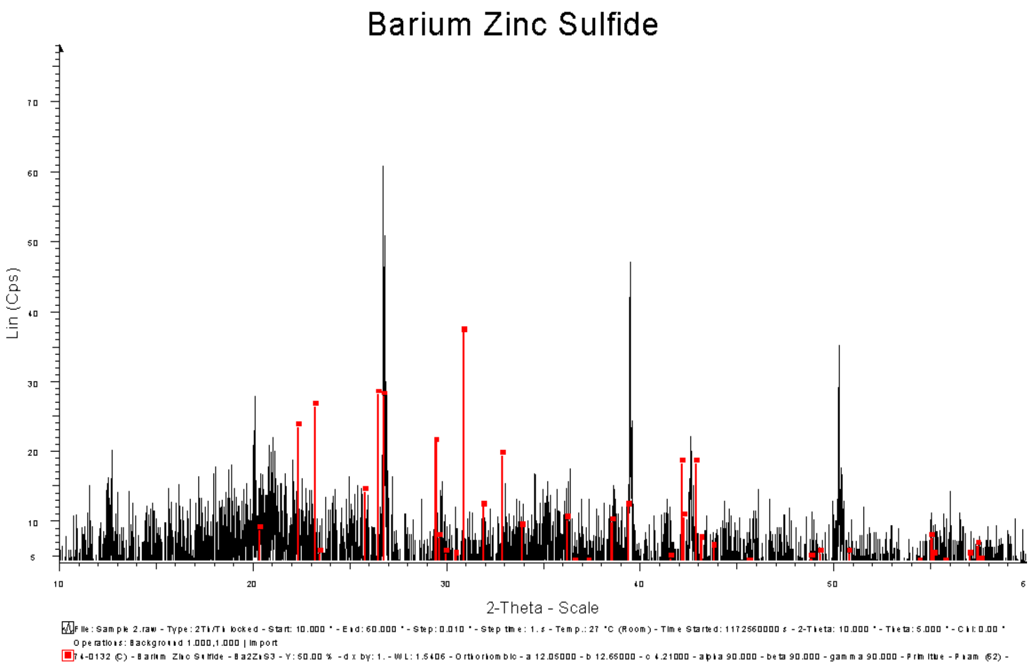

Figure 4 : XRD analyses for zinc.

In this research also XRF is used to determine the composition of zinc ion in the sludge. Table 2 shows that in 15g of sample sludge tested, there is 2.57% of zinc element. This is clearly indicate how mush amount of each element in the sludge in term of percentage of composition.

Table 2 : XRF analysis for sludge.

Component	**Composition (%)**	**Component**	**Composition (%)**
Mg	0.859	Mn	0.276
Al	9.66	Fe	32.5
Si	18.9	Cu	0.206
P	3.53	***Zn***	***2.57***
S	15.7	Se	0.261
Ca	7.01	Br	0.0604
Ti	1.07	Sr	0.228
Cr	0.0776	Mo	0.958
Ba	1.07	Tb	0.0398

Before the sludge being solidified and stabilized, the amount of Zn in the sludge is 38.352 mg/l. This shows the concentration of zinc is within the limit stipulate by Kualiti Alam which is 100mg/l. However, s/s process still proceeds to ensure the protection of the environment.

1.4.2 Selection of Binder.

Fly ash is the non-combustible mineral portion of coal generated in a coal combustion power plant. Fly ash is useful as a binder in many applications because it is an alumino-siliceous material that, when in the presence of water, it will combine with calcium hydroxide to form cementations compounds.

Two classes of fly ash are defined in ASTM C618 are Type F and Type C. Class F Fly Ash was used in this experiment was taken from Jana Manjung Power plant. It is produced from the burning of bituminous coal. The advantages of using fly ash include increased compressive strength, workability, durability, permeability, and reduced sulfite attack, bleeding, shrinkage, and heat or hydration. However fly ash has be to used at optimum amount. Too mush of fly ash will cause the structure of concrete more brittle thus reduce the strength of concrete. [4] Although fly ash as a partial replacement for cement has been utilized for many years, it has been almost exclusively used in low-volume percentages, such as 10 or 20% cement replacement [6]

Portland cement is the most common type of cement in general usage. It is a basic ingredient of concrete, mortar and plaster. It consists of a mixture of oxides of calcium, silicon and aluminum. Portland cement and similar materials are made by heating limestone with clay, and grinding this product with a source of sulfate. When mixed with water, the resulting powder will become a hydrated solid over time [1].

1.4.3 Concrete Mixing

During this experiment, efforts were made to ensure the homogeneity of the fly ash, cement, sludge, sand and aggregate by preparing a few samples of concrete. Concrete by definition, is a particulate composite consisting of hydrated cement and aggregate.

Based on the 1:2:4 concrete proportions, where 1 proportion is cement, 2 is sand and 4 is aggregate, concretes were mixed using machine[6]. Sand and aggregate proportions were kept constant while the sludge, fly ash and cement amount were played around in the proportion of one. Table 3 shows eight different ratios of fly ash, sludge and cement in terms of percentage.

Table 3 : Composition of concrete

Ratio	Sludge (%)	Fly Ash (%)	Cement (%)
1	30	10	60
2	10	10	80
3	20	20	60
4	20	15	65
5	30	20	50
6	20	30	50
7	15	15	70
8	25	25	50

Based on the proportion, the materials were weighted and mixed. Figure 5 shows the composition of eight different set of recipe of concrete in terms of weight in kg. The weight of aggregate and sand is constant at about 1.515 kg aggregate per mold of and 0.757 kg sand per mold of concrete.

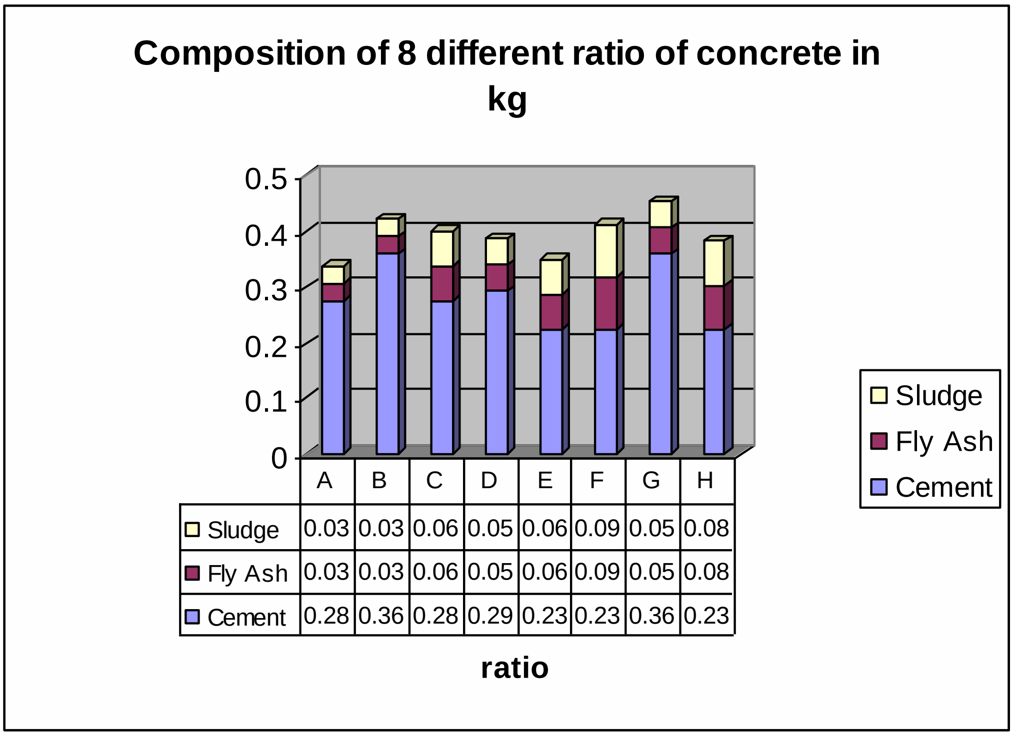

Figure 5 : Composition of eight different set of recipe of concrete.

The sludge-cement-fly ash mixtures were cast in 100 mm cubes and these solidified samples then were cured at room temperature for 28 days. This is to ensure the maximum strength of the concrete can be obtained.

1.4.4 Concrete Testing.

After 28 days of the maturity period, the concrete design was tested in terms of the leaching of zinc ion, and the strength of the concrete. The Toxicity Characteristic Leaching Procedure (TCLP) was adopted in this project to study the leachable zinc before and after s/s process. The TCLP is widely used to evaluate the effectiveness of the stabilized material. The unconfined compressive strength test was conducted to determine the strength of the stabilize concrete.

CHAPTER 2
LITERATURE REVIEW AND / THEORY

2.1 Cement based technology.

Cement-based stabilization is best suited for inorganic wastes, especially those containing heavy metals. As a result of the high pH of cement, the metals are retained in a form of insoluble hydroxide or carbonate salts within the hardened structure. Other additives also incorporated into stabilizing mix with the cement to reduce organic contaminant interference with cement hydration and enhance stabilization. In the simplest form, the reaction of the tricalcium silicate can be express by the reaction equation

$$2(3CaO{\bullet}SiO_2) + 6H_2O \rightarrow 3\ CaO{\bullet}2\ SiO_2{\bullet}3\ H_2O + 3Ca(OH)_2$$

and the reaction of dicalcium silicate by ;

$$2(2CaO{\bullet}SiO_2) + 4H_2O \rightarrow 3\ CaO{\bullet}2\ SiO_2{\bullet}3\ H_2O + 3Ca(OH)_2$$

The reaction is quite slow, contributing to the lengthy curing time associated with Portland cement concretes. The most rapid reaction in Portland cement is :

$$3CaO{\bullet}Al_2O_3 + 6H_2O \rightarrow 3\ CaO{\bullet}\ Al_2O_3{\bullet}6H_2O + heat$$

2.2 Mechanism of S/S

The process of s/s employs one or more of the following mechanism:

- ***Macroencapsulation***
- ***Microencapsulation***
- ***Absorption***
- ***Adsorption.***

Macroencapsulation is the mechanism by which the hazardous waste, in this case is zinc are entrapped in a larger structural. They are held in discontinuous pores within the stabilizing materials. On physical degradation of the stabilized material, even to relatively large particle sizes, the entrapped materials are free to migrate. The stabilized mass may break down over time because of environmental stresses. Thus, contaminants stabilized by only macroencapsulation may find their way into the environment .

Microencapsulation. In micro encapsulation, zinc is entrapped within the crystalline structure of the solidified structure at microscopic level. As a result, even if the stabilized material degrades into relatively small particle sizes, most of the stabilized zinc remains entrapped. However, as with macroencapsulation, because the zinc is not chemically bound, the rates of contaminant release may increase as the particle size decrease.

Absorption is the process where the zinc are taken into the sorbent in the same way as a sponge on water. In s/s, absorption requires addition of solid material to adsorb the free liquid to improve the waste handling characteristics, which is to solidify the waste. The most common absorbents include soil, fly ash, cement, lime and clay material. Absorbents used in this study are fly ash and cement.

Adsorption. In addition to the physical entrapment within the stabilized and solidified mass, electrochemical interaction may occur. Adsorption is the process where zinc are chemically bonded to stabilizing agents which are fly ash and cement within the structure. These are typically known as surface phenomena and the nature of the bonding may be through Van Der Waals or hydrogen bonding. Zinc that chemically adsorbed within the stabilize structure less likely to be release into the environment than those are not fixed. Unlike microencapsulation and macroencapsulation, where simple particle breakdown may enhance the rate of contaminant migration, additional physicochemical stress is necessary to desorb the material from their adsorbing surface. Thus, the treatment will be more permanent [1]:.

However, some fundamental aspects such as the chemical mechanism of hydration, the bonding between the waste material with cement and detailed microstructural and microchemical studies are still poorly understood and lack quantification.

2.3 Curing Period

The effectiveness of cement-based process to stabilize and immobilize zinc has been demonstrated. The effectiveness of the s/s process requires the measurement of physical, chemical and engineering properties of the stabilized material. As the complication, the curing may take a month. Civil engineers traditionally test samples of concrete 28 days after mixing, and this procedure has carried over into the evaluation of stabilized materials. However, since the hydration process goes on long after 28 days after mixing, the leaching test results from 28 days samples may overestimate the leachibility of fully cured sample. In one study, reduction in moisture content on samples obtained 9 and 18 month after curing demonstrated this ongoing, long term nature of the process.

Adding water to Portland cement to form the water-cement paste that holds concrete together starts a chemical reaction that makes the paste into a bonding agent. This reaction, called hydration, produces a stone-like substance—the hardened cement paste. Both the rate and degree of hydration, and the resulting strength of the final concrete, depend on the curing process that follows placing and consolidating the plastic concrete.

Hydration continues indefinitely at a decreasing rate as long as the mixture contains water and the temperature conditions are favorable. Once the water is removed, hydration ceases and cannot be restarted.

2.4 Laboratory testing.

As natural outcome of the complexities, a large number of laboratory tests are utilized to evaluate the effectiveness of the stabilization. The selection of appropriate test and interpretation of the test results depend on the objectives of the stabilization process. As in this project, the calculated risk from the groundwater might be in the function of the amount of zinc that is estimate to leach.

A number of studies have been carried out on the s/s of heavy metal sludge and other hazardous waste using cement, chemicals, lime and coal plant fly ash. Research has also been done on the use of fly ash as a cementious binder and on the use of cement to stabilize fly ash. Based on the study by these people, some said the optimum design which to immobilize the heavy metal was agreed as 45% fly ash, 5% cement and 50% sludge[5]. But at this design ratio, it just the very minimum leaching value of the heavy metal containing the structure. With that with 5-15% cement, it will not give desired strength which is 30 MPa. So, this project will try to find optimum ratio that can give minimum leaching behaviors and also high strength of the concrete.

2.5 Application of S/S

Even in Malaysia, Kualiti Alam, the only organization who handle the schedule waste also implement s/s to treat the waste. In their Solidification Plant, inorganic wastes, which do not fulfill the criteria for disposal directly into the Secure Landfill, are treated. Such wastes are typically metal hydroxide sludge containing heavy metals such as lead, arsenic, nickel, zinc and chromium.

During the solidification process the heavy metals become insoluble and the wastes therefore can safely be disposed off in the secure landfill. Fly ash from the Incinerator Plant and sludge from the PCT Plant are also treated at their Solidification Plant.

At their Solidification Plant, waste is loaded into waste bunkers, where it will be mixed with other similar waste. It is then loaded into the waste hopper before being transferred to the mixer by screw conveyors. In the mixer, waste is carefully mixed with consumables such as cement, lime and water. The system is able to handle waste that contains foreign materials such as stones, wood and scrap iron. A typical solidification recipe is as in Table 4[2].:-

Table 4 : Composition of concrete applied by Kualiti Alam S/S plant.

Waste	Fly Ash	Cement	Lime	Sand	Water
100	20	35	7	100	30

After treatment, the waste will appear as a concrete mixture. The mixture is disposed off to the secure landfill for the final curing over a few days. The objective of the whole process is to fix all the heavy metals in the inorganic solid waste into a concrete/silica matrix for long-term disposal in the secure landfill. As a result, hazardous heavy metals will not leach out to the environment. Table 5 shows type of hazardous waste treat by Kualiti Alam using S/S methods. [2]

Table 5 : Type of hazardous wastes undergoes S/S process in Kualiti Alam.

N016	Sludge from oil storage tank(low content of oil and grease)
N151	Metal hydroxide sludge from wastewater treatment system
N203	Residues from recovery of acid pickling liquor
N204	Oxide or sulphate from wastewater treatment system

CHAPTER 3
METHODOLOGY

As describe the scope of studies, this experiment has 4 stages that have been completed which are:

- Selection of sludge and the characterization,
- Selection of binders and its characterization,
- Mixing the concrete and finally
- Testing the concrete strength and leaching.

During the sludge characterization, SEM, XRD, XRF and TCLP test was done to study the present of zinc in the sludge. The procedure for each testing method above will be discussed below. Figure 6 shows the overall methodology of the experiment.

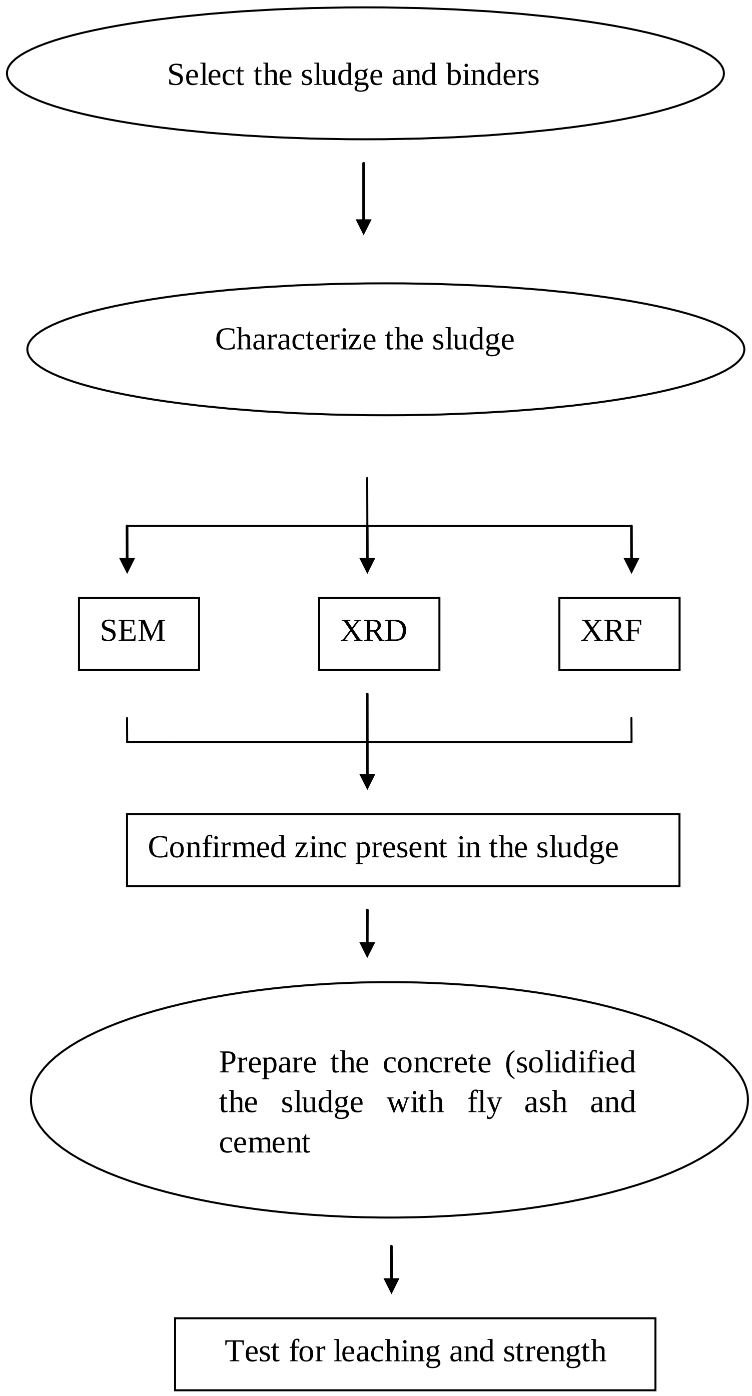

Figure 6 : Methodology of the project

The brief description for each of the testing method such as SEM, XRD, XRF, TCLP and AAS will be discuss next

:

3.1 Testing Procedure.

3.1.1 Scanning Electron Microscopy

Figure 7 : Scanning Electron Microscopy.

SEM was done to observe morphology of the sludge to study weither zinc present or not in the sludge. The procedure of doing the experiment shows in Figure 8

One pellet of sludge were placed

↓

Sample were coated with the gold palladium metal to reduce the oxide effect on it, which block the microscope lenses

↓

The coated samples were then put on microscope and analyze at working distance 15 mm, magnification 5.01kX

Figure 8 : SEM working procedure.

3.1.2 X-Ray Diffraction (XRD)

Figure 10 shows the procedure to test the sample using X- Ray Diffraction.

Figure 9 : XRD

The sample of sludge were scanned using nickel filtered radiation in the range of 10^{o} $<2\Theta <$ 75^{o} in the step mode.

↓

The mean crystalline size of ZnO were determined from the line broading of the diffraction line for ZnO

Figure 10 : XRD working procedure.

3.1.3 Toxicity Characteristic Leaching Procedure. (TCLP)

In TCLP (shown in figure 11) test, samples was crushed into fine particle and extracted with a lechant (acetic acid) at pH 3. The acetic acid is added only once at the start of the extraction. A liquid to solid ratio 20: 1 was used for an extraction period of 18 hours. The lechate then filtered prior to conducting the contamination analysis. The filtrate is acidified using concentrated nitric and hydrochloric acid and analyze for zinc component using Atomic Absorbtion Spectrophotometer (AAS).

Figure 11 : TCLP

3.1.4 Atomic Absorbtion Spectrophotometer (AAS).

AAS has been standard tool employed by the analysts for the determination of the trace level of metals. In this technique, a fine spray of the analyst is passed into a suitable flame, frequently as acetylene oxide which converts the element to an atomic vapor is passed through radiation at the right wavelength to excite the ground state atom to the first excited electronic level. The amount of radiation absorbed can be measured and directly related to the atom concentration, a hollow cathode.

3.2 Mixing Concrete Procedure.

Mixing and sampling the fresh concrete is recommended by BS 1881 : Part 125 : 1986 .the method as in the Figure 12.

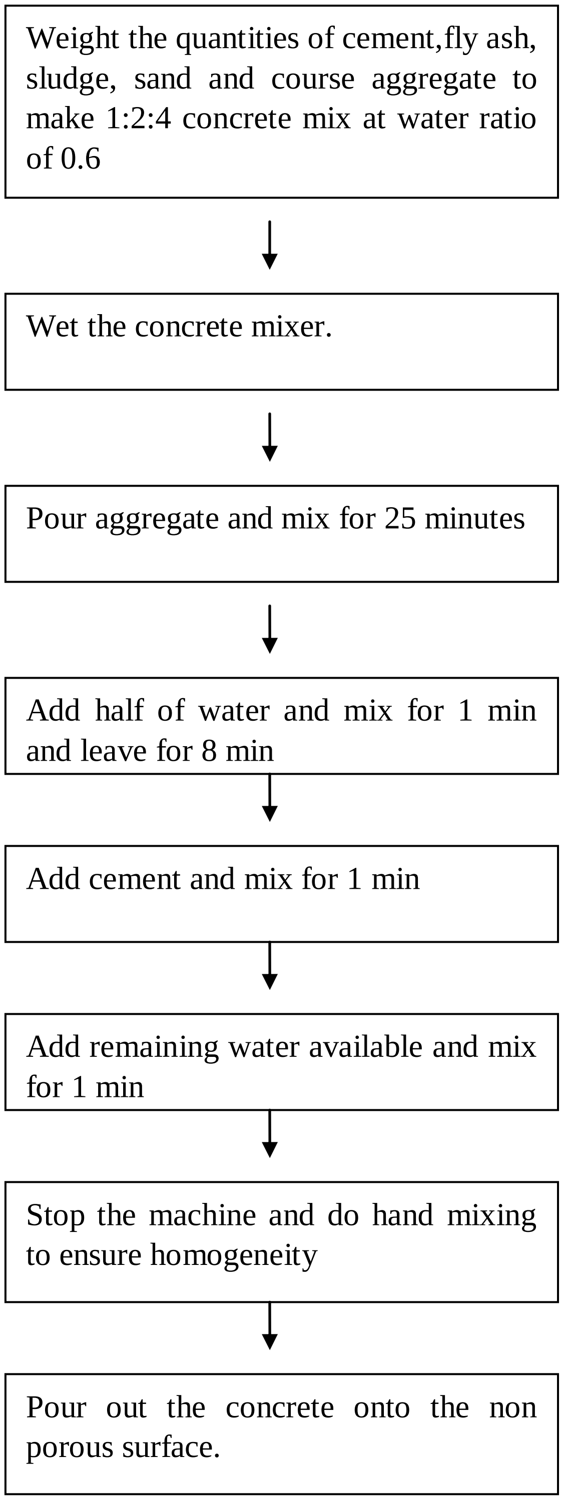

Figure 12 : Machine mixing concrete procedure.

3.3 Compressive Strength Test Cubes – Test for Strength

Figure 13 explain how the compressive strength test was done.

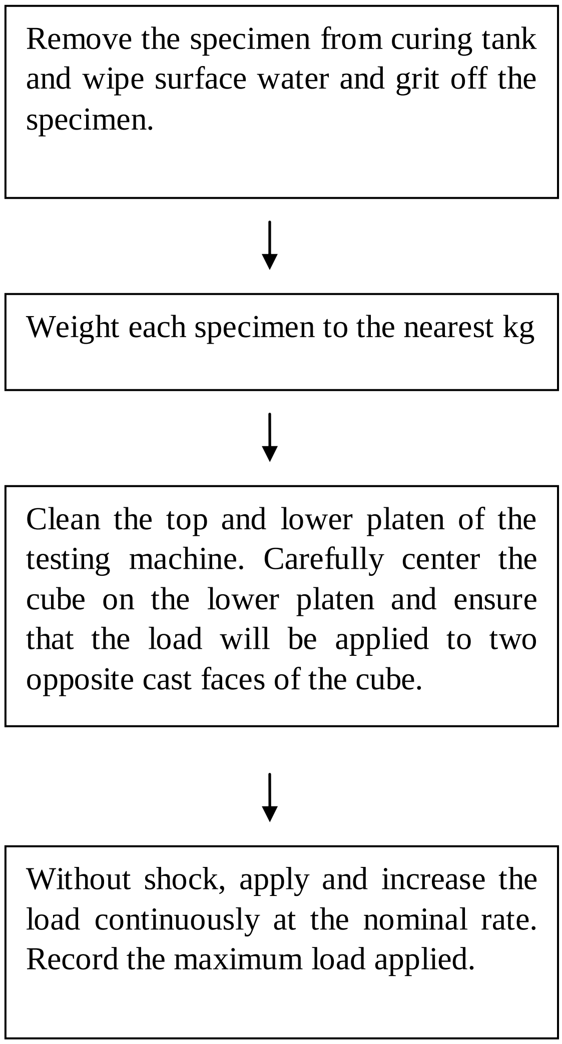

Figure 13 : Compressive strength test procedure.

In figure 14, the load has to be slowly applied until the concrete crack. That is when the maximum load will be obtained.

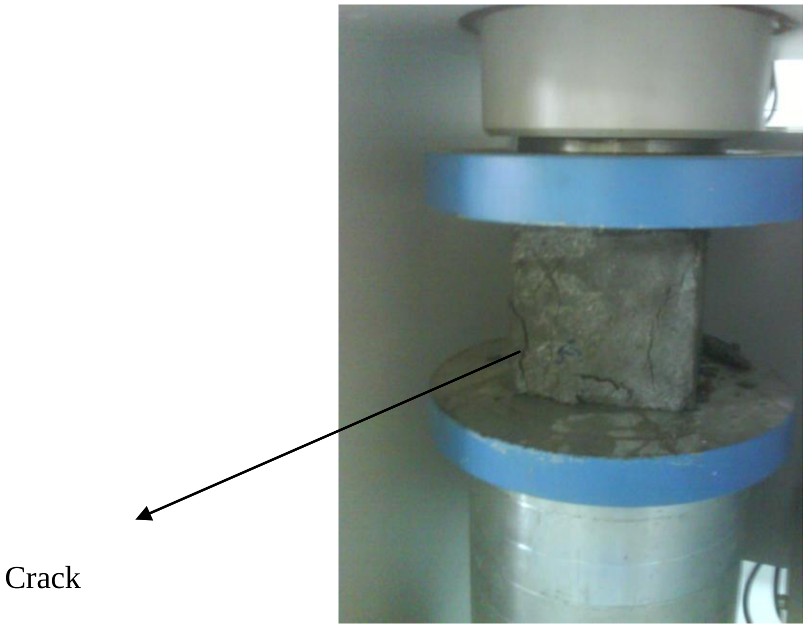

Figure 14 : Compressive strength test.

CHAPTER 4
RESULT AND DISCUSSION

Once the concrete curing period reach 28 days, the sample of concrete was taken out to be tested on their leaching amount of zinc and the strength of the concrete.

4.1 Leaching of Zn after S/S.

Table 6 shows the amount of concentration of zinc leach into the acetic acid as comparison to the groundwater after 28 days.

Table 6 : Leaching amount of zinc after s/s process

Set of concrete	Ratio Sludge:FA:Cement	Limit for zinc (mg/l)	Concentration of leaching before S/S (mg/l)	Concentration of leaching after S/S (mg/l)	% of zinc immobilize	Rate of leaching,*l* (cm/day)
A	**30:10:60**	**100**	**38.352**	22.07385	42.44407	0.20555689
B	**10:10:80**			15.69105	59.08675	0.146118753
C	**20:20:60**			14.8932	61.16708	0.138688986
D	**20:15:65**			9.5742	75.03598	0.089157205
E	30:20:50			18.6165	51.45885	0.173361233
F	**20:30:50**			15.957	58.3933	0.148595342
G	15:15:70			5.58495	85.43766	0.05200837
H	**25:10:65**			19.41435	49.37852	0.180791

From this table, it shows ratio G with **15:15:70** ratio of **sludge: fly ash: cement** give the best result of in terms of leaching behavior. It is because at this mixing ratio, 85.44% of zinc will remain in the solidified concrete and not leached out. The leaching rate at this ratio 0.05200837cm/day. Even the number is quit small, it cannot be neglected. The effect might not be in short period of time, but after a few years later when this amount accumulate and affect the environment.

However, since the amount of sludge in the entire ratio is not constant, comparison of leachibility cannot be made. In order to study the effectiveness of the experiment, three ratios as highlighted in blue above are selected. As we can see, all these three has same amount of sludge which 20%. The affect of addition of fly ash a binder was observed as in the Figure 15.

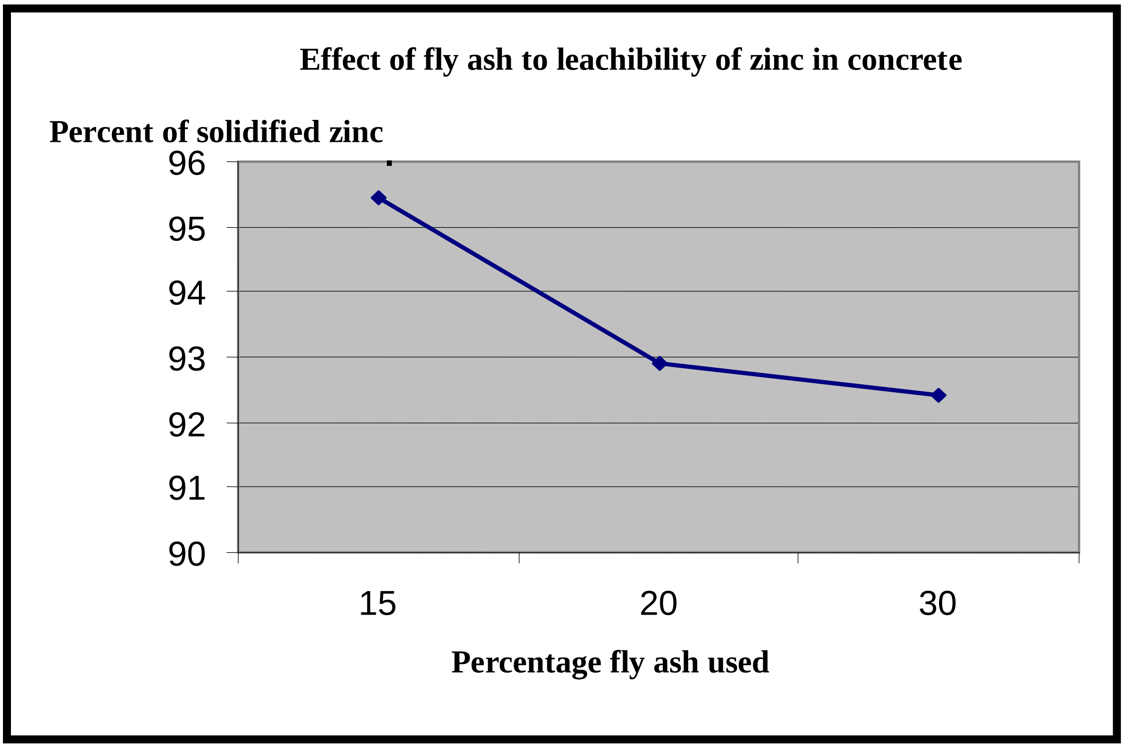

Figure 15 : Graph on Percent of solidified zinc in concrete vs. percentage of fly ash used.

From the graph above, as the amount of fly ash increase from 15 to 30 percent, the percentages of zinc solidified are reduced. This confirmed what have been discussed in the previous studies. Fly ash is good as a binder; however, too much fly ash will cause the concrete to be brittle [4]. Thus, it will reduce the strength of the concrete and cause the zinc to mobilize. The optimum amount of fly ash has to be used. This will help in reduction of the cost of s/s process since the fly ashes are available at the low cost compared with the cement. In this study, 15 % of fly ash is the optimum amount which gives the best leaching performance.

Since the concentration of zinc in the sludge studied in this experiment already below the limit required by regulation, the focused of this experiment is to minimize the amount of zinc leach to the environment as mush as possible was succeed.

Based on this equation below [1], leaching rate (l) (cm/day) for Zn at 28 days was calculated. The data was tabulated in the Table 6.

$$l = \frac{a_n \, V}{A_0 \, S t_n}$$

Where

a_n = *amount of heavy metal leach at n day*
V/S = *Volume of the concrete / Area of the surface of concrete.*
A_0 = *Amount of heavy metal before s/s*
t_n = *Curing period*

As the amount of fly ash used larger, the lechate of Zn also increases as in Figure 16

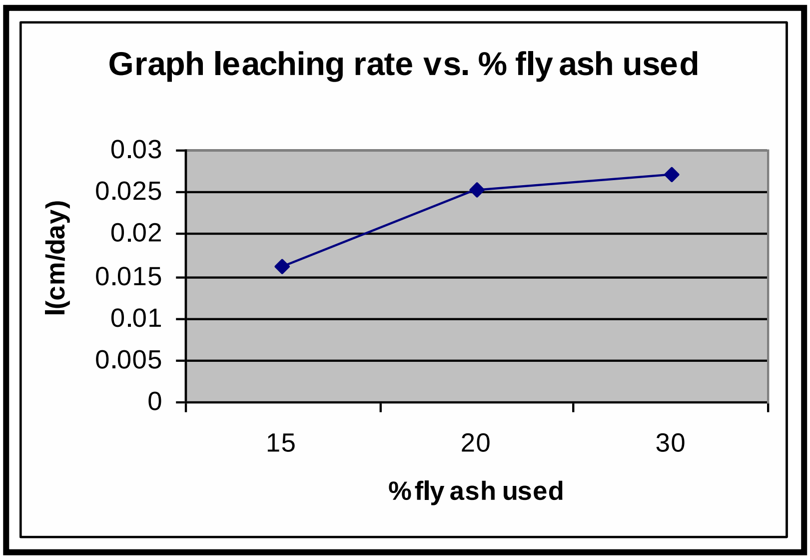

Figure 16 : Graph leaching rate vs. percentage of fly ash used.

4.2 Strength Result.

Effective in the leaching behavior is not good enough for the solidified concrete if the strength of the concrete is low. If the strength is low, the concrete has higher possibility to crack thus; it will cause the Zn to mobilize. The ratios which give the optimum strength will be discussed based on the result in table 7.

Table 7 : Strength of the solidified concrete.

Set of concrete	Ratio Sludge:FA:Cement	Limit for concrete strength (Mpa)	Strength of concrete after S/S (Mpa)			% of concrete strength
			14 days	**21 days**	**28 days**	
A	**30:10:60**	**30**	**11.45**	**13.75**	**15.26**	**50.8**
B	**10:10:80**		**13.63**	**17.98**	**20.7**	**59**
C	**20:20:60**		**10.91**	**14.02**	**15.64**	**52.13**
D	**20:15:65**		**17.85**	**21.74**	**25.71**	**85.7**
E	**30:20:50**		**10.38**	**13.5**	**14**	**46.67**
F	**20:30:50**		**10.43**	**13.25**	**13.36**	**44.53**
G	**15:15:70**		**19.06**	**23.65**	**28.16**	**93.66**
H	**25:10:65**		**8.97**	**13.25**	**13.4**	**44.67**

Based on the data collected, the graph was plotted as below. In the graph shows ratio G which is 15 % sludge, 15 % fly ash and 70 % cement give highest strength which is 28.19 MPa. This result complements each other to the result of the leaching. As the strength increases, the amount zinc leach to environment also decreases. It shows in the strength table that ratio G gives the highest percentage of Zn that has been immobilized in the concrete. Thus, it can be concluded that G which is 15% fly ash, 15% sludge and 70% cement is the optimum ratio to get the best leaching and strength behavior.

Reduction of 30% cement usage can reduce the investment cost for implementing this way of treatment. Furthermore the strength given at this optimum ratio is 28.16 MPa. Considering there are errors occur during the experiment, the strength is quite acceptable compared to standard strength of the concrete which is 30 Mpa.

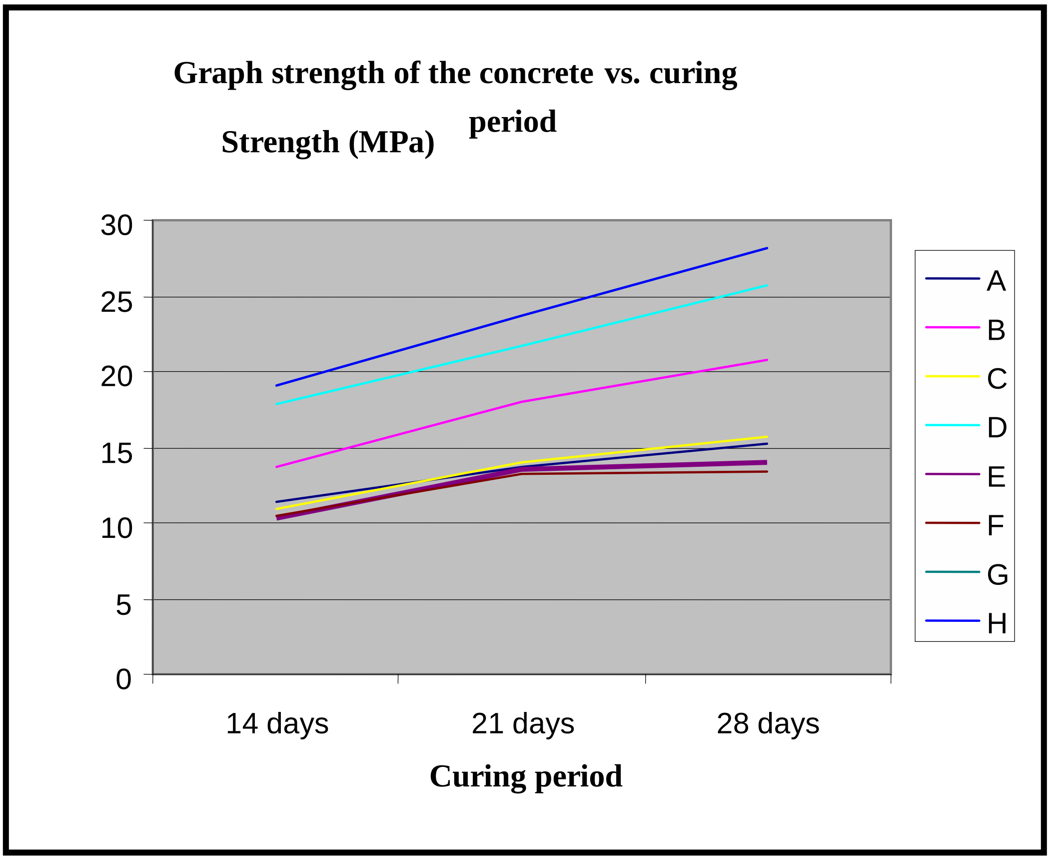

Figure 17 : Graph strength of the concrete vs. curing period.

It also shows in figure 18, when the concrete was kept in for maturity to the 28 days; the trend of the graph shows the strength will be increasing from day 14 up to 28 which the maximum strength can be obtained. This is due to time taken to the material to react and bind which each other to give 100% strength after 28 days.

As in the leaching test, to compare the effectiveness of the s/s employed in this study, the ratio of C, D and F was selected as they having the same amount of sludge. Figure below shows how the amount of fly ash affects the strength of the solidified concrete.

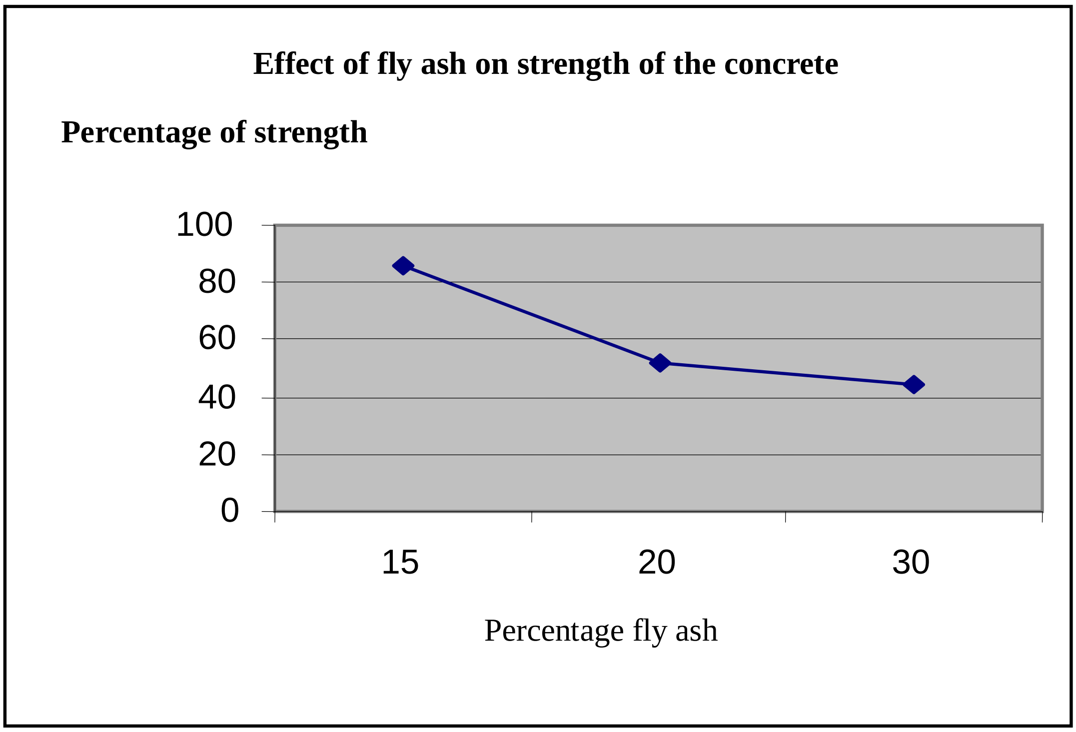

Figure 18 : Graph strength of concrete vs. percentage of fly ash used.

As discussed above, when the amount of fly ash increase, the strength of the concrete will be decrease. This is due to the properties of the fly ash that make the structure of the concrete brittle as the amount is bigger.

4.3 Error in experiment

Through out the experiment there are some errors occur which affect the result of the experiment. The errors will be discussed below:

4.3.1 Human and Apparatus error.

During the experiments, human errors are common since the laboratory work involved much of the dilution used measuring cylinder with small scale.

Other than that, apparatus such as AAS and XRF can give error in terms of measurement. XRF for example can give error in measurement with the present of moisture in the sample tested.

4.3.2 Inconstant curing temperature.

The appropriate temperature for curing is at 24^{o}C. However the temperature of room used for the experiment could not be maintained as it was affected due to electric open and shutdown of electricity through out the curing period.

4.3.3 Concrete mixtures composition.

Proportionate composition must be prepared. There is no exact composition, thus trial and error is required to obtain the best composition. Water to cement ratio is also important factor that will affect the S/S process. Error during weighing and preparing the exact amount of material can affect the result also.

CHAPTER 5
CONCLUSION AND RECOMMENDATION

5.1 Conclusion.

The aimed at producing a new solidification recipe with the using of fly ash and Portland cement was achieved. Utilization of fly ash is focused by varying its ratio in the samples. The variation will differentiate the effectiveness of the fly ash as additives by comparing the leaching rate between the samples. The strength of the concrete also observed.

From the leaching test done, the Zn able to be bind up to 85.44% with 15% sludge, 15% fly ash and 70% cement. The leaching rate at of Zn this optimum ratio is 0.00052 cm/day. The strength of this ratio given as 28. 16MPa. Reducing the cement up to 30% will able to reduce the cost of concrete as fly as is the residue of the process and available at low cost.

However, amount of fly ash used cannot be more than 15% because it will affect the strength of the concrete. The relationship between percentage of fly ash used shows as the amount of fly ash increase, strength will be decreasing. Same with the leaching, as fly ash increasing, the amount of zinc immobilize will be decreasing.

It can be concluded as this experiment succeeded to solidify the sludge contain Zn ion using fly ash. Because of the high strength of the concrete, it also can be further use for the construction material.

5.2 Recommendation.

Several things can be done in order to improve the project:

- Make sure all the equipment is well calibrated and maintenances are done frequently. Therefore errors could be avoided.
- Try the combination of other type of binders such as lime and clay and study how that affect the performance of leaching and strength of the concrete.
- Varying the composition of fly ash and cement and concentrate the composition around most selective ratio in this experiment, thus the more accurate composition can be obtained.
- Study more such as the sample for porosity, durability and moisture content.

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< http://www.pubs.asce.org/WWWdisplay.cgi?0600579>

APPENDIX A

PROJECT GANTT CHART

A	B	C	D	E	F	G	H	I	J	K	L	M	N	O
2. Find out the objectives of the project	■													
3. Gather information	■													
Decide type of material to be used														
1. Study the characteristic of fly ash, cement and sludge	■	■												
2. Get the material	■	■												
3. Check the availability of the equipment (XRD,SEM,TCLP,AAS & ACM)	■	■												
Submisstion of literiture review		■												
Practical /Laboratoty Work														
Pre test the material - sludge cake, cement and fly ash														
1. TCLP		■	■											
2. XRD		■	■											
3. ACM		■	■											
4. AAS		■	■											
Submission of preliminary report			■											
Practical /Laboratoty Work														
1. Ratio selection				■										
2. Bind the concrete				■										
3. Stabilization process of the concrete				■	■	■	■							
Submission of Progress report								■						
Practical /Laboratoty Work														
Study the result (test concrete)									■	■				
1. TCLP									■	■				
2.TGA									■	■				
3. ACM									■	■				
Preparation and Submission of Dissertation report											■	■		
Preparation for pre EDX											■	■		
Oral Presentation														■

APPENDIX B

LIMIT OF HEAVY METAL DIPOSED TO LANDFILL

2) LANDFILL CRITERIA

Free from bad smell/odour
No presence of fumes/gas
No presence of free liquid/water

Parameter	Limit	Parameter	Limit	Parameter	Limit
Total Solids	>20%	f. Copper	100 mg/L	Chloride	2%
pH Slurry	5.5 - 12	g. Lead	5 mg/L	Oil and	1000 mg/kg
		h. Mercury	0.2 mg/L	Grease	<10%
Leachable Metals	5 mg/L	i. Nickel	100 mg/L	TOC	0.5 mg/kg
	100 mg/L	j. Selenium	1 mg/L	Cyanide	
a. Arsenic	400 mg/L	k. Silver	5 mg/L		
b. Barium	1 mg/L	l. Tin	100 mg/L		
c. Boron	5 mg/L	m. Zinc	100 mg/L		
d. Cadmium					
e. Chromium					

APPENDIX C

TEST RESULT AND DATA

Data

Sample Leaching calculation.

Ratio	leaching1	lecahing 2	Avg	leaching corrected	% immobilize	leaching rate.
A	0.34	0.49	0.415	22.07385	42.44407071	0.20555689
B	0.37	0.22	0.295	15.69105	59.08674906	0.146118753
C	0.37	0.19	0.28	14.8932	61.16708385	0.138688986
D	0.19	0.17	0.18	9.5742	75.03598248	0.089157205
E	0.32	0.38	0.35	18.6165	51.45885482	0.173361233
F	0.29	0.31	0.3	15.957	58.39330413	0.148595342
G	0.21	0	0.105	5.58495	85.43765645	0.05200837
H	0.32	0.41	0.365	19.41435	49.37852003	0.180791

1. Leaching 1 and 2 taken from AAS data.

Leaching corrected

100g sample mixed with 2000ml lechant

Volume sample = mass/density

= 0.1g/1020kg/m3

=98 m3

For concrete, density = total mass/volume

=2658m3

2. % immobilize = $\frac{\text{initial- final (Zn)}}{\text{initial}}$

= $\frac{38.352- 22.07385 \text{ (Zn)}}{38.352}$

42.4%

3. Leaching rate =

$$l = \frac{a_n}{A_0} \frac{V}{S t_n}$$

a_n = *amount of heavy metal leach at n day*
V/S = Volume of the concrete / Area of the surface of concrete. (Vol= 1000cm3, area=10cm2)
A_0 = *Amount of heavy metal before s/s(38.352mg/l)*
t_n = *Curing period(28 days)*

$$= \frac{22.07385*1000}{38.352*100*28}$$