WELL INTEGRITY ASSURANCE IN EOR INJECTION WELLS THROUGH THE LENS OF THERMAL PROPERTIES OF PORTLAND CEMENT

by

NAHVIN JAYARATNAM (10883)

Dissertation submitted in partial fulfillment of the requirements for the
Bachelor of Engineering (Hons.) (Petroleum Engineering)

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CERTIFICATION OF APPROVAL

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Approved by,

Amin

(MR. MOHD AMIN SHOUSHTARI)

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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(NAHVIN JAYARATNAM)

ABSTRACT

Heavy oil production is one of the challenges faced by the Oil and Gas industry today with reserves of trillions of barrels. When considering production, heavy oil viscosity must be reduced to gain mobility and have oil flowing. Between all possible techniques, steam stimulation is the most promising.

Steam stimulation is one of the viable methods to extract heavy oil from oil sand reservoirs. In this thermal process, steam is injected through the well down to the reservoir to warm it up to 320°C hence, subjecting the well to high temperatures. These injectants are pumped from the surface at very high temperatures and pressures and are required to enter the targeted zone with minimal amount of heat loss to the surrounding formation.

The cement sheath in steam injection wells are required to have good thermal insulation properties to minimise the amount of heat loss to the formation while ensuring the integrity and flow assurance of the well.

This research focuses on the thermal conductivity of cement and how the structural properties of cement affect heat transfer from the casing to the surrounding formation. Cement samples cured at varying temperatures are analysed using a Micro Focus X-Ray Computerised Tomography (CT) System thus, allowing the mapping of pores and microfractures within the internal cement structure. Cement cured at elevated temperatures show significant differences in microstructure compared to baseline samples. The thermal conductivity (k-values) of the samples are then measured and correlated to the pore distribution and curing temperatures. This will allow for the modelling of heat flow from the casing to the surrounding formation. The end result of this research should identify the effects of curing temperature on the thermal properties of cement.

ACKNOWLEDGMENTS

I would like to express my hearties gratitude to Mr. Mohd Amin Shoushtari for his supervision during the course of my Final Year Project. His guidance and trust has given me great strength to complete this research. He spent a great amount of time guiding and providing valuable advice on how to overcome the problems and issues faced during the research. His ideas, insight and guidance are very much appreciated.

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Chapter 1: INTRODUCTION

1.1 Background of Study

Well-integrity is an important part of health, safety, environment and quality-assurance programs in the petroleum industry. It is the design, installation, operation and maintenance of all well equipment and installations to a standard that ensures the safe containment of produced well fluids and injectants for the life of the well (Callum Munro, 2004).

Cementing is an important component of well integrity as it is part of the barrier system in a Tubing/Casing Integrity Management System. In EOR injection wells, the cement and casing systems are required to withstand high temperatures and high pressures from the formation as well as from the injectants. The casing and cements are supposed to allow for the flow of fluids (injectants) from the surface to the targeted zone without compromising on the integrity of the well. These injectants are pumped from the surface at very high temperatures and pressures and are required to enter the targeted zone with minimal amount of heat and pressure losses to the surrounding formation.

However, there is always a certain amount of heat which will be transferred from the casing to the surrounding formation. This is because the temperature in the wellbore is much higher compared to the temperature of the surrounding formation. The dispersion of heat from the wellbore to the formation will pass through the cement sheath. The cement is required to have good thermal insulation properties to minimise the amount of heat lost to the formation (Dwight K. Smith, 1987). It must also be able to ensure the integrity of the casing.

This research will focus on the thermal conductivity of cement and how the structural properties of cement affect its thermal conductivity. The structural properties of cement will vary based on the setting and curing time and temperatures of the cement. The end result of this research should identify the effects of curing temperature on the thermal properties of cement.

1.2 Problem Statement

The use of suitable cements and cementing practices is essential in an EOR injection well. Thermal insulation properties of cement should be good in order to minimize heat loss to the surrounding formation. This will ensure the injectant reaches the targeted formation at the required temperature. The natural thermal insulation properties of cement are due to the formation of pores and microfractures within the cement structure during the hydration of cement at high temperatures. These structural features decelerate the heat transfer between the wellbore and the formation. Hence, varying cement curing temperatures affect the thermal conductivity of cement.

1.2.1 Problem Identification

In EOR injection wells, the heat of injected fluid is lost to the surrounding formation while flowing down through the wellbore to the targeted injection zone. This causes the injected fluid to arrive at the targeted zone with a temperature much lower than expected and this directly affects the performance of the EOR. This heat loss, may also cause loss of circulation and affect the integrity of the well.

1.2.2 Significance of Project

The aim of this research is to study the thermal conductivity of cement cured under varying elevated temperatures. This research is significant as there are very limited resources that focus on the thermal properties of cement particularly thermal conductivity. No existing research has been undertaken on the effects of cement structure on the thermal properties of cement. This research will provide an insight into how cement curing temperatures can affect the thermal properties of cement. This is crucial in the estimation of heat loss to the surrounding formation in EOR injection wells. Injectants in EOR wells can then be prepared at higher temperatures to counter the heat loss to the formation.

1.3 Objectives

There are several objectives to be achieved when completing this project. The objectives are:

1. Identify the structural features (pores, microfractures) of cement that ensure thermal insulation.

2. Qualitatively analyze how cement curing temperature affects the development of pores and microfractures within the cement microstructure.

3. Relate the volume of void space (pores, microfractures) in the cement structure to the thermal conductivity of cement.

4. Analyse and chart trends on how the thermal conductivity of cement is directly affected by the curing temperatures of cement.

1.4 Scope of Study

This research will involve the understanding of heat transfer and thermal conductivity. These topics are related to the Mechanical Engineering course, Thermodynamics. It will also involve the study of cement cured at elevated temperatures and how this affects the development of pores and microfractures within the cement structure. The volume of pore spaces (porosity) and pore distribution within the cement structure is then related to the thermal insulation properties of the cement. The hypothesis for this research is the higher the porosity, the lower the thermal conductivity of cement. The scope of this research will be limited to the thermal conductivity/insulation properties of cement. It will not cover the strength of cement.

1.5 Feasibility of the Project within the Scope and Time Frame

The time allocated for this research was 20 weeks. The first step in this project was getting an introduction to the related topics by reading books, journals and research papers. All relevant research papers and journals were available either online (OnePetro) or was requested from the UTP Information Resource Centre. Research was done in order to understand better, the thermal conductivity properties of the cement mortars. The process of understanding the properties of cement that affects heat transfer took 2 month. Although cement mortar fabrication equipment were available at the Concrete Laboratory at Block 14, cement mortar fabrication took about a month due to limited and shared facilities with the Civil Engineering Department. The study and analysis of the cement microstructure using the Micro Focus X-Ray Computerised Tomography (CT) System took 2 weeks. The cement mortars were sent to the Physical Test Laboratory at Malaysian Palm Oil Board (MPOB), Bangi for the thermal conductivity test to obtain the k-values. This process took about a month to complete. Analysis of the results took an additional week.

CHAPTER 2: Literature Review

2.1 Well Integrity

Well-integrity is an important part of health, safety, environment and quality-assurance programs in the petroleum industry. It is one of the main assets of oil and gas producers. It is not a matter of chance; it is a matter of choice. It is not a thing to wait for; it is a thing to be achieved. Well integrity requires the protection of an investment as it covers the safety of the people, equipment and the business.

Well integrity is defined as the design, construction, installation, operation, maintenance and abandonment of a well to a standard that ensures the safe containment and control of produced well fluids and injectants for the life of the well (Callum Munro, 2004). In simplified terms, it means keeping the hydrocarbons in the pipe and the prevention of uncontrolled flow of fluids (Gunnar Andersen, 2006). NORSOK D-010 defines well integrity as the application of technical, operational and organizational solutions to reduce risk of uncontrolled release of fluids throughout the lifecycle of the well.

A typical Well Integrity Life Cycle consist of 5 phases (Joe Anders, 2008):

Design → Construct → Operate → Maintain → Abandon

In each phase, well integrity is vital to ensure the well is operated and maintained in a safe manner to save money and keep up production. While designing a well, it is practical to look at its planned life cycle, keeping in mind that the well will be abandoned in the future. A well can start production as a high-pressure reservoir; then, later in life, it may require a gas lift, while at a later stage, it may serve as an injector before it is abandoned. In addition, there might be plans for sidetracks. Some wells may have a lifetime of a hundred years, including possible sidetracking out of old casings and a final abandonment that lasts "forever."

Well integrity is a growing concern in the industry. Many fields are maturing and new reservoirs are getting far more complex. Some of the well integrity challenges currently being faced include (SINTEF Petroleum Reaserch, 2010):

1. Aging infrastructure
2. Increasing number of subsea wells
3. Smaller reservoir targets
4. More complex wells with multiple legs
5. New technology and downhole equipment
6. Reservoir with multiple pressure regimes
7. Reusing well infrastructure while changing well applications
8. Well life exceeds design life
9. HPHT injection and production wells

Well Integrity Management (WIM) addresses some of the challenges during the drilling and completion phase of a well. A typical Well Integrity Management (WIM) system consists of the following (Gunnar Andersen, 2006):

1. An Accountable Person
2. Qualified Well Operating Procedures
3. Qualified Intervention Procedures
4. Tubing/Casing/Annulus Integrity Management and Monitoring Program
5. Well Head and Tree Maintenance and Testing Policy
6. A Downhole-Safety-Valve (DHSV) Program
7. A Drilling and Well Operations Policy.

Based on our research on the effects of curing temperature on the thermal properties of cement, cement is considered as a barrier system within the Tubing/Casing/Annulus Integrity Management and Monitoring Program. NORSOK D-010 states that cement is required to provide a continuous, permanent and impermeable hydraulic seal along the hole of the casing annulus or between casing strings. It is required to prevent flow of formation fluids, resist pressures from above and below and support the casing and liner strings structurally. For EOR Injection Wells, cement is required to have good thermal insulation properties to ensure the amount of heat loss of the injectants on the way down the well is kept to a minimum. This will ensure the integrity of the well is not compromised.

2.2 The Mechanism of Cement Hydration

The mechanism of cement hydration is an interaction of cement with water. Hydration causes the dissolution of anhydrous compounds to their ionic constituents, the formation of hydrates (hydroaluminates, hydrosilicates, hydrogarnets) in solution, and their eventual precipitation due to their low solubility.

The hydration rates of individual clinker materials are dissimilar. The quickest to enter into reaction is tricalcium aluminate, with tetracalcium alumoferrite coming next and bellite (dicalcium silicate) undergoing hydration at the slowest. Cement in which bellite prevails undergoes hydration at a much slower rate than allite (tricalcium silicate) cement. Gypsum present in cement rapidly dissolves and interacts with tricalcium aluminate, forming calcium hydrsulphoaluminate at standard conditions.

When cement is mixed with water, the surface layers of the cement grains enter into reactions. This causes a supersaturated solution to emerge from which a gel-like mass of crystals precipitates. The internal layers of the cement grains get coated with little permeable gel films. This initial stage of hydration proceeds very intensively, with the liberation of large amounts of heat. Once the film has been formed, the hydration process slows down materially. Only through diffusion, water can gain access to the internal layers of the cement grains that did not partake in the initial reaction.

After sometime, the film breaks up and the internal grain layers become exposed. This causes the hydration rate to increase once again. This is attended by an intensified evolution of heat. The cracking of the surface films occur under the effect of a high osmotic pressure that arises as a result of the liquid phase supersaturation in the pore space of the film. This second period of intense hydration continues until the particles get coated with gel films. During this second period of setting, the cement paste loses its plasticity and becomes more fragile. It is at this point the cement slurry starts its change from a true hydraulic fluid that transmits full hydrostatic pressure to a solid set material that has measurable compressive strength. The cement mass starts gaining strength, while its hardness increases to a degree where it acquires a lithic structure and becomes almost fully water impermeable.

The hydrated compounds that emerge during the first stage of hydration are unstable (metastable). The fibrous crystals of the gel particles themselves occupy a larger volume and contain more water than in a stable state. With the passage of time, the excess water is released from the gel. With hardening proceeding in dry air, shrinkage of gel is observed, while in humid environment no shrinkage (contraction) occurs and the reduction in the volume of gel due to the release of water is compensated for by a continuous hydration of the cement grain layers which did not react to water. Hence, the unstable hydrated new growths gradually turn into stable forms.

The composition of hydroxides appearing upon hydration and their quantitative proportions are in a great measure dependent on temperature. With rising temperatures, it is not only the water content in hydroxides that changes, so does the ratio of $CaO/SiO2$ and even the shape of the crystal framework.

The hydration reaction rate exercises a material influence on the setting and hardening time of the cement paste. It increases with mounting temperature, pressure, fineness of cement grinding and depends on the volumetric water-cement ratio, the cement composition and that of the water electrolytes.

The strength of cement stone depends on the degree of cement hydration. The early strength of stone is determined by allite that is liable to undergo a fairly quick hydration. Bellite however is hydrated at a much slower pace, but continuously. It also plays a vital role in influencing the strength of the cement stone. The strength of the stone augments most intensively during the first 2-3 days of hardening. But since the hydration of cement does not terminate over this period of time, the stone gathers strength over a period of several weeks and even months. With rising temperature, all reactions proceed at a greater speed. The cement slurry sets quicker and the stone acquires its final strength at an earlier date. At elevated temperatures, the final strength of the stone will be determined by bellite.

2.3 The Effects of Curing Temperature on Cement Hydration Rate

The chemistry of cement and its hydration is still not thoroughly understood. Portland cement is a solid solution of the components tricalcium silicate, dicalcium silicate, tricalcium aluminate, tetracalcium aluminoferrite, calcium oxide (free lime) and magnesium oxide (magnesia). 4.0% of gypsum is also added as a retarder.

The hydration products of cement have a more complex chemistry than the clinker. The main hydration product at normal temperature is calcium hydrate of unknown composition with variable water content, a calcium sulphoaluminate and lime. At temperatures above 120°C, the reactions result in different hydration products.

With mounting temperatures, the hydration process proceeds at a greater rate. The cement slurry sets quicker and the stone acquires its final strength at an earlier date. At the same time, the higher the temperature, the sooner the cement stone starts ageing with a concurrent decline in strength. Furthermore, with an elevated temperature (80°C) the maximum strength of the set Portland cement is below the one at a temperature of 20°C.

The longest gain in strength is seen to occur at above 0°C. In such conditions, the growth of strength can go on for many years. With the temperature exceeding 70°C, the time needed for attaining the peak strength is cut down substantially. If at temperatures below 70°C the maximum strength is independent of the temperature, at elevated temperatures, the level of peak strength is significantly lower. In other words, the greatest strength of the stone at a temperature of 80°C is lower than at a temperature of 40°C.

The fall of the stones strength at temperatures above 70°C is due to the fact that with the high rate of the hydration process, a smaller number of cement particles have adequate time to undergo hydration by the time the slurry starts hardening in these conditions as compared to a temperature of 40°C. A considerable proportion of cement is hydrated after it has acquired some degree of initial strength. Hydration in a solid state gives rise to the appearance of internal stresses which are conducive to the reduction in strength of the Portland cement.

Mounting temperatures have a great influence on the appearance of pores, microfractures and the increased perviousness of the set Portland cement. As the temperature rises above 80°C, the perviousness of the set cement increases linearly. A particularly sharp growth of permeability is seen with the temperature climbing to a level above 100-110°C. At 120°C, the permeability of a 24 hour old Portland cement stone is roughly ten times that of a stone formed of the same mixture at 60°C. Causes underlying the increase of permeability with rising temperatures are basically the same as those responsible for reduced strength. The heat resistance of cement stone is judged by the nature of changes in the strength and permeability of it in the course of a protracted storage at elevated temperatures.

Pressure as a rule has little effect on the strength and permeability of Portland cement.

2.4 Thermal Conductivity

Thermal conductivity is a material property for which the values depend on the chemical composition, porosity, density, structure, and fabric of the material (Jumikis, 1966). Thermal conductivity is used to determine heat flow through a material.

The coefficient of thermal conductivity, k [W/(m·K)], is a measure of the rate q (W) at which heat flows through a material. It is the coefficient of heat transfer across a steady-state temperature difference ($T_2 - T_1$) over a distance ($x_2 - x_1$), or

$$q = k\,(\Delta T/\Delta x)$$

Thermal conductivity can be measured by transient heating of a material with a known heating power generated from a source of known geometry and measuring the temperature change with time. The method assumes isotropic materials.

For a full-space needle probe, the length L can be assumed to be infinite and the problem is reduced to two dimensions. Given the resistance R of a looped wire in a needle, the generated heat is

$$q = 2\,i^2\,R / L,$$

where *R/L* is the resistance of the needle per unit length. At any time, *t* after heating has started, the temperature *T* is related to the thermal conductivity *k* by

$$T = (q / 4\pi k)\ \ln(t) + C$$

where *q* is the heat input per unit length and unit time and *C* is a constant. A simple way of calculating the thermal conductivity coefficient *k* is by picking T_1 and T_2 at times t_1 and t_2, respectively, from the temperature versus time measurement curve (ASTM, 1993):

$$k_a(t) = q / 4\pi\ [\ln(t_2) - \ln(t_1)] / (T_2 - T_1)$$

$k_a(t)$ is the apparent thermal conductivity because the true conductivity, *k*, is approached only by a sufficiently large heating duration. This method assumes that the measurement curve is linear and ignores the imperfections of the experiment expressed in the constant *C* (Peter Blum, 1997).

In practice, the correct choice of a time interval is difficult. During the early stage of heating, the source temperature is affected by the contact resistance between the source and the surrounding material. During the later stage of heating, the boundary effect of the finite length of the source affects the measurement. The position of the optimum interval generally differs from measurement to measurement (Peter Blum, 1997).

CHAPTER 3: METHODOLOGY

3.1 Research Methodology

The assessment on the effects of curing temperature on the thermal conductivity of cement will be based on several studies and experiment conducted on fabricated cement mortars. The research methodology and project workflow is listed in the flow chart below:

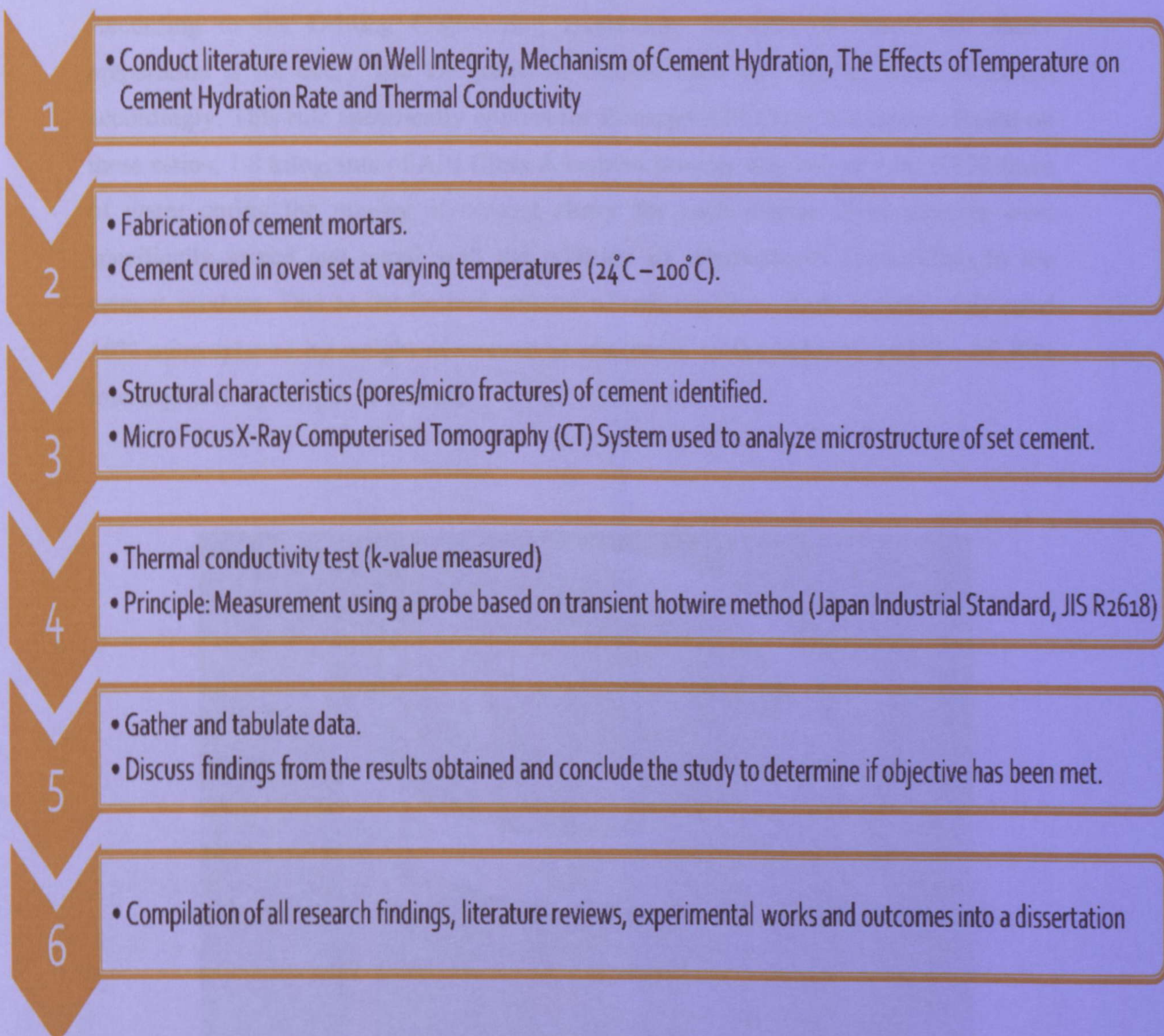


Figure 1: Research Methodology Flow Chart

3.2 Experimental Setup

The experimental setup involves the fabrication of the cement mortars cured at varying temperatures. The mixing of cement slurry and curing at elevated temperatures was carried out by utilizing facilities at the Concrete Laboratory in Block 14. Due to limited resources, Portland API Class A Cement was used instead of the industrial standard, API Class G Cement. API Class A is equivalent to regular construction cement.

According to the Drilling Engineering Handbook, the rule of thumb for slurry preparation is for every 100 kilograms of cement used 46 litres of water is mixed accordingly. This rule specifically applies for Portland API Class A Cement. Based on these ratios, 1.8 kilograms of API Class A cement powder was mixed with 0.828 litres of water during the mixing of cement slurry for each mortar. Two mortars were specifically mixed and cured with the addition of microspheres (Spherelite) to the cement mixture. Due to the limited amount of microspheres, both samples only used 10% microspheres by weight of cement as compared to the industry practice of 20% microspheres by weight of cement.

Figure 2: *Weighing of Cement Powder (Weight of Cement + Container = 1954.3 g)*

Figure 3: *Cement powder transferred into mixing bowl*

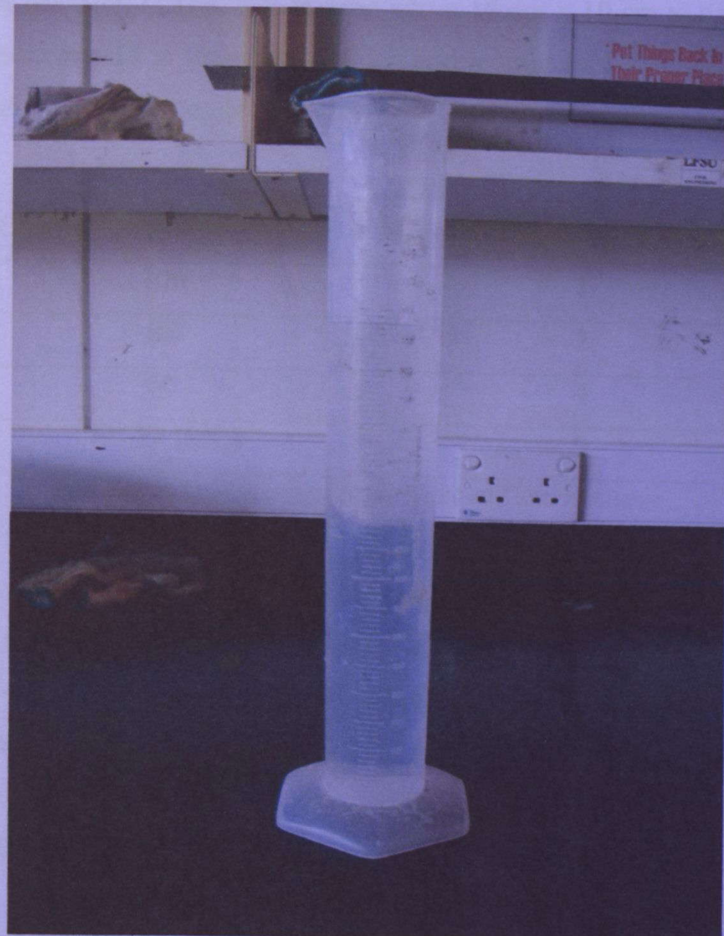


Figure 4: *0.828l of water measured in a measuring cylinder*

After water is poured into the mixing bowl containing cement powder, the cement is immediately mixed for approximately 5 minutes using a Cement Mortar Mixer.

Figure 5: *Mixing of Cement (1.8 kg) and Water (0.828l)*

Once the cement has been mixed, it is poured into a 10 cm x 10 cm x 10 cm cast iron mould. The cement mould is then vibrated for approximately 1 minute before being transferred into the oven. The cement mortars were cured as follows:

Curing Temperature (°C)	Minimum Setting Time (hours)
100	1
80	2
60	4
24 (S.C)	9

Table 1: *Minimum Setting Time of Cement Slurry*

The table above shows the minimum setting times for the cement mortars. All the cement mortars fabricated were allowed to set according to their respective minimum setting times. However, some mortars were allowed to be cured for longer durations than the minimum setting times. This was to see if the additional setting duration affected the structural properties of cement. The use of the oven was limited to a maximum of 100°C and 24 hours due to Laboratory Rules and Regulations.

After, the cement mortar's setting time had lapsed, the cement mortars were removed from the oven and were allowed to cool at room temperatures for approximately 3 to 4 hours before they were removed from their moulds.

Figure 6: *Cement Mortars cooling at room temperature*

From the cement mortars fabricated, 11 samples were chosen for the experiment stage:

Samples		
No.	**Curing Temperature (°C)**	**Curing Time (hrs)**
1	24 (Standard Condition)	–
2	60 – I	4
3	60 – II	4
4	60 – III	4
5	80 – I	2
6	80 – II	17
7	80 – III	17.5
8	100 – I	18.5
9	100 – II	19
10	60 (Microspheres)	8
11	80 (Microspheres)	8

Table 2: *Sample Details*

3.3 Experiment 1: Analysis of Cement Microstructure

The cured cement mortars were analysed using a Computerized Tomography (CT) Scanner. This was done by utilizing the Micro Focus X-Ray Computerized Tomography (CT) System at Block 15. This system allowed for the analysis and study of the internal structure of the cement mortars. The cement mortars were divided into 3 different layers; top, center and bottom. The size and distribution of pores and microfractures at each layer were qualitatively analyzed. Scan images clearly showing pore distribution at each layer were saved as image files to be included in the dissertation.

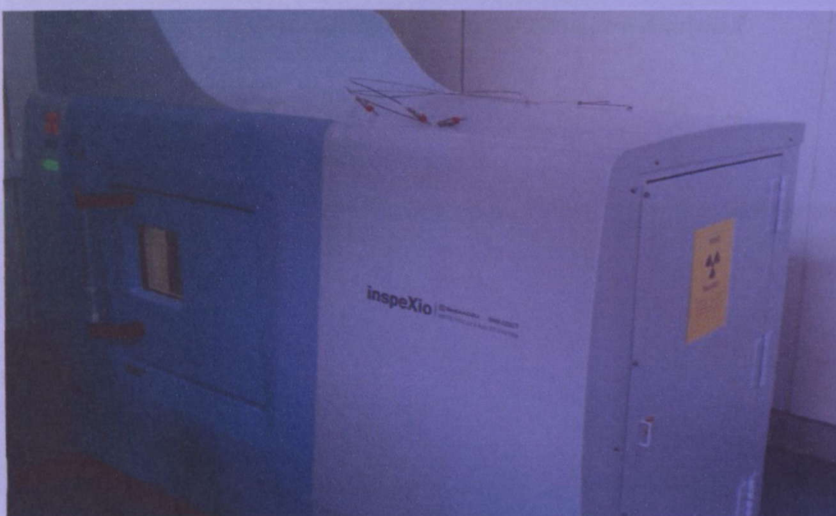


Figure 7: *Micro Focus X-Ray Computerized Tomography (CT) System*

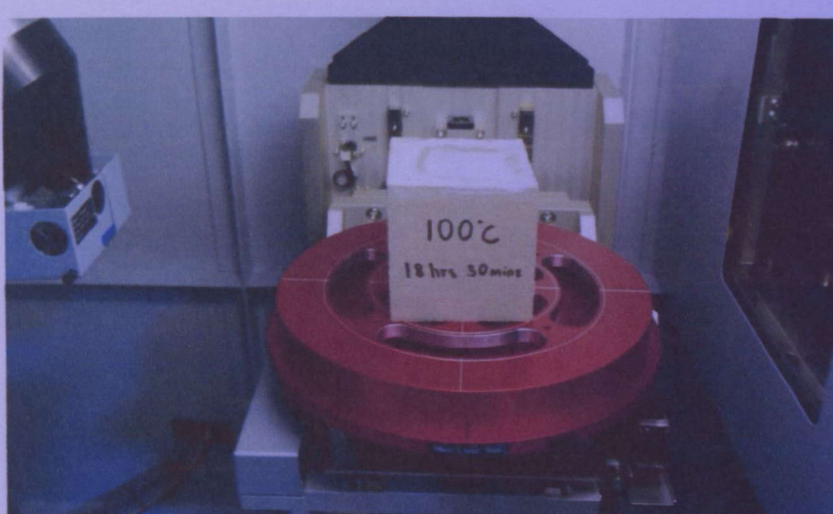


Figure 8: *Cement Mortar being scanned*

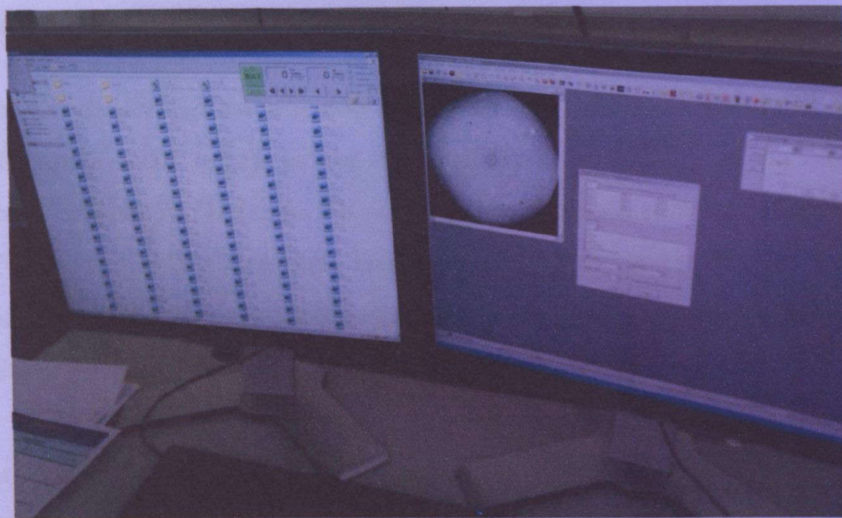
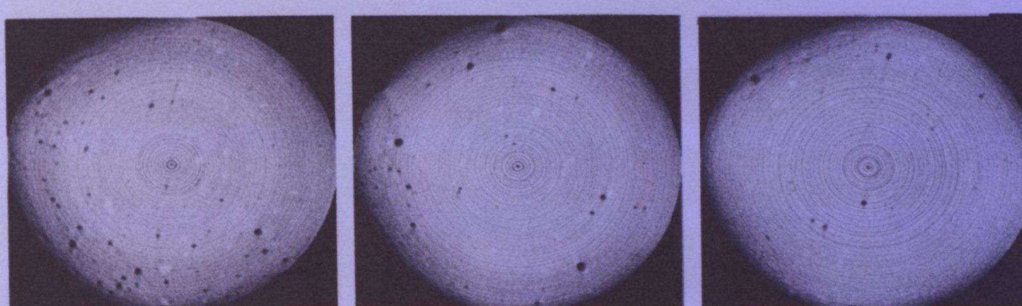


Figure 9: *Scanned Image of Cement Structure Analysed*



Top Center Bottom

Figure 10: *Images of Top, Center and Bottom of Cement Structure Saved*

3.4 Experiment 2: Thermal Conductivity Test (Determination of k-values)

The Thermal Conductivity Test was carried out by the Malaysian Palm Oil Board at Bangi. 11 Samples were tested at the Physical Test Laboratory in the Advanced Oleochemical Technology Division. The samples were tested at a test temperature of 29°C to 33°C under ambient moisture. Thermal conductivity measurements were done using a probe based on the transient hotwire method in accordance with the Japan Industrial Standard, JIS R2618. The thermocouple is attached to the surface of a rectangular probe. The following procedures were undertaken while measuring the thermal conductivity (k-value) of the samples:

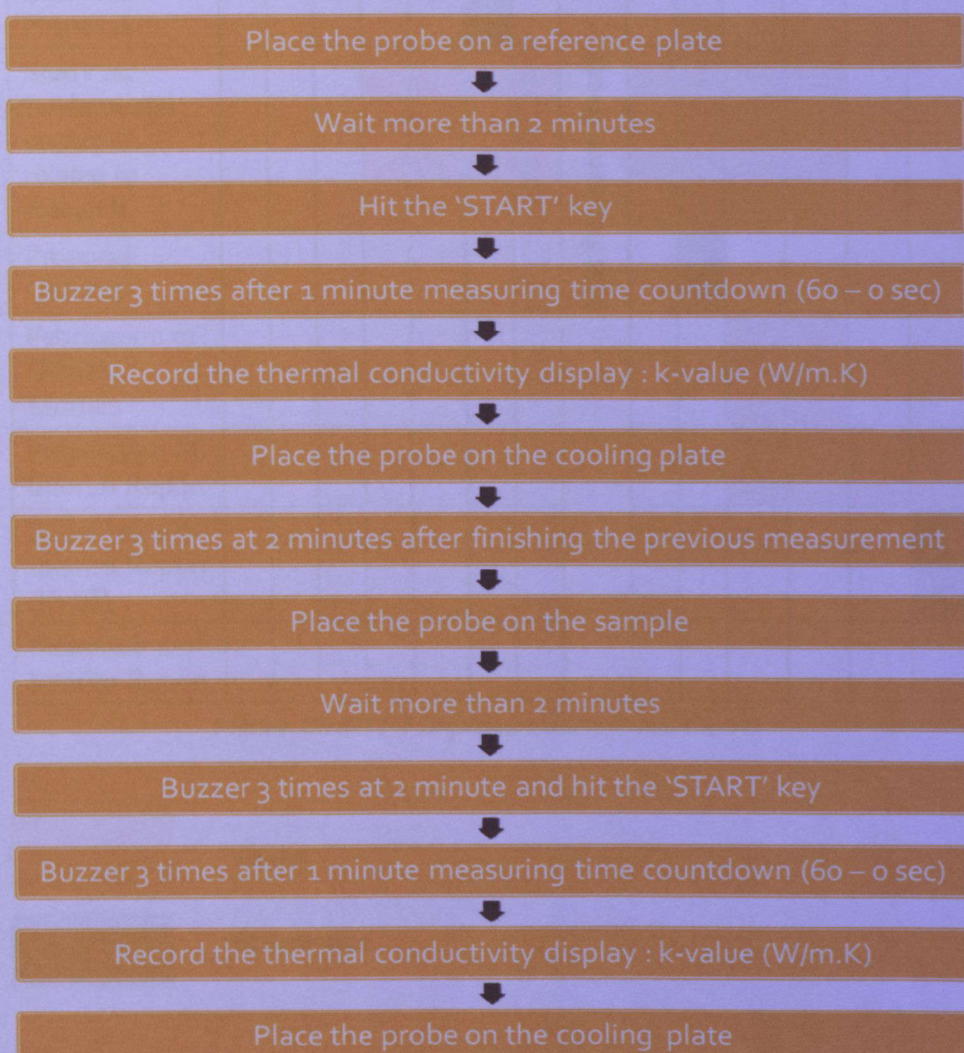


***Figure 11:** Thermal Conductivity Test Procedure*

The k-values were then correlated to the pore distribution of the samples based on their respective curing temperatures. This correlation is subsequently analyzed.

3.5 Gantt Chart and Key Milestone

September 2011 – January 2012

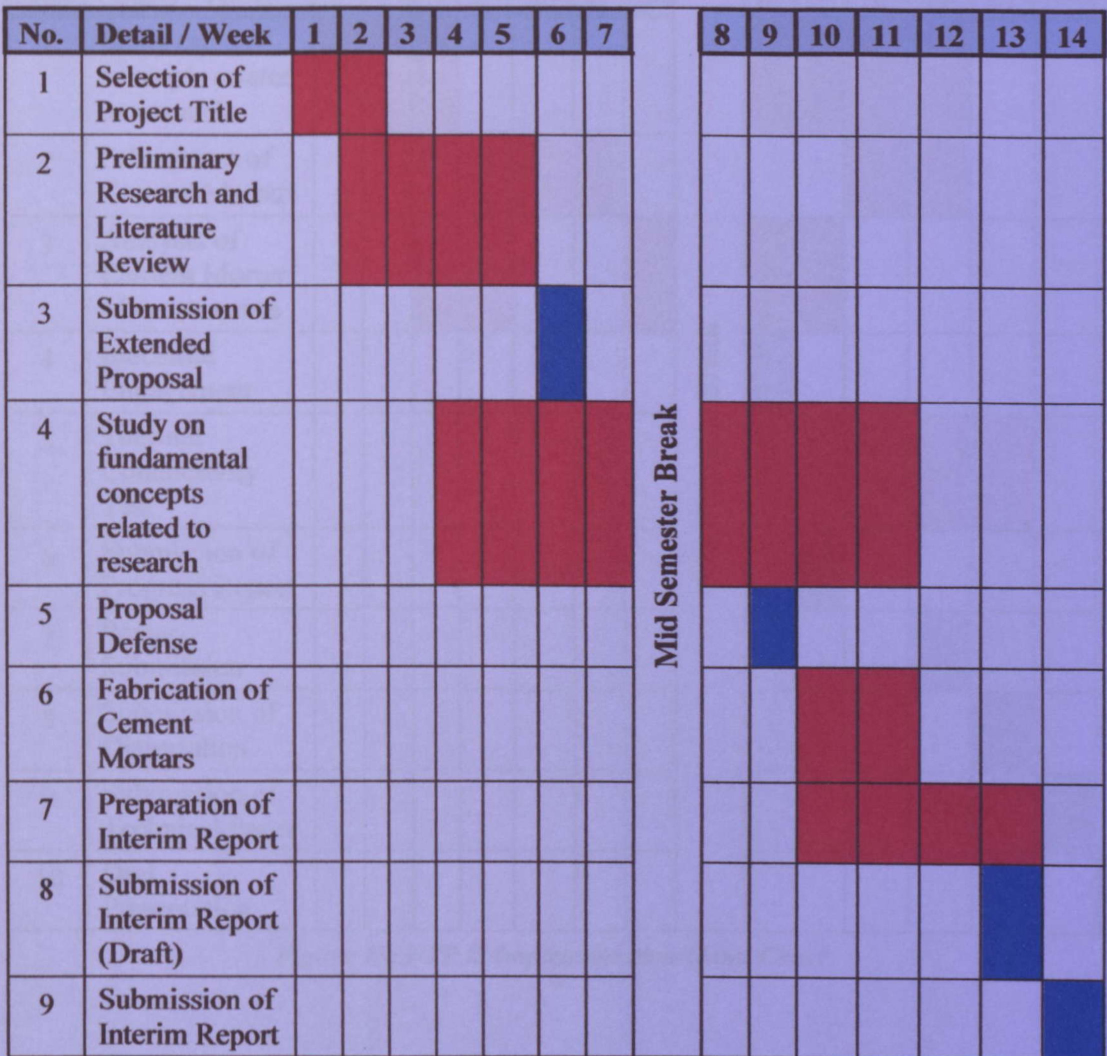
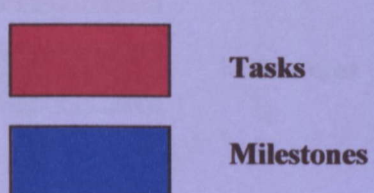


Figure 12: FYP I Implementation Gantt Chart



January 2012 – May 2012

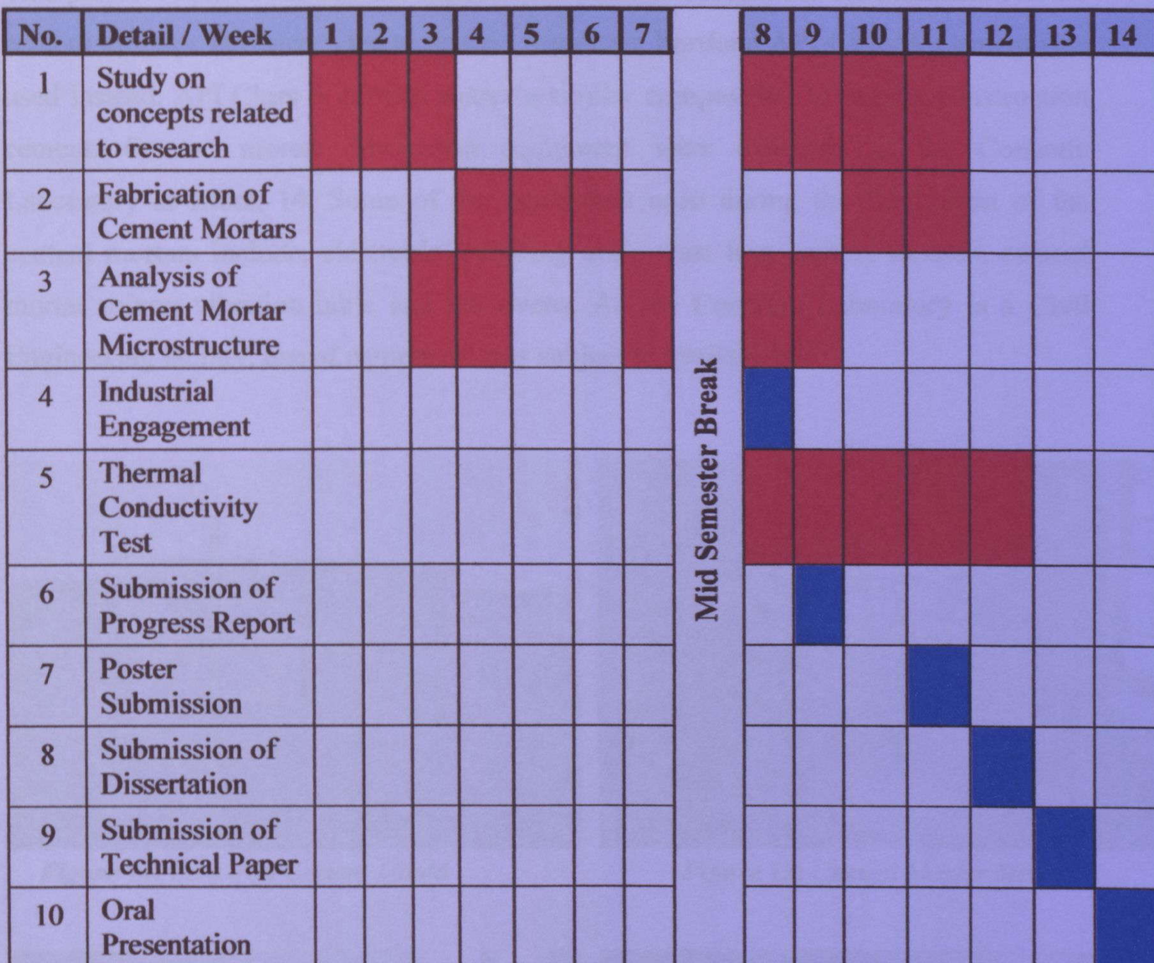


Figure 13: FYP II Implementation Gantt Chart

3.6 Equipment & Tools Utilized

This project required the use of Portland API Class G Cement for the fabrication of the cement mortars. However, due to limited resources, Portland API Class A Cement was used instead. API Class A cement shares a similar composition to regular construction cement. Cement mortar fabrication equipment were available at the Concrete Laboratory at Block 14. Some of the equipment used during the fabrication of the cement mortars include, electronic weighing scale, cast iron cement moulds, cement mortar mixer, vibration table and lab ovens. As the Concrete Laboratory is a Civil Engineering facility, use of equipment was subject to availability.

Figure 14: Cast Iron Cement Mould

Figure 15: Cement Mortar Mixer

Figure 16: Vibration Table

Figure 17: Laboratory Oven

The fabricated cement mortars were analysed using a Micro Focus X-Ray Computerized Tomography (CT) System. The Shimadzu inspeXio SMX-225CT is a top-of-the-line Micro Focus X-ray CT system, which features a high precision CT stage and sophisticated image processing software. This system is ideal for non-destructive testing and analysis of core and cement microstructure. This advance equipment belongs to the EOR Centre at Block 15.

Specifications of the equipment are provided in the table below:

X-ray generator	Open tube Max voltage 225 kV, Max current 1000 µA,
Detector	High sensitive Image intensifier, 9 / 7.5 / 6 / 4.5 inch selectable
Mountable sample	300 dia. x 300 mm H, 9 kg
CT field of view (FOV)	X Y direction 5 to 200 mm dia Z direction 4.5 to 100mm
CT image size	512X512 1024X1024 2048X2048 4096X4096

Table 3: *Shimadzu inspeXio SMX-225CT Micro Focus X-Ray CT System Specification*

Figure 18: *Shimadzu inspeXio SMX-225CT Micro Focus X-Ray CT System*

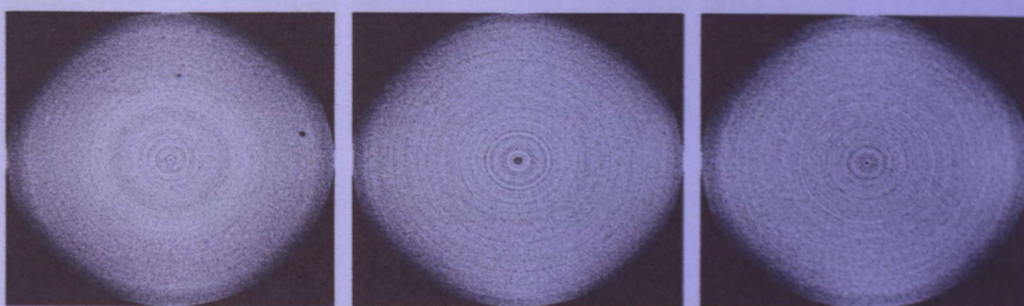
The thermal conductivity test (measurement of k-value) on the cement mortars were carried out at the Physical Test Laboratory, Malaysia Palm Oil Board (MPOB) in Bangi. The tests were conducted using a KEMTHERM QTM-D3 measuring equipment by Kyoto Electronics Manufacturing Co. Ltd. This equipment utilizes a probe based on the transient hotwire method. It makes thermal conductivity measurements by keeping a probe on any reasonably flat surface for 60 seconds. Measurable test temperatures of the KEMTHERM QTM-D3 ranges from –10°C to 200°C. The k-values obtained are in SI units: W/m.K.

CHAPTER 4: RESULTS AND DISCUSSION

4.1 Qualitative Analysis of Cement Mortar Cured at Elevated Temperatures

The internal structure of all cured cement samples were analysed using a Micro Focus X-Ray Computerised Tomography (CT) System. CT Scan Images at the top, center and bottom of the samples were captured and saved. Pore distribution is represented by the black spots in the image.

Cement mortar cured at Standard Condition (24°C)

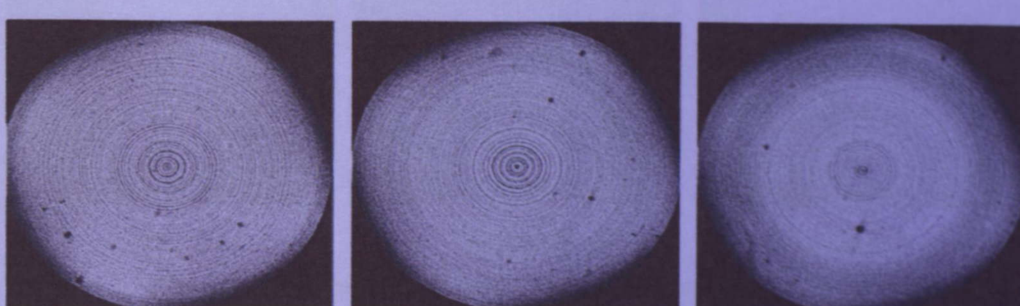


Top Center Bottom

Figure 19: *Microstructure images of cement mortar cured at Standard Condition (24°C)*

The cement mixture for this sample was poured and cured at a room temperature of 24°C.It shows very limited pore distribution. There are a few noticeable pores at the top of the cement. This is due to the upward movement of excess water due to difference in density between water and the cement mixture.

Cement mortar cured at 60°C

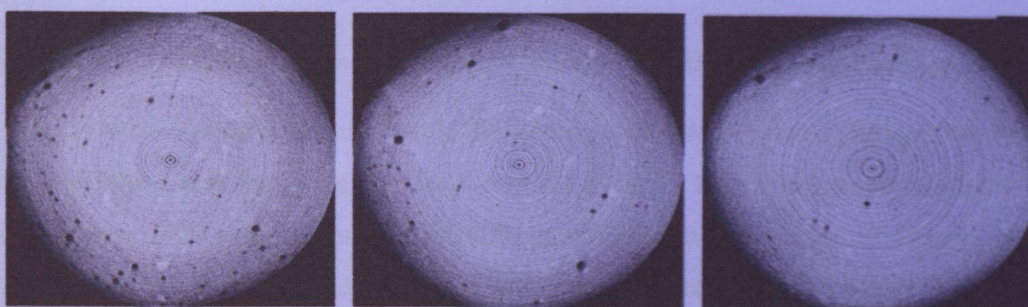


Top Center Bottom

Figure 20: *Microstructure images of cement mortar cured at 60°C*

The cement cured at 60°C shows more pore distribution compared to the cement cured at room temperature. It is noticeable that pore distribution within the cement structure is higher at the top and gradually decreases towards the bottom. This is due to the advent of capillary effect when trapped water particles move upwards during the setting of cement. This causes the formation of pores within the set cement.

Cement mortar cured at 80°C

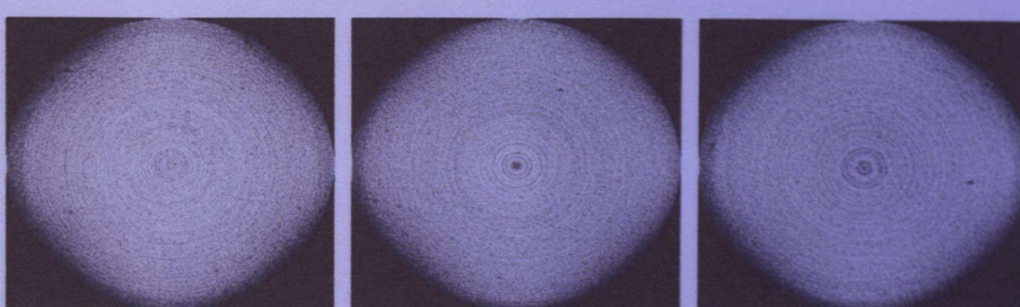


Top Center Bottom

Figure 21: Microstructure images of cement mortar cured at 80°C

The cement cured at 80°C shows similar pore distribution characteristics to the cement cured at 60°C. However, pore count is noticeably higher compared to the sample cured at 60°C. Causes underlying the increase in porosity at the top of cement structure compared to the bottom are basically the same as the sample cured at 60°C

Cement mortar cured at 100°C



Top Center Bottom

Figure 22: Microstructure images of cement mortar cured at 100°C

The cement cured at 100°C shows very limited pore distribution as opposed to the trend set by the samples cured at 60°C and 80°C. This sample was supposed to show a much higher pore count compared to the previous two samples. The reason as to why there is very limited pore distribution is because all excess water particles evaporate very quickly at 100°C. This happens even before the cement has had enough time to harden. However, there may still be pores which are minute in size that were not captured by the CT Scan System. We will analyse the thermal conductivity test of this sample in the following part and compare it to the other samples.

4. 2 Qualitative Analysis of Cement Mortar Cured with Microspheres (Spherelite)

Two samples were mixed and cured with the addition of microspheres (Spherelite) to the cement mixture, one sample at 80°C and another at 60°C. Due to the limited amount of microspheres, both samples only used 10% microspheres by weight of cement as compared to the industry standard of 20%. It must also be noted that bentonite was not added to the cement mixture to control the separation of microspheres. Hence, the microspheres tend to float in the cement slurry.

The addition of microspheres to the cement slurry causes the cement structure to have a disruption to its continuous phase. There is a distribution of high and low density solid particles. This will affect the thermal conductivity of the cement as heat flows faster through high density material as compared to low density materials.

Cement mortar cured with microspheres (Spherelite) at 60°C

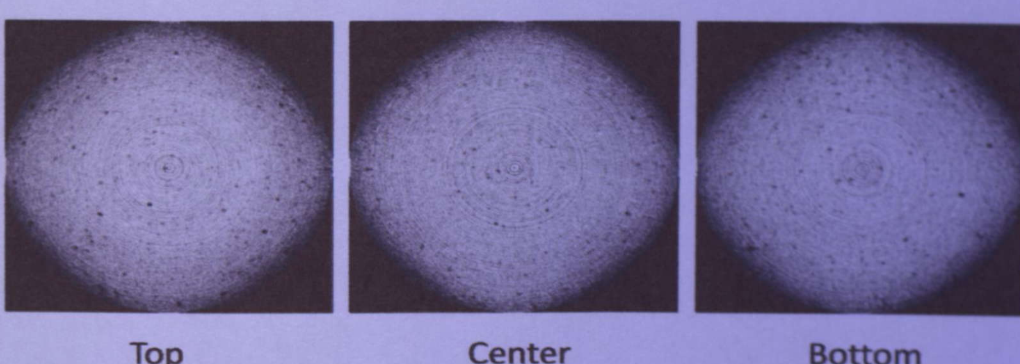
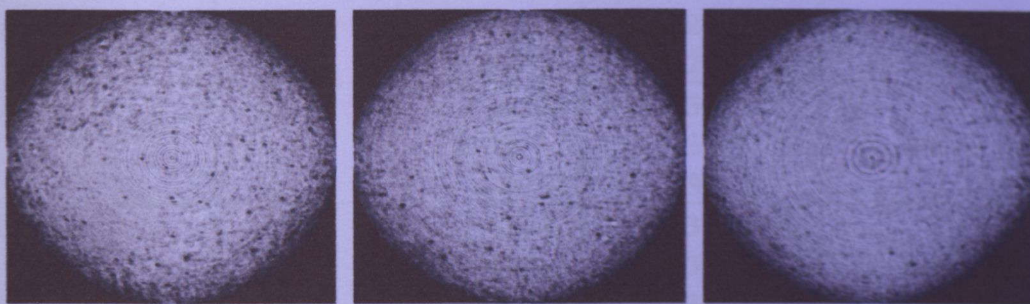


Figure 23: *Microstructure images of cement mortar cured with microspheres at 60°C*

Microsphere distribution in this sample is pretty even along the cement structure. Naturally occurring cement pores can also be observed in the sample with pore distribution being higher at the top and gradually decreasing towards the bottom.

Cement mortar cured with microspheres (Spherelite) at 80°C



Top Center Bottom

Figure 24: *Microstructure images of cement mortar cured with microspheres at 80°C*

Microsphere distribution in the sample cured at 80°C is higher at the top of the sample and decreases towards the bottom. Excess water in this sample has moved upwards after the pouring of the mixture due to the density difference with the cured mixture. This has allowed the low density microspheres to flow with the water to the top of the cement structure. Naturally occurring pores can similarly be observed in this sample with pore distribution being higher at the top and gradually decreasing towards the bottom. The pore count is higher compared to the pore count in the previous sample.

4.3 The Effects of Curing Temperature on the Thermal Conductivity of Cement

Table 4 shows the Thermal Conductivity Test Results for the 11 Samples:

Samples		Thermal Conductivity Test		
Curing Temperature (°C)	**Curing Time (hrs)**	**k-value (W/m.K)**	**Testing Condition (°C)**	**Average k-value (W/m.K)**
24 (Standard Condition)	-	0.9223 0.9101 0.9306	31 29 29	0.921
60 – I	4	0.827 0.8333 0.8301	32 32 31	0.830
60 – II	4	0.8002 0.8055 0.8188	33 31 30	0.808
60 – III	4	0.7774 0.7623 0.772	33 33 31	0.771
80 – I	2	0.7165 0.7106 0.7012	33 31 30	0.709
80 – II	17	0.5869 0.5992 0.6095	33 33 32	0.599
80 – III	17.5	0.6664 0.6591 0.6452	33 30 30	0.657
100 – I	18.5	0.5803 0.5813	31 31	0.581
100 – II	19	0.6143 0.6154 0.6151	32 30 30	0.615
60 (Microsphere)	8	0.5403 0.5405 0.5467	33 32 31	0.543
80 (Microsphere)	8	0.4373 0.4472 0.4347	33 33 31	0.440

Table 4: *Thermal Conductivity Test Results*

Based on the 3 readings of k-values obtained from the thermal conductivity test of each sample, an average k-value is calculated for each sample. The average k-value is then plotted against the respective sample in the form of a graph. The average k-values of the cement mortar with microspheres cured at 60°C and 80°C are included for comparison purposes.

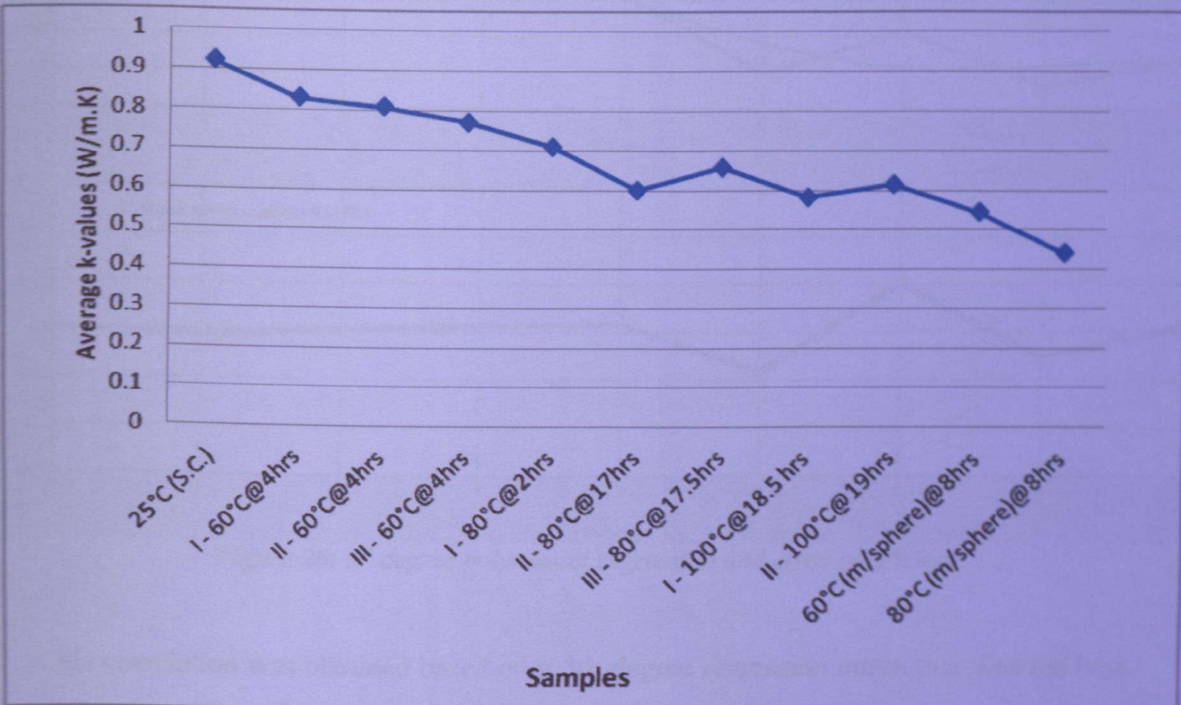


Figure 25: *Average k-values (W/m.K) vs. Samples-Curing Temperature (°C)*

From the graph above, it can be observed that the k-value (W/m.K) decreases with increasing cement curing temperatures. As the cement curing temperature increases, the appearance of pores within the cement structure increases. This directly affects the heat transfer across the cement sheath due to the reduced density within the cement phase. A higher density solid phase has a higher thermal conductivity (k-value) compared to a lower density solid phase. Hence, as the pore count within the cement sheath increases with increased curing temperature, the thermal conductivity of the cement is greatly reduced.

Utilizing the MATLAB Software, the Average k-value vs. Samples-Curing Temperature (°C) graph was polynomially regressed. The following correlation was obtained:

$$k_{avg} = 0.0002\ T^5 + 0.0053\ T^4 - 0.05\ T^3 + 0.22\ T^2 - 0.45\ T + 1.2$$

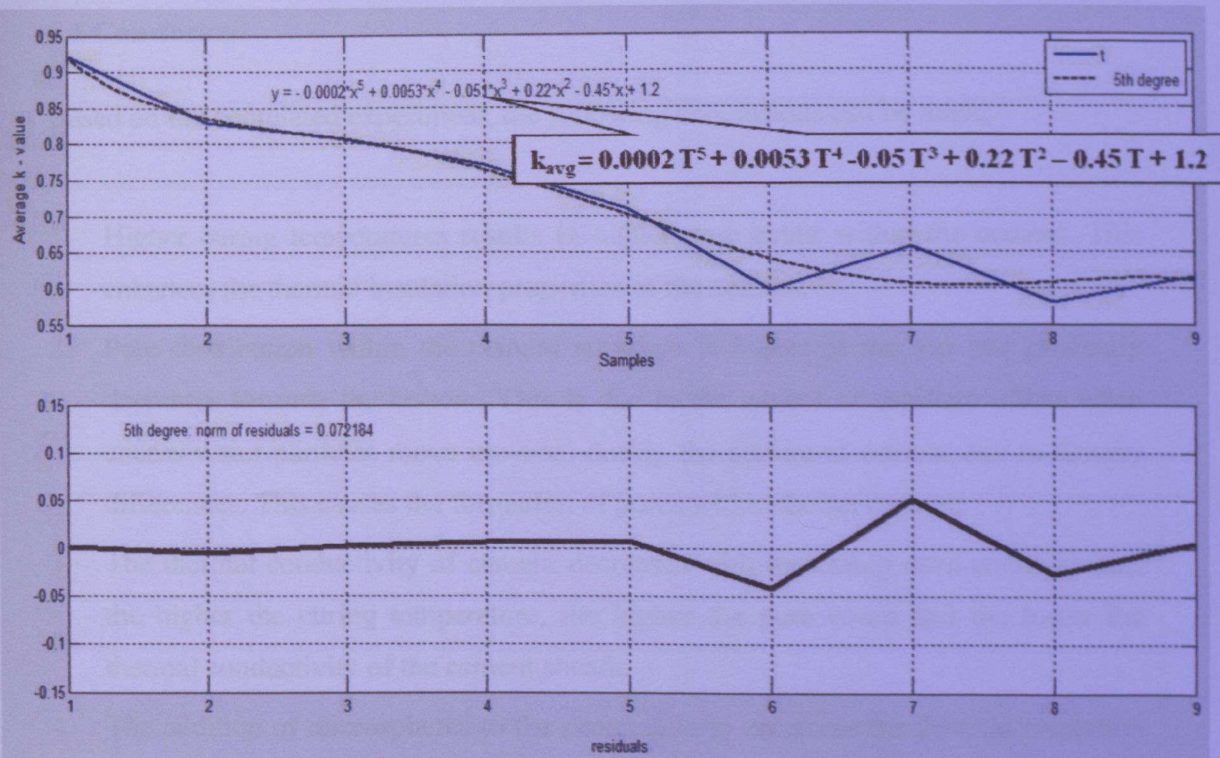


Figure 26: 5th degree polynomial regression and error coefficient

This correlation was obtained based on a 5th degree regression curve that was the best fit for the Average k-value vs. Samples graph. This correlation has an error coefficient of 7.218 %.

It must be noted that the samples cured with microspheres were omitted from this correlation as the samples and test results were inadequate to be correlated. However, from the average k-values calculated for both samples, it can be noted that cement mortars cured with microspheres share a similar trend with the normal cement mortars, with the increase of curing temperature reducing its thermal conductivity. Thermal conductivity values for these samples are also much lower compared to the normal cement samples as the microspheres assist with reducing thermal conductivity while enhancing thermal insulation properties of the cement sheath.

Although curing times of the samples varied independently with accordance to the minimum setting time noted in the Experimental Setup, it was not taken into account as a factor that affects the thermal properties of the set cement.

CHAPTER 5: CONCLUSION AND RECOMMENDATION

5.1 Conclusion

Based on the completed experiment, the following conclusions can be made:

1. Higher curing temperatures results in higher pore count within the cement. This enhances the thermal insulation properties of the cement.
2. Pore distribution within the cement structure is higher at the top and gradually decreases towards the bottom. This is due to the advent of capillary effect when excess water particles move upwards during the setting of cement due to density differences. This causes the formation of pores within the set cement.
3. The thermal conductivity of cement decreases with increasing pore count. Hence, the higher the curing temperature, the higher the pore count and the lower the thermal conductivity of the cement sheath.
4. The addition of microspheres to the cement slurry enhances the thermal insulation properties of cement. Curing of cement slurry with microspheres at elevated temperatures further increases the thermal insulation properties. This is due to the reduction in thermal conductivity assisted by the increased pore distribution.
5. Based on the correlation deduced by the MATLAB Software, the average thermal conductivity (k_{avg}) of cement can be estimated. This will allow for the estimation of heat loss to the surrounding formation in EOR injection wells. Injectants in EOR wells can then be prepared at higher temperatures to counter the heat loss to the formation.

5.2 Recommendations

There is still much work that can be done on this project. The following are some of the recommendations that should be taken into account for any future research:

1. Portland API Class G Cement should be used as this is the industrial standard that is used for the cementing of EOR Injection Wells
2. Further analysis can be done for cement mortars cured at elevated temperatures as cement slurries can be cured at temperatures ranging from 24°C to 200 °C to simulate actual formation temperatures.
3. The curing times of cement should also be given significant input as this may affect the structural properties of the set cement. Cement curing times for each curing temperature should vary between the minimum setting times to 3 days.
4. The addition of microspheres to cement mortars should also be considered. Microspheres should be mixed into the cement slurry based on the industrial practice of a minimum 20% by weight of cement. Recommendations 2 and 3 should also be considered during the preparation of cement mortars with microspheres.
5. A quantitative analysis of pore structure within the cement mortars should be done as this will provide a more accurate study of pore space within the cement structure. It is suggested that porosity of the cement mortars be measured using a mercury intrusion porosimeter.
6. Research should also be done on obtaining the optimum balance between thermal insulation properties of cement and cement strength. Increase in thermal insulation of the cement sheath is usually at the cost of cement strength.

CHAPTER 6: REFERENCES

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