

**PORE WALL STABILITY IN CARBONATES UNDER VARYING  
GEOCHEMICAL CONDITIONS**

by

**WAN MOHD SHAFIE BIN WAN IBRAHIM**

Dissertation submitted in partial fulfillment of  
the requirement for the  
**BACHELOR OF ENGINEERING (Hons)**  
**(PETROLEUM ENGINEERING)**

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CERTIFICATION OF APPROVAL

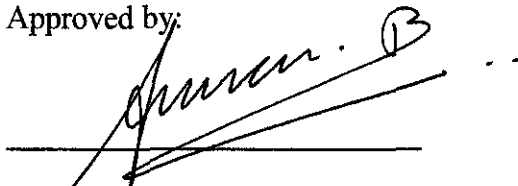
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A project dissertation submitted to the  
Petroleum Engineering & Geoscience Programme  
Universiti Teknologi PETRONAS  
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BACHELOR OF ENGINEERING (Hons)  
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Approved by:



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PERAK, MALAYSIA

January 2011

## **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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**WAN MOHD SHAFIE BIN WAN IBRAHIM**

## **ABSTRACT**

Most of the world's giant fields produce hydrocarbons from carbonate reservoirs. As more than 60 per cent of the world's remaining conventional oil is estimated to lie in carbonate reservoirs, the need to better understand carbonate production capabilities. The objective of this research is to studies the effects on dissolution rates of carbonate by varying the geochemical conditions.

Acid dissolution is one of the important parameters that can affect the porosity and the stability of the rock. Two different samples of carbonate have been selected to complete this project and collected from Sg. Siput (limestone) and Simpang Pulai Quarry (marble). The problem statement, objectives and scope of study will also being discussed in Chapter 1 of this paper. The literature review about carbonate rock has been discussed in Chapter 2.

In Chapter 3, it is about the methodology to be applied in measuring the important parameters of the samples. It focuses more on lab activities such as core sampling, thin section, Microscopy Polarization Image, XRD (X-ray Diffraction), XRF (X-ray Fluorescence), Field Emission Scanning Electron Microscope (FESEM), Thermal Conductivity, Acid Dissolution Experiment and Mercury Porosity Test.

Next, the results of both the test and experiment are discussed in Chapter 4 with the relevant tables and graphs followed by conclusions or summary. At the end, the author cited lists of references and few appendixes.

## **ACKNOWLEDGEMENT**

With full of humble, I thank the Almighty Allah, in which His blessing has allowed me to write this report, and most importantly to finish my final year project.

I would like to thank my supervisor, AP. Dr Eswaran Padmanabhan for all his guidance, careful supervision and advice. Without his contribution and generous sharing, I doubt this project would go this far, as we are currently working on posting this project to any research paper conference. This is all because of the ambitious quality of him.

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**CHAPTER 1: INTRODUCTION****1.1 PROJECT BACKGROUND**

Calcium Carbonate is an important and often dominant of marine sediments. This is reflected by the observation that about 20% by volume of Phanerozoic (0-547 million years ago) sedimentary rock is made up of the carbonate minerals calcite ( $\text{CaCO}_3$ ) and dolomite ( $\text{CaMg}(\text{CO}_2)_2$ ). Much of the calcium carbonate that is formed is dissolved in the water column. Almost all deep sea carbonate-rich sediments are composed of calcite low in magnesium (>99%  $\text{CaCO}_3$ ). This materials is primarily derived from pelagic skeletal organisms, with coccolithophores (plants) followed by forminifera (animals). Interest in calcium carbonates dissolution in the ocean has increased because of the central role these reactions will play in the ocean's response to the increasing partial pressure of carbon dioxide ( $\text{pCO}_2$ ) in the atmosphere. Majority of carbonate minerals deposited in modern marine environments is biogenic in origin; that is, these minerals are actively precipitated from seawater by organisms to form skeletal hard parts. The crystal structure of naturally occurring carbonates, as a group, is relatively simple, particularly when compared with oxides, silicates and other sedimentary minerals. Despite their structural simplicity, the behavior of these minerals in seawater is complex. This complexity primarily reflects the fundamental role of chemical kinetics in this system.

Today, carbonate reservoirs continue to pose a number of challenges. After all, the recovery factor from these reservoirs is below the 35 per cent average for fields of all types and some fractured carbonate reservoirs have recovery factors below 10 per cent. The challenge in coming years will be to integrate data even more effectively to produce ever-more accurate reservoir models, with an even better definition of features. With continued collaboration to refine existing techniques and the commitment to develop specialist new technologies, the future for carbonate reservoirs is looking more productive.

## **1.2 PROBLEM STATEMENT**

### **Problem Identification**

The challenge to the earth scientists' and petroleum engineers' to estimate the effects of varying geochemical conditions on pore wall stability of carbonates. Lack of information about important parameters in carbonates such as mineralogy, solids content, thermal conductivity and its mineral compositions.

## **1.3 OBJECTIVE AND SCOPE OF STUDY**

This project focuses on effects of varying geochemical conditions on pore wall stability of carbonates. . In order to successfully achieve the main objective, the following objectives must be fulfilled;

- To get information about kinetic dissolution of limestone and marble;
- To study about the changes in porosity of limestone and marble during acid dissolution;
- To understand the acidifying trends of hydrochloric acid and formic acid in acid dissolution.

#### **1.4 THE RELEVENCY OF THE PROJECT**

Since, there are a lot of idle well in Malaysia, it is very significant for acid fracturing and matrix acidizing. So, it is an opportunity to do a research that can be related to the effects of varying geochemical conditions on pore wall stability of carbonates in order to increase the production of oil and gas.

#### **1.5 FEASIBILITY OF THE PROJECT**

This project is feasible within the time given to accomplish the Final Year Project course according to the schedule from Gantt-chart.

**CHAPTER 2: LITERATURE REVIEW****2.1 CARBONATES FORMATION****2.1.1 SEDIMENTOLOGY**

Carbonate sediments are particularly sensitive to environmental changes. Carbonate sedimentation is rapid but easily inhibited. Temperature variations influence biogenic activity and affect sediment production; thus most carbonate production is strongly depth dependent. When conditions are favorable for carbonate sedimentation, organic productivity is high; when unfavorable, organic productivity ceases. Carbonates form in special environments and in contrast with sandstones, are biochemical in nature. Environments range from near-shore lagoons, platform organic buildups, and shelf margin shoals to slope and basinal settings. Carbonates typically are found in warm, shallow, clear marine water in low latitudes. Carbonates are essentially autochthonous, as they form very close to the final depositional sites. Texture is more dependent on the nature of the skeletal grains than on external influences. Intrabasinal factors control facies development. In contrast, sandstone and shale were formed of sedimentary particles derived from sources outside the depositional basin. Reefs, bioherms and biostromes are examples of in-place local deposition where organisms have built wave-resistant structures above the level of adjacent time-equivalent sediments. Many reefal deposits are commonly composed of fragmented, locally-transported skeletal debris and a minor volume of in situ framework organisms. Biofacies and lithofacies often correlate, or in other words, organisms produce typical lithofacies. Substrates control inhabiting organisms. Basin configuration and water energy are the dominant controls on carbonate deposition. Organic productivity varies with depth and light (photic zone); upwellings and water agitation influence organic productivity.

### 2.1.2 DIAGENESIS

Carbonates are particularly sensitive to post-depositional diagenesis, including dissolution, cementation, recrystallization, dolomitization, and replacement by other minerals. Abundant unstable aragonite (in bioclasts and cements) converts to more stable low-magnesium (or high-magnesium) calcite. Calcite can be readily dolomitized, sometimes increasing porosity. Complete leaching of grains by meteoric pore fluids can lead to textural inversion which may enhance reservoir quality through dissolution or occlude reservoir quality through cementation. Burial compaction fracturing and stylolithification are common diagenetic effects in carbonates, creating high-permeability zones and permeability barriers or baffles, respectively.

### 2.1.3 RESERVOIR CHARACTERIZATION

Reservoir characterization issues, including the geological framework, rock fabric, facies, and porosity and permeability distributions, are some of the most widely investigated production issues today. Many challenges exist in characterizing, quantifying, and predicting carbonate reservoir quality. The key to understanding carbonate reservoirs is recognizing the critical link between geological heterogeneity and reservoir quality and performance. Finding the link between geological heterogeneity and reservoir quality often becomes a matter of finding the appropriate data and sampling the heterogeneity at the appropriate scale. For example, many carbonates are characterized by abundant macrofauna and macroflora that are larger than the scale of some sampling methods, such as 1" core plugs, and may be better sampled with longer, whole cores or wireline logs. In this case, geological heterogeneity must be sampled at greater scales to be valid. In another example, very fine-grained mudstones may be extremely uniform in reservoir quality at a scale much smaller than a 1" core plug. The recognition of the appropriate scale of investigation is, therefore, critical to reservoir characterization efforts in carbonates.

#### 2.1.4 CLASSIFICATION

Carbonates have a special textural classification (Dunham, 1962) based on the presence or absence of lime mud and grain support. Textures range from grainstone, rudstone, and packstone (grain-supported) to wackestone and mudstone (mud-supported); where depositional texture is not recognizable, carbonates are classified as boundstone or crystalline. Other workers have classified carbonates based on fabric selective and non fabric selective pore types (Choquette and Pray, 1970). Porosity under this classification includes (1) fabric selective (interparticle, intraparticle, intercrystal, moldic, fenestral, shelter, and framework) and (2) non-fabric selective (vug and channel, cavern, and fracture).

#### 2.1.5 MINERALOGY

Carbonate mineralogy is usually simple – principal minerals are calcite, dolomite, and minor clay. Secondary minerals like anhydrite, chert, and quartz are common. Accessory minerals like phosphates, glauconite, ankerite, siderite, feldspars, clay minerals, pyrite, etc. are also present depending on the environment of deposition and diagenetic history. Disseminated pyrite present in minor quantities can affect the resistivity logs and result in apparently pessimistic estimation of oil saturation. Total gamma ray logs are insufficient to estimate clay volumes because of the presence of phosphate or organic matter, which result in relatively high uranium content. Diagnostic crystal structure of the different carbonate minerals is revealed by x-ray studies; these indicate that chemical tests for magnesium, a common basis for the classification of limestones and dolostones, are insufficient to prove the existence of the mineral dolomite. High magnesium calcite occurs in many carbonates, often indicating little diagenesis. Correct mineralogy is important for accurate estimation of porosity using nuclear devices. Elemental concentration spectroscopy logs provide valuable information to address this problem. Also, there may be a relationship between mineralogy and reservoir quality.

### 2.1.6 CORE SAMPLING

Core samples provide a valuable data source for investigating geological heterogeneity and understanding reservoir quality and performance. Many analytical techniques are employed to investigate heterogeneities at different scales such as slabbed core description, thin section petrography and mineralogy, core plug analyses, special core analyses, geomechanical analyses, and wireline log/seismic data calibration. As an initial effort, very small-scale heterogeneities are examined using thin sections and samples from slabbed cores, such as reservoir texture, fauna/flora, grain size, mineralogy, and diagenetic history. Conventional core plugs examine larger scale heterogeneity and reveal pore size distributions, reservoir quality (such as porosity and permeability), fluid saturation, and capillary pressure variations. Special core analyses of whole core samples examine a larger scale and provide data on relative permeability, saturations, capillary pressures, and restored state. On a local or semi-regional scale, geomechanical studies of core samples provide data concerning stress-strain relationships, rock properties, and fracture and fault trends. Finally, calibration of core-derived heterogeneities with wireline logs and seismic response provides data concerning field-wide reservoir quality and performance at the largest scale. Core recovery and quality are of serious concerns in carbonate reservoirs. Cores from fragile formations are lost or damaged leading to depth matching issues and unreliable measurements of reservoir properties. Mechanical aspects of coring like retrieval, surface handling, preservation, transportation, plugging, washing, and drying need special attention. Each coring technique (sponge coring, gel coring, pressure coring and horizontal coring) has certain advantages and disadvantages. The use of specific coring techniques depends on the objective and understanding of the formation characteristics. Optimization of drilling fluids and coring parameters are important factors for achieving efficiency, quality and best recovery. Factors that affect the reliability of the measured properties are preserved vs. restored samples, cleaning efficiency for the contamination caused by mud filtrate invasion, precipitation of wax, asphaltene & high-end components during retrieval by reduction in temperature and pressure, drying and oxidation during transportation and storage, burial history, hysteresis of adsorption/deadsorption, etc. Appropriate sampling (plugs) for physical measurements is very



important for representing the reservoir in highly heterogeneous cases. It is important to understand and define the different scales of heterogeneity so that SCAL and field-scale data can be compared. Overburden will change pore size, shape and aspect ratio. As pore structure controls relative permeability ( $K_r$ ), pore structure and connectivity must not be altered for  $K_r$  measurements representing in-situ conditions. Irreversible changes occur to rock properties, as stresses are relieved during coring and retrieval. Representative pore pressure and overburden must be used in lab experiments. Core porosity, cementation factor ( $m$ ) should be measured under appropriate overburden conditions covering all RRT. Overburden corrections to the core porosity should be based on the RRT, as different rock types may respond differently to confining pressure. Carbonates have mixed wettability. Saturation exponent ( $n$ ) which is an important parameter in log analysis depends on the wettability. Alteration of wettability in the process of coring, transportation, and storage is another issue which needs careful consideration. Different RRT, mixed wettability, faults, anisotropy and heterogeneity in the reservoir result in different  $K_r$  and capillary pressure ( $P_c$ ). Porous plate/centrifuge data are preferred over mercury injection data for  $S_{wi}$  in rocks that are texturally complex (i.e. where different pore types coexist). The directional measurement of the properties on core plugs is important. The orientation of flow in a core sample (plug or whole core) must be identical to the alignment of sedimentary stratification in the formation.

### 2.1.7 FLUID CONTACTS

The interpretation of fluid contacts involves many factors. Improper evaluation and understanding result in the misinterpretation of the fluid contacts. Long transition zones may result from low and varying permeability near the oil-water- contact. The reaction of sulfate-reducing bacteria near the oil-water interface may result in the precipitation of calcite, forming a seal between oil and water parts of the reservoir and limiting aquifer support to the oil reservoir. In some cases, this is the reason for the presence of  $H_2S$  near the oil-water-contact. Another complication is highly vuggy carbonates with closed vugs, which may contain oil in the vugs even below the oil-water-contact, as the oil may have been trapped during migration and flow

channels may have been sealed due to post-migration diagenesis. Also, oil and water may coexist in the matrix and fractures in fractured carbonate reservoirs.

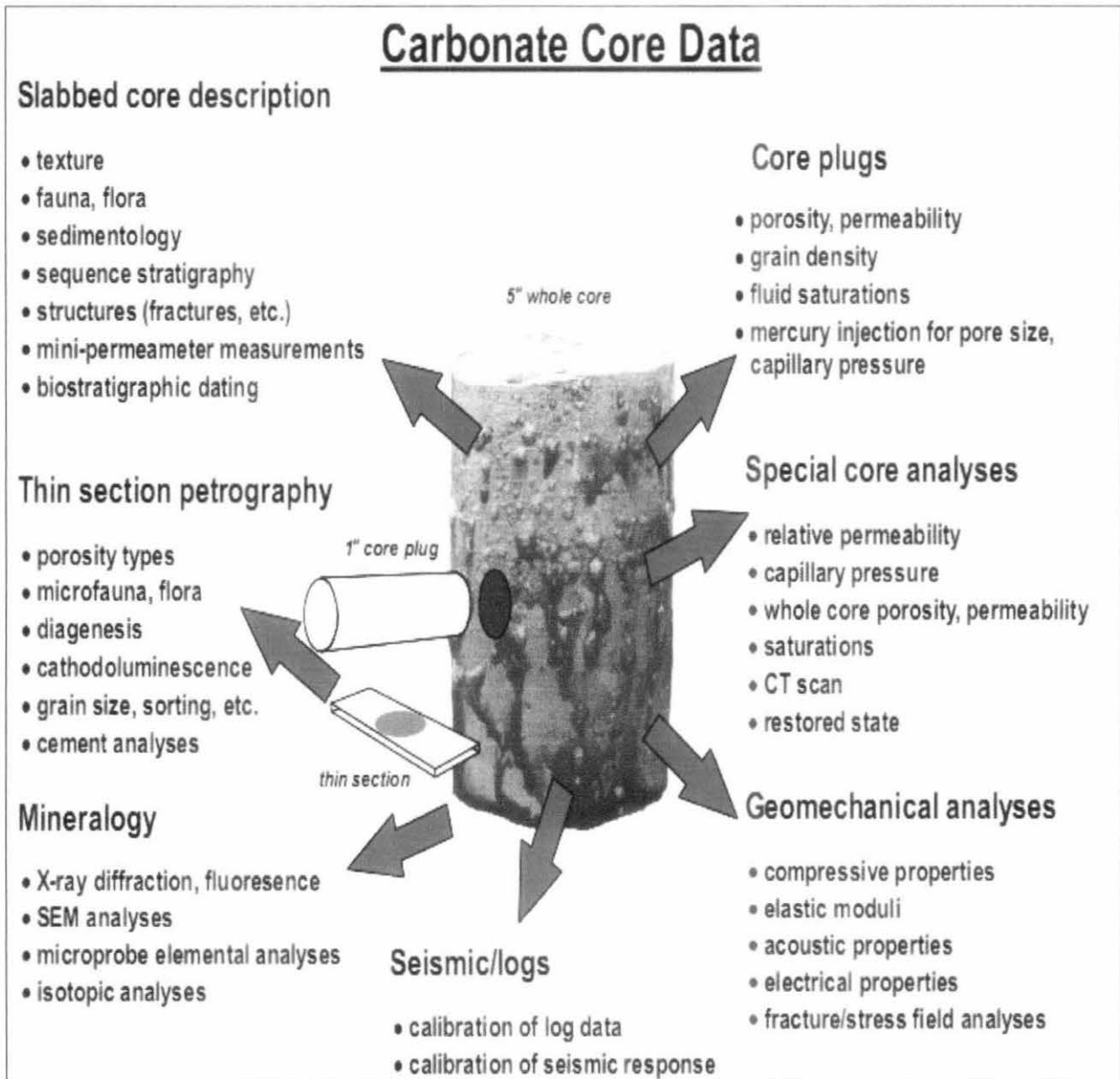


Figure 1: Carbonate Core Data

### 2.1.8 GEOMECHANICS / ANISOTROPY

Dynamic geomechanical models are of significant value for carbonates due to the elastic nature and susceptibility of carbonates to in-situ stress changes. Production of fluids results in pore pressure changes and may contribute to alteration of reservoir rock properties. Permeability is highly dependent on the in-situ state of stress. Secondary permeability generated by small fractures can have an impact on the effectiveness of fluid injection and production. Mobility of fluids is much higher in the direction of maximum principal stress than in the direction of least principal stress. Natural fractures trending in the direction of maximum horizontal principal stress are more permeable as they often remain open, whereas fractures perpendicular to the maximum horizontal principal stress are generally sealed. The challenge is the evaluation of the stress state (both magnitude and orientation) and the rock strength. This can be achieved by estimating the magnitude of minimum principal stress and other geomechanical parameters, like Young's modulus, Poisson's Ratio, bulk modulus, shear modulus, etc. The main inputs to this processing are acquired through Dipole Shear Imaging (DSI). Leak-off or extensometer tests can be made on some wells to calibrate or verify the log results. Orthogonal calipers and other data from FMS and FMI for example, are also used to determine the direction of in-situ stresses. Removing the wellbore cylinder alters the ambient stress field and considerable strain is imparted upon surrounding rock after drilling a hole. After such alteration, the principal component directions could be rotated, in particular if they were not aligned vertically with the maximum component in the beginning. Tectonic stresses could easily rotate the principal components out of a simple frame of reference. In the classically used relationship, elastic strain is neglected (assumed zero) which can not represent the case of fractured media when the fractures are open by post burial process, either by stress or pore pressure. The issue is to distinguish between elastic deformation and permanent non-elastic modification. By definition, elastic strain is reversible and recovered when the applied stress is released. However, tectonic processes produce large-scale non-elastic deformation, some of which might be described as the cumulative result of long-term elastic strain components. The amount of true in-situ elastic strain in fractured media is difficult to gauge. Geomechanical model of the

## 2.2 CHALLENGES RELATED TO CARBONATE RESERVOIR

The depositional processes of carbonate formations are quite different from clastics. Calcium carbonate is much more chemically active than the silica that constitutes sandstones. It is easily dissolved in water, even more so in acidic water. Mechanical properties are another significant difference. Carbonate rocks tend to be more prone to fractures than sandstones. For all these reasons carbonates form different rock types with a heterogeneous distribution throughout the reservoir. Moreover, the poor correlation between porosity and permeability, and the presence of vugs, fractures, and other large scale heterogeneities such as stylolites create very complex paths for fluids making it difficult to accurately model the distribution of permeability in carbonate reservoirs. Finally, unlike sandstone reservoirs that are typically strongly water-wet most carbonate reservoir rocks are known to be mixed-wet or oil-wet to some degree. All of the above make carbonate reservoirs more challenging in terms of reserves evaluation, reservoir modeling and simulation, and also for maximizing oil recovery.

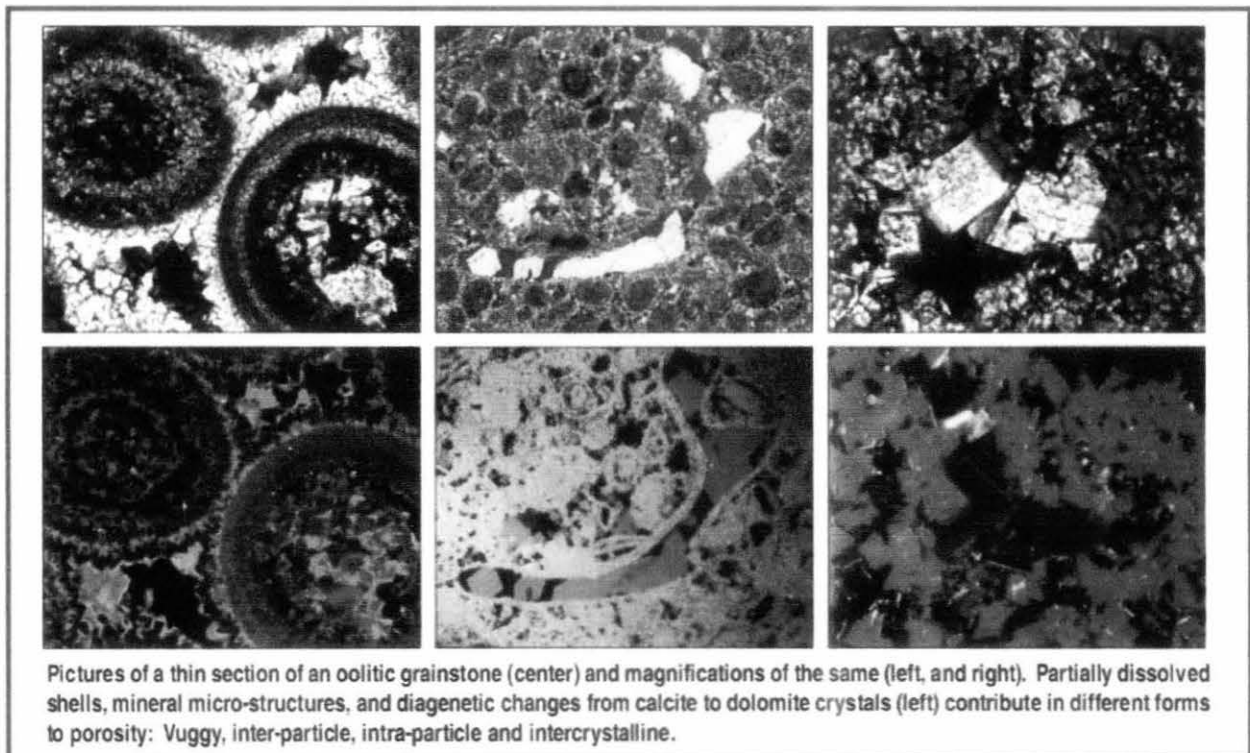


Figure 2: Pictures of thin section result

### **2.3 THERMAL CONDUCTIVITY**

The basic concept of heat flow defines this property as a temperature difference between two locations resulting in a heat flow  $q$ . The magnitude of  $q$  depends on the thermal conductivity of the material and the distance between the two locations mentioned earlier. Heat flow studies provide information on the occurrence and nature of geothermal resources, oil source rock maturation, secondary migration of petroleum and subsurface structures. Heat flow studies in geological systems are paramount interest to the oil and gas industry. The studies give an indication on the thermal maturity of the hydrocarbon reserves and also provide a better enhanced understanding of the reservoir.

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 ( $x_2 - x_1$ ).

$$q_i = K \cdot \frac{\partial T}{\partial x}$$

Otherwise, the theory of a thermal conductivity is also defined by the classical Fourier's Law of heat conduction.

$$q_i = \lambda \frac{\partial T}{\partial x_j}$$

Thermal conductivity is an intrinsic material property for which the values depend on the chemical composition, porosity, density, structure, and fabric of the material. In marine geophysics, mainly thermal conductivity profiles of sediment and rock sections are used, along with temperature measurements, to determine heat flow. Heat flow is not only characteristic of the material, but an indicator of type and age ocean crust and fluid circulation processes at shallow and great depths.

## CHAPTER 3: METHODOLOGY

### 3.1 RESEARH METHODOLOGY

In order to achieve the objectives of this project, some researches had been done on some resources from journals and technical papers. Firstly, obtain all the information on the kinetics dissolution, calcium carbonate dissolution, and thermal conductivity of carbonates rock. Then, select and get the sample of the carbonates rock that significant with the project. Afterward, experiment will be conduct that included of coring of the sample, thin section, thermal conductivity test, scan the sample under Field Emission Scanning Electron Microscope (FESEM), X-Ray Diffraction (XRD), X-Ray Fluorescence (XRF) and experiment to measure the rate of the samples dissolve in the selected acid. Lastly, gather all data required in this project for analysis and discussions.



Figure 3: Methodology of the project



Sample of limestone (Sg. Siput)



Sample of marble (Simpang Pulai)

Figure 4: Samples of limestone and marble

**3.2 PROJECT ACTIVITIES**

**I) CORE SAMPLING**

TOOLS / EQUIPMENTS REQUIRED

Tools / Equipments	Description
<p><b>Rock Cutting and Trimming machine</b></p>	<p>Cutter tools are required for sample procurements where all the rock samples will be cut into block with the same sizes using rock cutting machine. Crusher tools are used to crush sample into powder for XRD &amp; XRF analysis.</p>
<p><b>Coring Machine</b>  <b>Brand: NALIK</b>  <b>Model: ZIZ-CF-130/ NL1006</b></p>	<p>Machine that will be used to make the samples with 1.5” diameter core.</p>

Table 1: Equipments required for core sampling

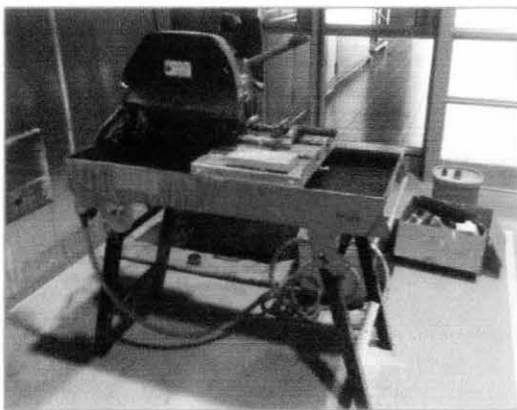


Figure 5: Rock Cutting Machine



Figure 6: Trimming Machine

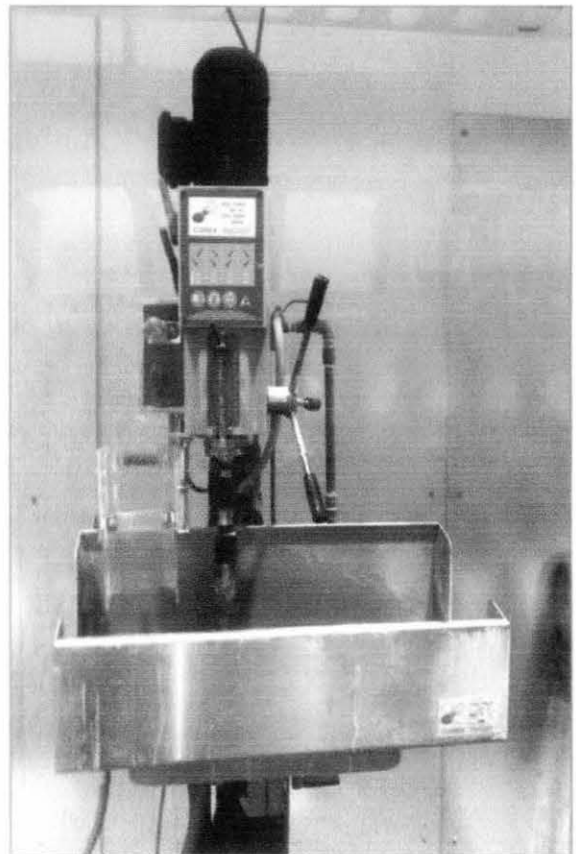


Figure 7: Coring Machine

Procedure for core sampling:

1. Mark the rock sample with marker pen after measured the length of the sample needed.
2. Cut the sample with rock cutting machine.
3. Make the core sample with 1.5" diameter by using coring machine. (keep the fragments of the rock after finish the coring)
4. Cut the sample with trimming machine to make all the samples with same height. (keep the fragments of the rock after trimming)



Figure 8: Core sampling process supervised by technician



II) THIN SECTION

TOOLS / EQUIPMENTS REQUIRED

Tools / Equipments	Description
<b>Geological Cutter/ Lapping and Polishing Machine/ Precision Cutter</b>	These three equipments are required in order to do the thin section.
<b>Polarizing Microscope</b>	Analyzed microscopically in order to analyze the characteristics of the rock samples.

Table 2: Equipments required for thin section



Figure 9: Geological Cutter



Figure 10: Lapping and Polishing Machine



Figure 11: Precision Cutter

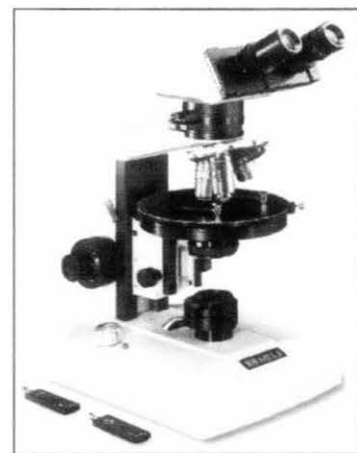


Figure 12: Polarizing Microscope

Procedure for thin section:

1. Cut sample in slice by geological cutter (2-4mm).
2. Polish 1 side of sample & let it dry.
3. Polish 1 side of sample on a glass slide by using polishing machine.
4. Use hot plate to heat up the epoxy on the glass slide to stick the slide sample on the glass (make sure no bubble seen on the back of glass).
5. The sample on glass need to be cut using precision cutter until 0.1mm sample obtained (frequently measure the sample thickness by using micrometer).
6. Polish the sample using polishing machine until the desired thickness obtained.
7. Samples are ready to be analyzing below the microscope.

Note: Refer Appendix B for equipment/machine for thin section.

### III) THERMAL CONDUCTIVITY

#### TOOLS / EQUIPMENTS REQUIRED

Tools / Equipments	Description
<b>Rock Cutting</b>	Cutter tools are required for sample procurements where all the rock samples will be cut into block with the same sizes using rock cutting machine.
<b>Thermal Conductivity Tool</b>	To estimate thermal conductivity values for rock samples. Then measurements will be taken between two locations with several different directions.

Table 3: Equipments required for thermal conductivity

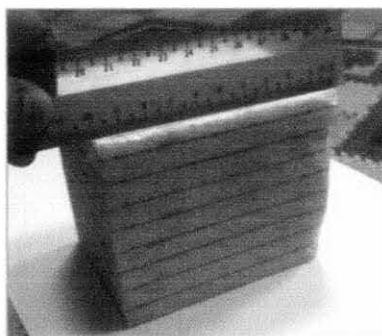


Figure 13: Sample of the rock for thermal conductivity test

Experimental set-up for the linear conductive heat transfer system are:

1. Making lines for each face (6 surfaces) on the block for 10cm
2. Calibrate temperature for the two needle probe so that both T1 and T2 are equally before measurement
3. 40 watt of heat being applied.
4. Time consuming was 15 seconds.
5. Put the needle probe on the edge of each line and T1 and T2 were taken.
6. Heat transfer being calculated.

#### IV) FIELD EMISSION SCANNING ELECTRON MICROSCOPE (FESEM)

##### TOOLS / EQUIPMENTS REQUIRED

Tools / Equipments	Description
<b>Field Emission Scanning Electron Microscope</b>	FESEM tool can produce very high resolution images of sample surface. It will be use to visualize the sample's surface in a high magnification and map the elements content on the specific area.

Table 4: Equipment required for FESEM

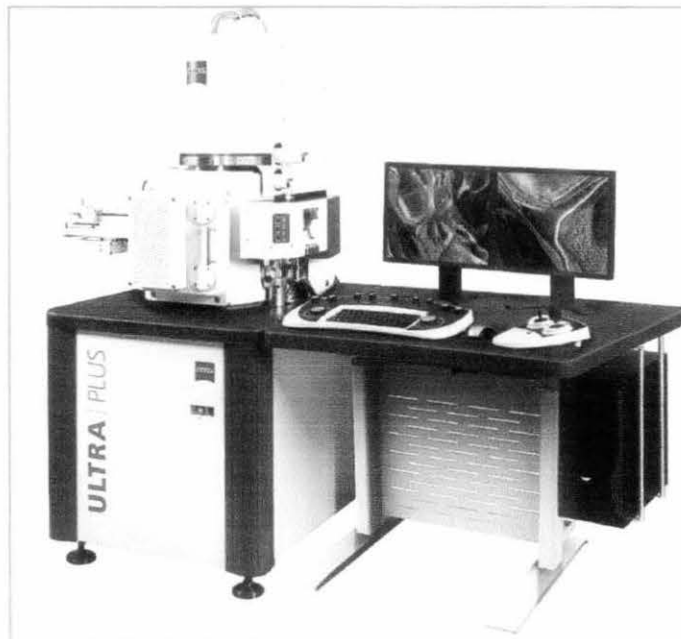


Figure 14: Field Emission Scanning Electron Microscope

The procedures of this experiment are designed as following steps:

1. Both samples in pieces form were coated with gold.
2. Samples are put in the chamber inside of SEM for 5 minutes to let it in vacuum condition.
3. As gas molecules interfere with the electron beam and with the emitted secondary and backscattered electrons used for imaging.
4. Samples were computerized in order for images capturing and for elemental mapping.

**V) X-RAY FLUORESCENCE (XRF)**

TOOLS / EQUIPMENTS REQUIRED

Tools / Equipments	Description
<b>X-Ray Fluorescence (XRF)</b>	XRF tool will be used to determine and analyze the chemical elements qualitatively and quantitatively by measuring their Characteristic radiation. Sample type: Liquid and Solid

Table 5: Equipment required for XRF



Figure 15: XRF Machine

The procedures of this experiment are designed as following steps:

1. To turn on the power to the unit, depress the POWER ON switch on the control panel; the unit will only function when the TIME SWITCH is rotated away from zero.
2. The filament of the x-ray tube should be illuminated. Wait 5 minutes, and then depress the X-RAYS ON switch. The x-ray machine needs to run with X-RAYS ON for about an hour to heat up enough to begin emitting photons.
3. Once count levels are high (100's per second) you should be able to obtain diffraction data.
4. Data acquisition is automated through a Lab View Program on the computer which controls the stepper motor.

## VI) X-RAY DIFFRACTION (XRD)

### TOOLS / EQUIPMENTS REQUIRED

Tools / Equipments	Description
<b>X-Ray Diffraction (XRD)</b> <b>Brand: Bruker</b> <b>Model: D8 Advance</b>	XRD tool will be used to characterize the structure, crystallite size (grain size) and grain orientation. Sample type: Powder type 2 theta: 3° to 60°, Scanspeed: 1°/sec

Table 6: Equipment required for XRD

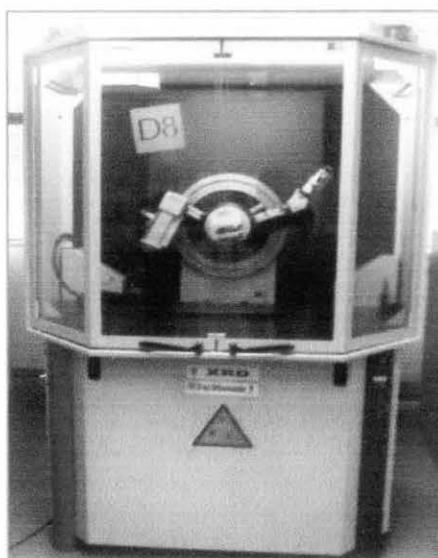


Figure 16: XRD Machine

The procedures of this experiment are designed as following steps:

1. Sample positioning and focusing can be performed easily using the standoff pointer provided with all systems and through the collimator laser pointer which allows the user to quickly locate measurement locations. This is particularly helpful when using the Automated Stress Mapping option.
2. The 4-Point bending fixture and Proto strain bridge are used for quick and easy determination of the effective x-ray elastic constant for new materials as per ASTM 1426-91, "Standard Test Method for Determining the Effective Elastic Parameter for X-ray Diffraction Measurements of Residual Stress".
3. The Proto Portable Electro Polisher is specifically for x-ray diffraction work, making material removal efficiently.
4. Continuous Research and Development from software available to match the plot obtained from XRD result.

## **VII) RATE OF DISSOLUTIONS**

The procedures of this experiment are designed as following steps:

1. The sample of rocks will be cut into a core samples about 3cm thick and diameter of 1.5".
2. Then, the core will put into the beaker that consists of two different acids, which are hydrochloric acid, HCl and formic acid, CH<sub>2</sub>O<sub>2</sub>.
3. Both pH and specific conductance measurements were recorded at specific time intervals.
4. Data will recorded every hour in the early stages of dissolution run and every few as the experiment progressed. Total run times will be about five days.
5. All runs will made in duplicate and repeat all steps above with another sample.

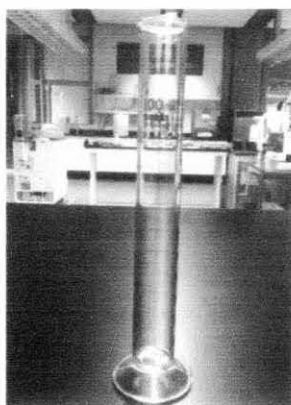
TOOLS / EQUIPMENTS REQUIRED

<b>Tools / Equipments/ Chemicals</b>	<b>Description</b>
<b>Beaker</b>	Simple container for stirring, mixing and heating liquids commonly used in laboratories
<b>Volumetric Flask</b>	For preparing liquids with volumes of high precision. It is a flask with an approximately pear-shaped body and a long neck with a circumferential fill line.
<b>Volumetric Cylinder</b>	Laboratory equipment used to accurately measure the volume of a liquid.
<b>pH Meter</b>	An electronic instrument measuring pH (acidity or alkalinity) of a liquid (though special probes). A typical pH meter consists of a special measuring probe (a glass electrode) connected to an electronic meter that measures and displays the pH reading.
<b>Digital Balance</b>	A measuring instrument for determining the weight or mass of an object.
<b>Hydrochloric Acid, HCl</b>	HCl (37%) M= 36.45 g/mol 1L= 1.19kg
<b>Formic Acid, CH<sub>2</sub>O<sub>2</sub></b>	CH <sub>2</sub> O <sub>2</sub> (100%) M= 46.03 g/mol 1L= 1.22kg
<b>Distilled water</b>	Water that has many of its impurities removed through distillation. Distillation involves boiling the water and then condensing the steam into a clean container.

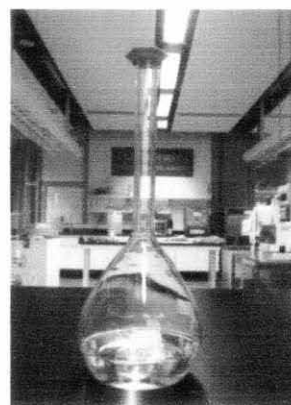
Table 7: Tools / Equipments/ Chemicals required for acid dissolution experiment



Beaker



Volumetric Cylinder



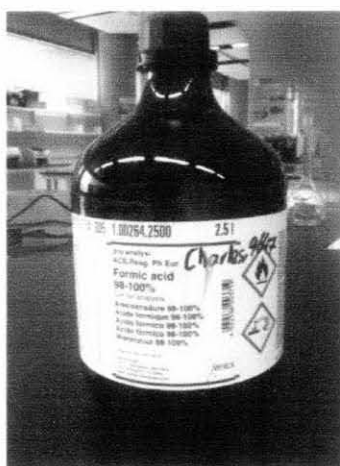
Volumetric Flask



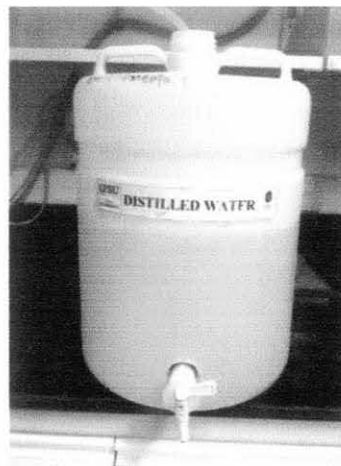
pH Meter



Digital Balance



Chemical



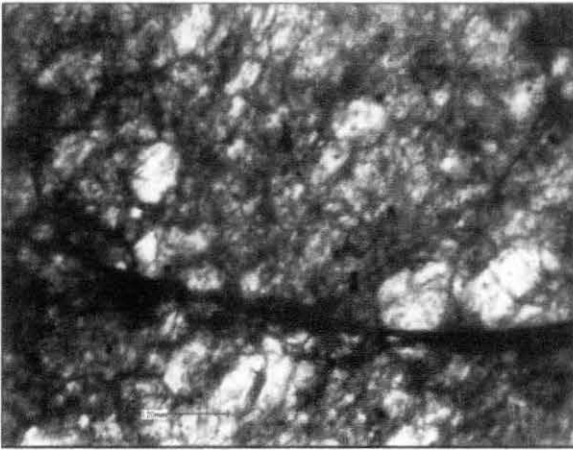
Distilled Water

Figure 17: Tools / Equipments/ Chemicals required for acid dissolution experiment

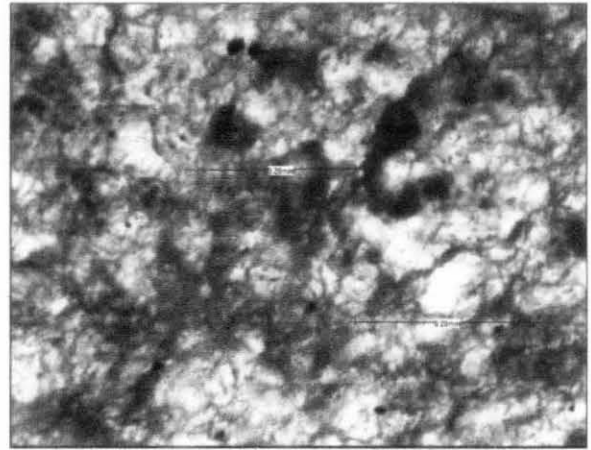


## CHAPTER 4: RESULTS AND DISCUSSION

### 4.1 MICROSCOPIC IMAGE FROM THIN SECTION



S #1(limestone)



S #3 (marble)

Figure 18: Microscopic Image from the thin section

Based on figure 6 and 7 (thin section images for limestone and marble), there are variety of colors have been found in this sample, which are black, grey and white. In general observation and analysis, the variety of colors shows the presence of some elements such as calcium and iron oxide. The particles size of limestone is around 0.05mm to 0.1mm, while the particles size for marble is around 0.1mm.

But, the analyses of this microscopic image from the limestone sample just a general view of the minerals contents in this sample. There are several test has been done such as XRF, XRD and FESEM to get the result and to confirm what the minerals are in the sample.

**4.2 X-RAY FLUORESCENCE (XRF)**

**Sample #1 (limestone)**

Element	O (-ve)	Al	Si	K	Ca	Fe	Sr	Na2O	P2O5
KCps	1000.00	3.70	12.20	6.80	377.50	15.40	10.10	0.00	1.00
%	34.00	4.05	7.49	0.78	52.76	1.02	0.09	5.73	0.00

Table 9: XRF result for S#1

**Sample #2 (limestone)**

Element	O (-ve)	Al	Si	K	Ca	Fe	Ne	SiO2
KCps	1000.00	3.60	11.40	6.20	345.40	14.50	0.00	11.40
%	34.00	4.26	7.65	0.78	52.29	1.04	6.21	16.40

Table 10: XRF result for S#2

**Sample #3 (marble)**

Element	O (-ve)	Si	Ca	N	Na2O
KCps	1000.00	1.90	461.90	-1000.00	0.00
%	29.000	1.200	69.600	6.569	0.000

Table 11: XRF result for S#3

**Sample #4 (marble)**

Element	O (-ve)	Ca	CO2
KCps	1000.00	453.90	-1000.00
%	29.000	71.470	6.911

Table 12: XRF result for S#4

XRF was run in order to re-analyze the element content inside the sample. For sample #1 and sample #2 (samples of limestone), main content are Calcium (Ca) with 52.76% and 52.29%, Silica (Si) with 7.49% and 7.65%, Aluminium (Al) with 4.05% and 4.26%, Iron (Fe) with 1.02% and 1.04% respectively. While the main content in sample #3 and sample #4 (samples of marble) is Calcium (Ca) with 69.6% and 71.47% respectively.

The different of compositions and amount of minerals content will effects the rate of acid dissolution process.

## 4.3 X-RAY DIFFRACTION (XRD)

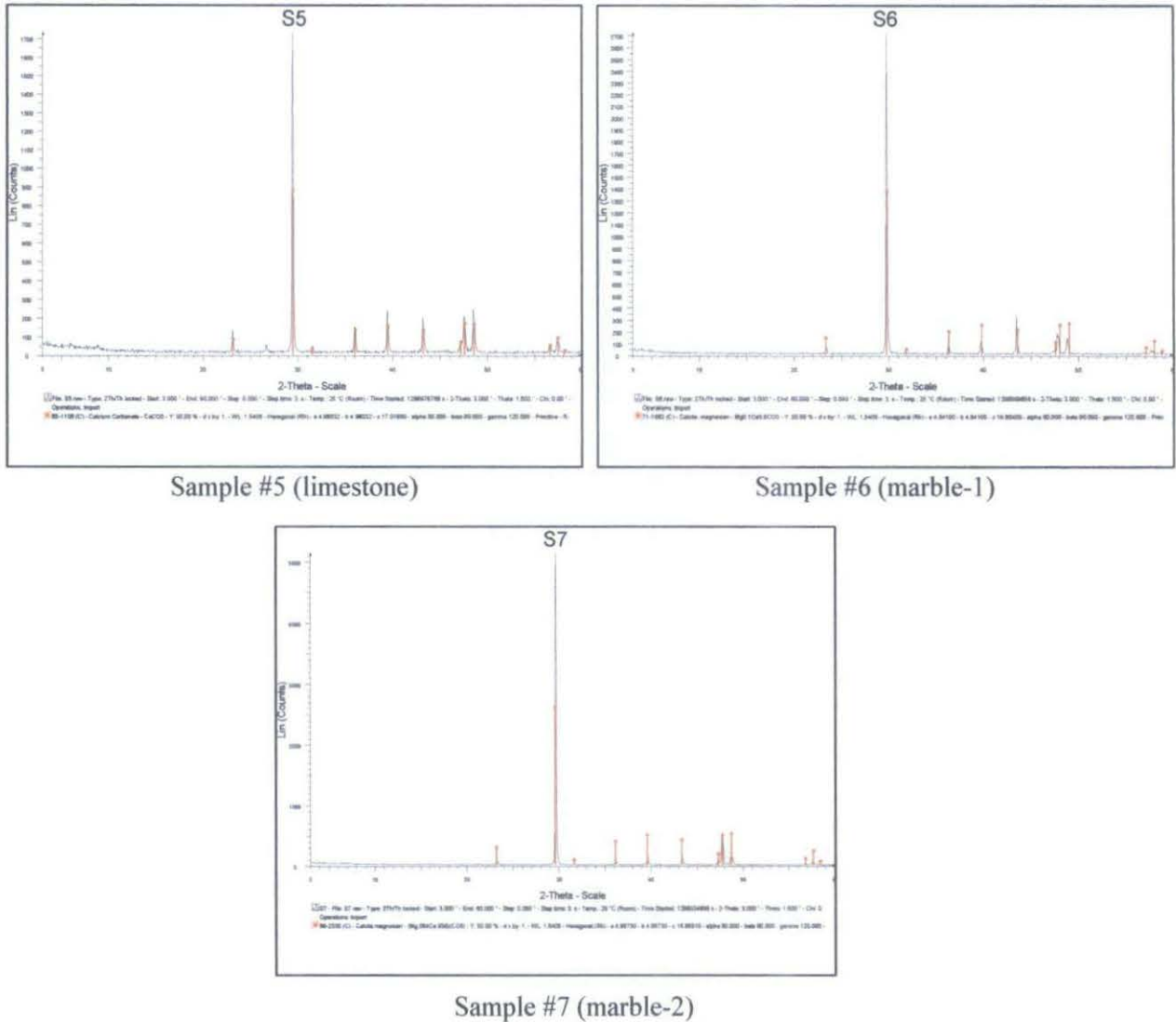


Figure 19: XRD Results with analysis

Based on XRD result, shows that sample #5 (limestone) main contain is Calcium carbonates, CaCO<sub>3</sub>. While, the main content of sample #6 and sample #7 (marble) is calcite and magnesium. The variations of mineralogy will effects the acid dissolution rates and thermal conductivity of the sample.

#### 4.4 FIELD EMISSION SCANNING ELECTRON MICROSCOPE (FESEM)

##### i. Sample#1 (S1-Limestone)

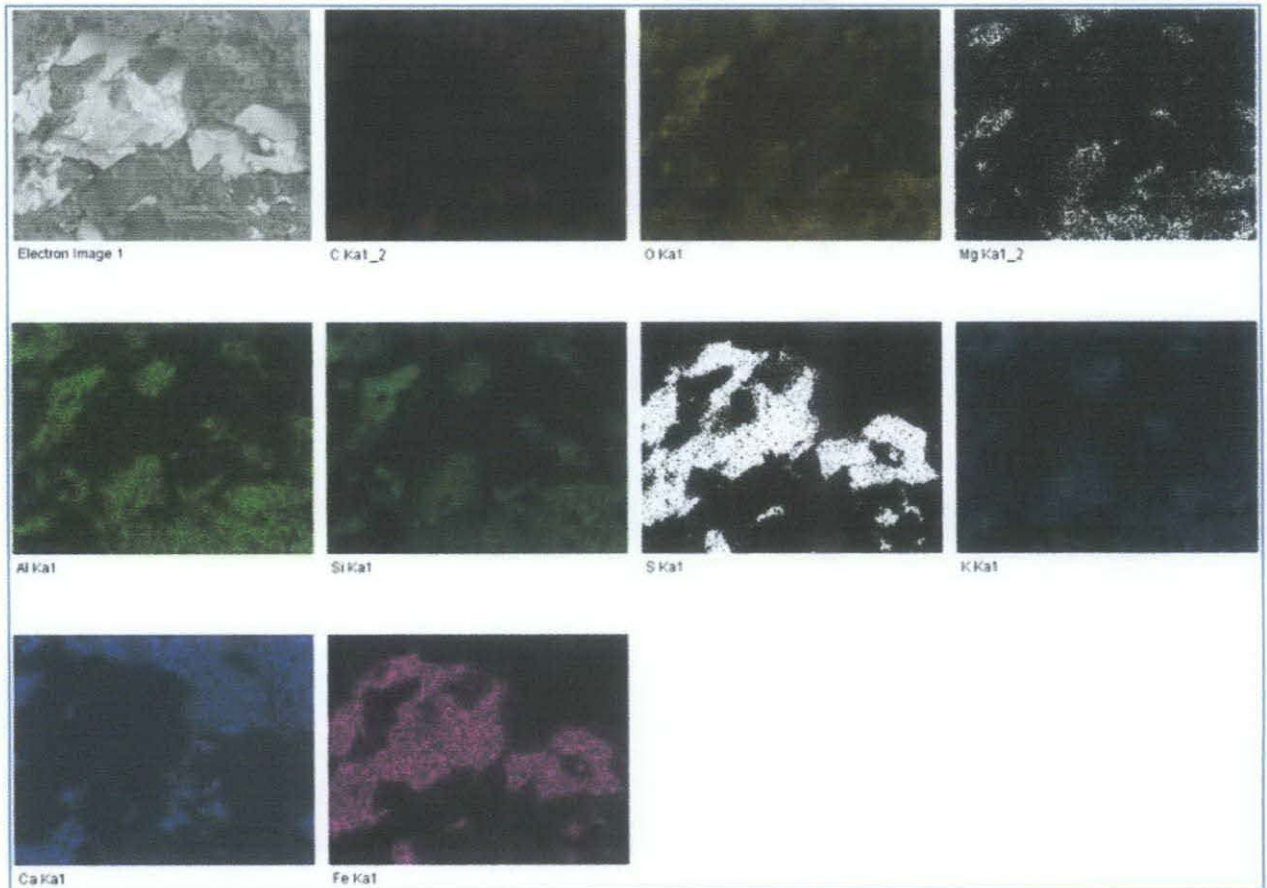


Figure 20: Distribution of element images for sample #1 (S1-limestone)

From the result above, it shows that Calcium, Ca is not homogenously distributed. These elements distribution will be uneven across the matrix that can effects the dissolution rate.

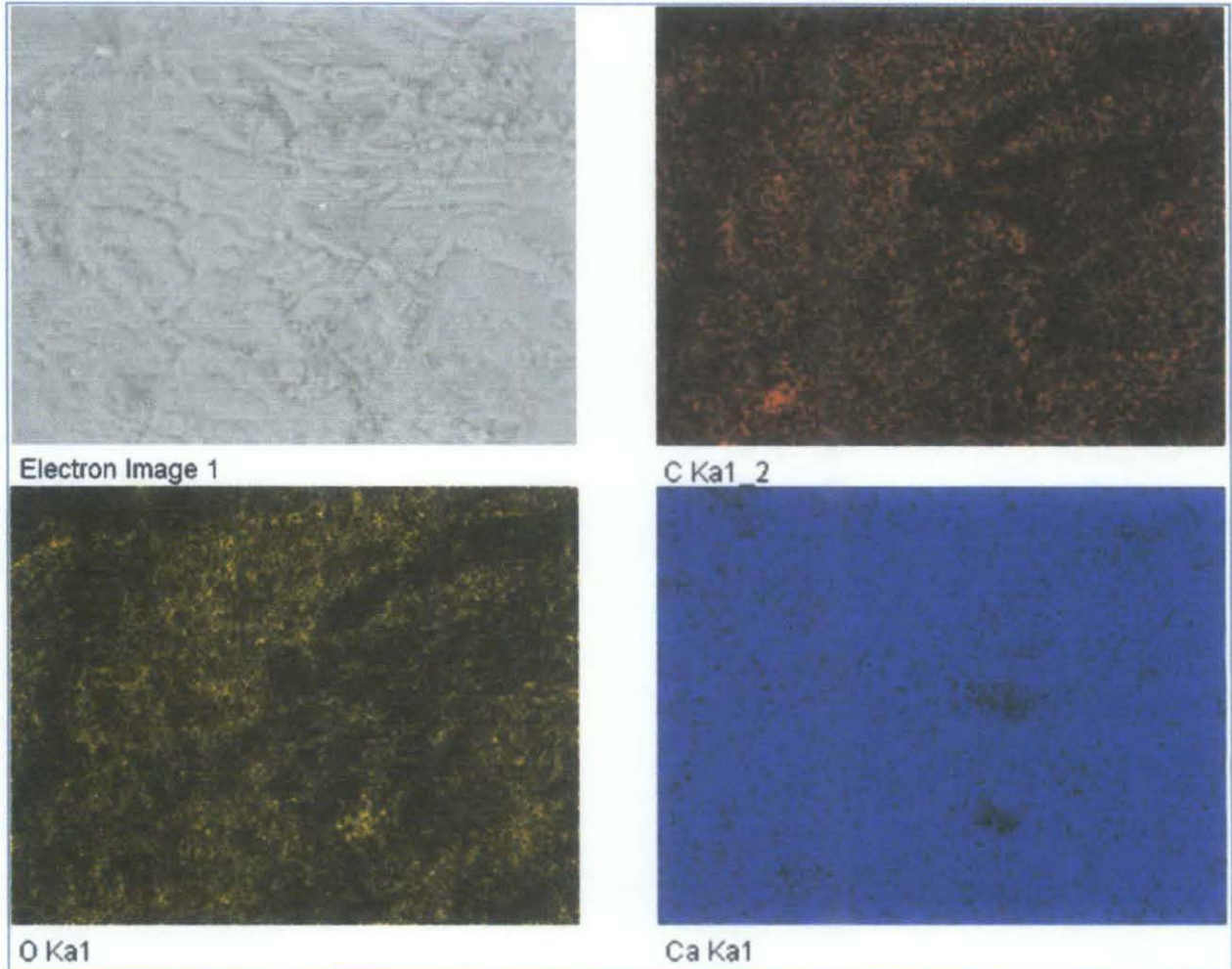
**ii. Sample#2 (S2-Marble)**

Figure 21: Distribution of element images for sample #2 (S2- marble)

Based on the result of distribution element above, it shows that the distribution of the elements in this sample (marble) is homogenous. The dissolution rate will not affect much because the element in this sample is distributed well.

#### 4.5 THERMAL CONDUCTIVITY (TC)

SAMPLE	PHASE	Average k
#1 (limestone)	a	0.2830
	b	0.2234
	c	0.0494
	d	0.0913
#2 (marble)	a	0.0165
	b	0.1331
	c	0.0231
	d	0.1963
#3 (marble)	a	0.0226
	b	0.1639
	c	0.0227
	d	0.1380

Table 13: Thermal conductivity result

Both samples of the rock might have different grain distribution and mineral composition due to different facies of deposition and depending on their rocks formation process. Due to these rock properties, different sets of result obtained for each of every different locations and direction (phase) of heat flow measurement which will use to estimate the thermal conductivity of the samples.

Based on result from table 8, four different phases (a, b, c and d) of each sample have been estimated, and the result varies in values ranging. Based on that, limestone has higher value of thermal conductivity than marble, as each of elements in this sample is able to conduct and transfer heat.

4.6 ACID DISSOLUTION

I. Hydrochloric acid, HCL (37%), M=36.5g/mol, 1L=1.19kg, pH=2.0, Temp (degC) = 23.6

Limestone:

Time (days)	1	2	3	4	5	6	7	8	9	10
Weight (g)	67.55	67.38	67.19	67.01	66.82	66.64	66.45	66.27	66.08	65.90
Volume (mL)	25.50	25.30	25.15	24.97	24.79	24.62	24.44	24.27	24.09	23.92

Time (days)	11	12	13	14	15	16	17	18	19	20
Weight (g)	65.72	65.53	65.35	65.16	64.98	64.79	64.61	64.42	64.24	64.06
Volume (mL)	23.74	23.57	23.39	23.22	23.04	22.87	22.69	22.52	22.34	22.17

Table 14: Acid dissolution result for limestone in HCl

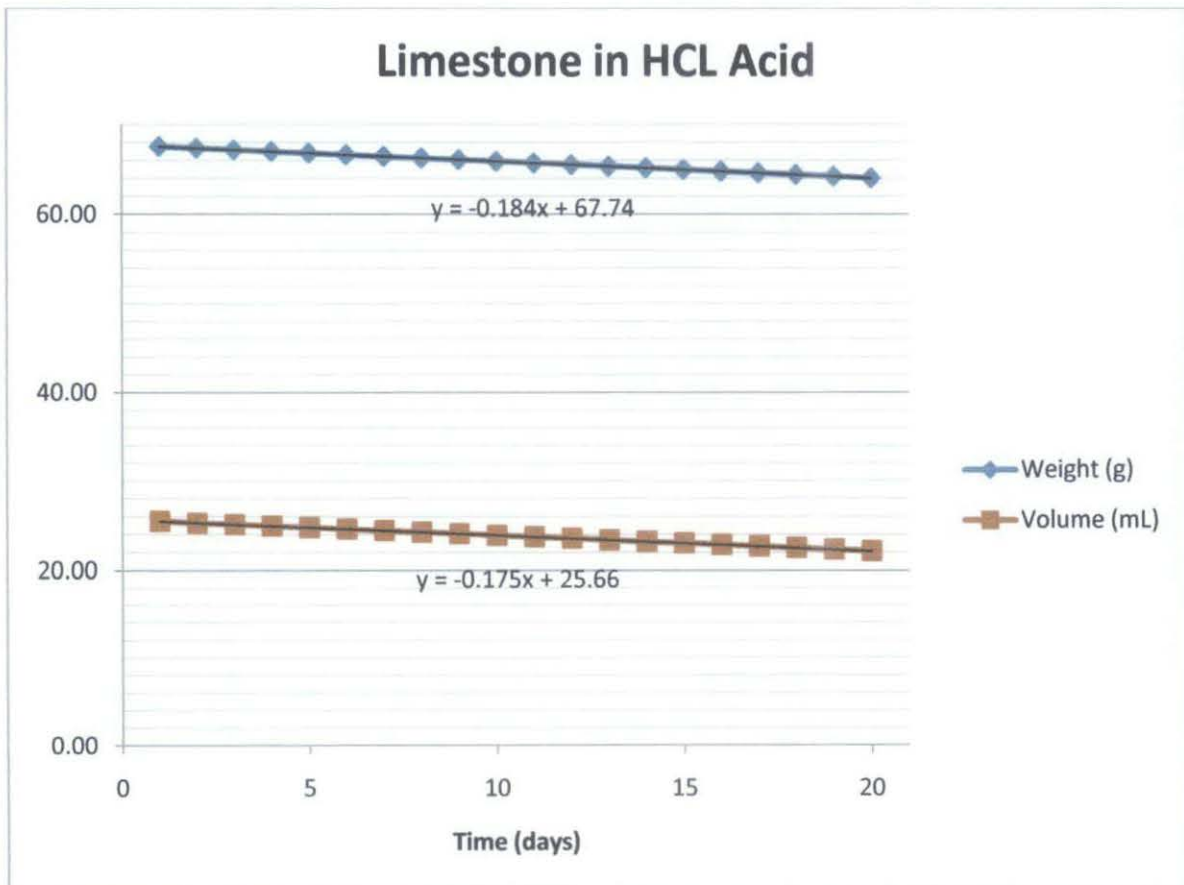


Figure 22: Dissolution of limestone in HCL Acid

Marble:

Time (days)	1	2	3	4	5	6	7	8	9	10
Weight (g)	81.31	81.14	80.95	80.78	80.59	80.41	80.23	80.05	79.87	79.69
Volume (mL)	29.80	29.65	29.57	29.44	29.33	29.21	29.10	28.98	28.87	28.75

Time (days)	11	12	13	14	15	16	17	18	19	20
Weight (g)	79.51	79.33	79.15	78.97	78.79	78.61	78.43	78.25	78.06	77.88
Volume (mL)	28.64	28.52	28.41	28.29	28.18	28.06	27.95	27.83	27.72	27.60

Table 15: Acid dissolution result for marble in HCl

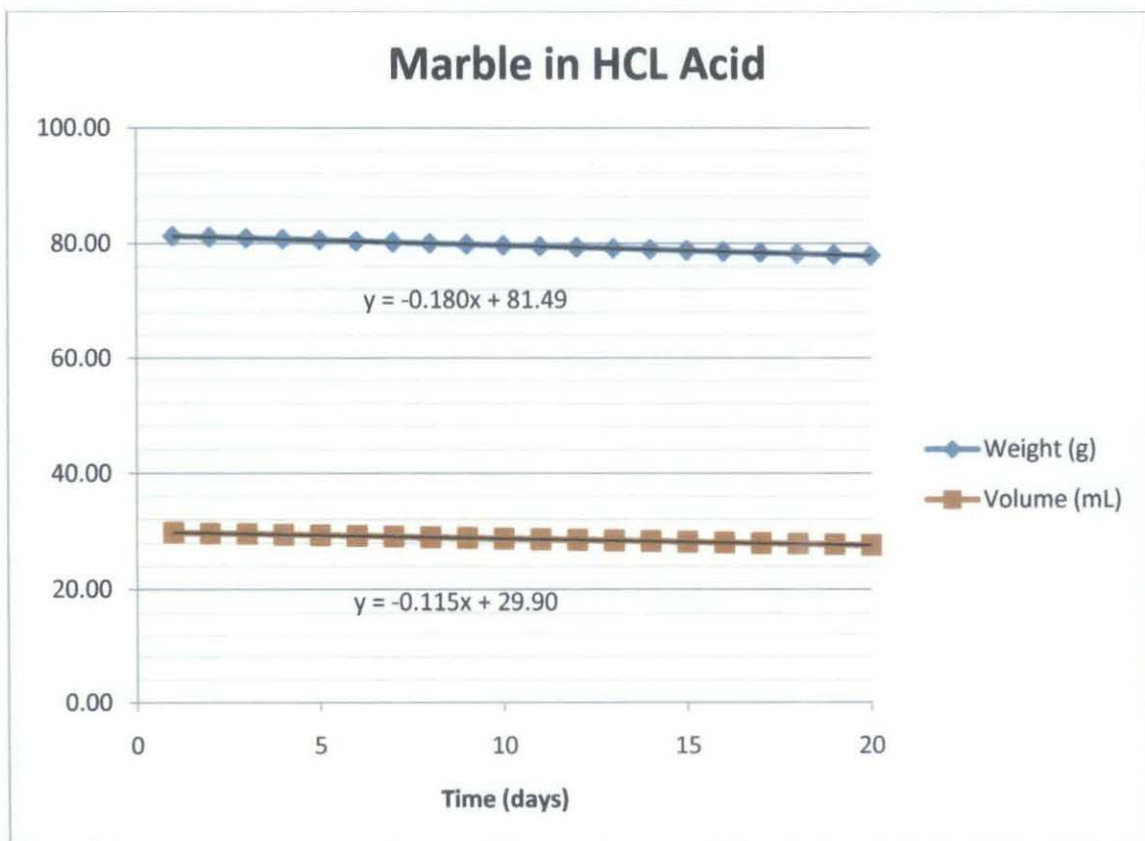


Figure 23: Dissolution of marble in HCL Acid



**II. Formic Acid, CH<sub>2</sub>O<sub>2</sub> (100%), M=46.03g/mol, 1L=1.22kg, pH = 2.58, Temp (degC) = 23.60**

Limestone:

Time (days)	1	2	3	4	5	6	7	8	9	10
Weight (g)	68.22	68.07	67.61	67.43	67.31	66.99	66.75	66.50	66.26	66.02
Volume (mL)	22.70	22.50	22.38	22.21	22.05	21.89	21.73	21.57	21.41	21.25

Time (days)	11	12	13	14	15	16	17	18	19	20
Weight (g)	65.77	65.53	65.28	65.04	64.79	64.55	64.30	64.06	63.81	63.57
Volume (mL)	21.09	20.93	20.77	20.61	20.45	20.29	20.13	19.97	19.81	19.65

Table 16: Acid dissolution result for limestone in Formic Acid

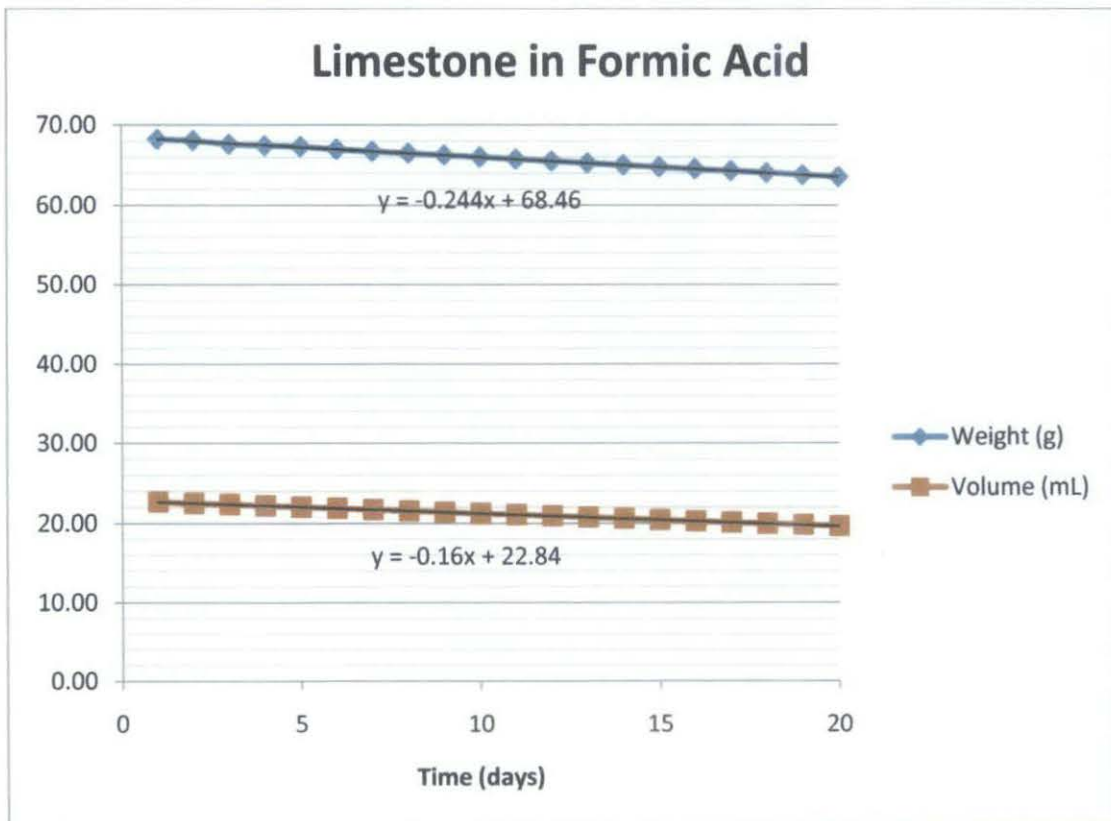


Figure 24: Dissolution of limestone in Formic Acid

Marble:

Time (days)	1	2	3	4	5	6	7	8	9	10
Weight (g)	81.69	81.54	81.17	81.00	80.88	80.61	80.39	80.17	79.96	79.74
Volume (mL)	29.50	29.10	28.95	28.63	28.36	28.08	27.81	27.53	27.26	26.98

Time (days)	11	12	13	14	15	16	17	18	19	20
Weight (g)	79.52	79.31	79.09	78.87	78.66	78.44	78.23	78.01	77.79	77.58
Volume (mL)	26.71	26.43	26.16	25.88	25.61	25.33	25.06	24.78	24.51	24.23

Table 17: Acid dissolution result for marble in Formic Acid

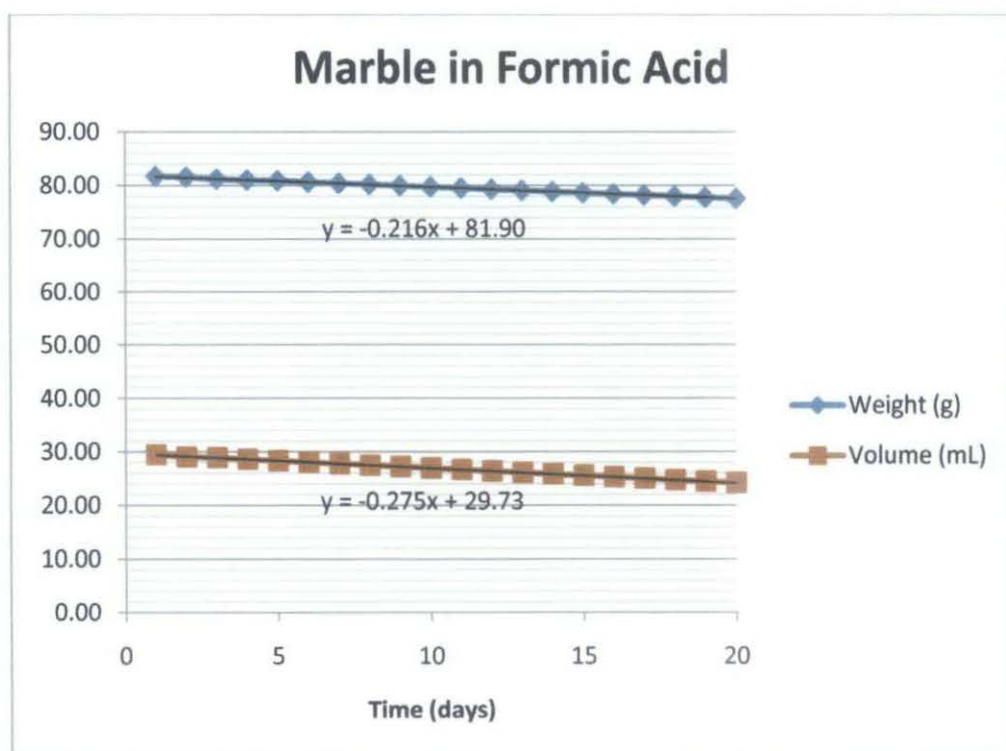


Figure 25: Dissolution of marble in Formic Acid

Based on the results and graph above, it shows that formic acid,  $\text{CH}_2\text{O}_2$  reacts faster than hydrochloric acid,  $\text{HCl}$  in carbonates, limestone and marble. It is proved that acidity of formic acid is higher compared to hydrochloric acid.

**RESULTS WITHOUT  
COMPRESSIBILITY CORRECTION  
(AFTER ACID DISSOLUTION)**

Total intruded volume (mm <sup>3</sup> /g):	3.76 at pressure of (Mpa):	200.6242	
Intrud. Vol (mm <sup>3</sup> ):	0.00		
Envelope density (g/cm <sup>3</sup> ):	2.5360		
Bulk density @ pressure (g/cm <sup>3</sup> ):	2.5360	at pressure of (MPa):	0.174
Apparent density (g/cm <sup>3</sup> ):	2.5604	at pressure of (MPa):	200.6135
Void volume by real density (mm <sup>3</sup> /g):	-110.73		
Accessible porosity (%):	0.96		
Inaccessible porosity (%):	-29.04		

Figure 26: Mercury porosity results for limestone

RESULTS WITHOUT  
COMPRESSIBILITY CORRECTION  
(AFTER ACID DISSOLUTION)

Total intruded volume (mm <sup>3</sup> /g):	35.36 at pressure of (Mpa):	200.7132	
Intrud. Vol (mm <sup>3</sup> ):	0.00		
Envelope density (g/cm <sup>3</sup> ):	2.7118		
Bulk density @ pressure (g/cm <sup>3</sup> ):	2.7118	at pressure of (MPa):	0.174
Apparent density (g/cm <sup>3</sup> ):	2.9994	at pressure of (MPa):	200.7132
Void volume by real density (mm <sup>3</sup> /g):	-15504.26		
Accessible porosity (%):	9.59		
Inaccessible porosity (%):	-4214.07		

Figure 27: Mercury porosity result for marble

Figure above shows the result of mercury porosity test (changes between before and after acid dissolution) for limestone is lower than the marble after acid dissolution process. The reasons are, the particle size of marble is smaller than limestone caused the dissolution rate in marble is faster than in limestone. The heterogeneity of the elements in limestone are also affects the dissolution rate. Various mineralogy in limestone also decrease the rate of dissolution since there are least mineralogy content in marble that will caused the sample easy to dissolved with acid.

## **CHAPTER 5: CONCLUSIONS**

At this phase of the project, the author has concluded the followings:

- Dissolution of limestone in hydrochloric acid and formic acid is faster than marble.
- The different in porosity changes of marble after and before acid dissolution is higher than limestone.
- Varying geochemical conditions will cause different reaction to limestone and marble. Formic acid reacts faster than hydrochloric acid. So, formic acid is most practical acid for acid fracturing in limestone and marble rather than HCl acid.

Thus, the outcome of this project/ study is very much anticipated to be a success, so the author can share the valuable knowledge to others regarding to acid dissolution by hydrochloric acid and formic acid that can be used acid fracturing or matrix acidizing. It is a particularly significant factor for carbonate reservoirs, since traditionally recovery tends to be low from these fields. With continued collaboration to refine existing techniques and the commitment to develop specialist new technologies, the future for carbonate reservoirs is looking more productive.

## **CHAPTER 6: REFERENCES**

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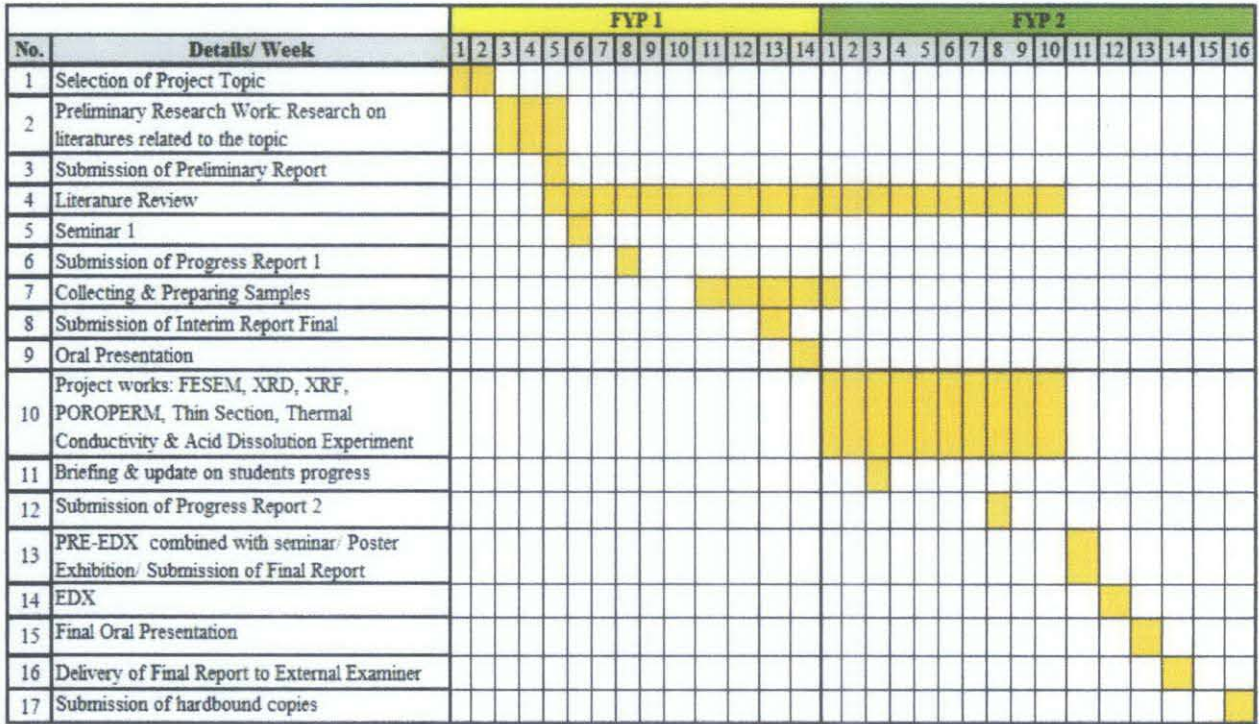
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**CHAPTER 7: APPENDICES****Appendix A: KEY MILESTONE**

No	Action Item	Action By	Date	Note
1	Briefing & update on students progress	Coordinator / Students / Supervisors	8 FEB	WEEK 3
2	Project work commences	Students		WEEK 1 - 14
3.	Submission of Progress Report	Students	16 MARCH	WEEK 8
4.	PRE-EDX combined with seminar/ Poster Exhibition/ Submission of Final Report (CD Softcopy & Softbound)	Students / Supervisor / Internal Examiner / Coordinator	4 APRIL	WEEK 11
5.	EDX	Supervisors / FYP Committee	11 APRIL	WEEK 12
6.	Final Oral Presentation	Students / Supervisors	20 APRIL 2011	WEEK 13
7.	Delivery of Final Report to External Examiner / Marking by External Examiner	FYP Committee / Coordinator	20-27 APRIL 2011	WEEK 14
8.	Submission of hardbound copies	Students	04 MAY 2011	WEEK 16



**Appendix B: GANTT CHART**



**Appendix C: Thermal Conductivity Result (details)**

SAMPLE	PHASE	Q	H	W	L	A	T1	T2	ΔT	k	
		watts	m	m	m	m <sup>2</sup>	°C	°C	°C		
#1 (limestone)	a	40.00	0.033	0.100	0.080	0.003	28.300	28.900	0.600	0.449	
		40.00	0.033	0.100	0.080	0.003	28.600	29.600	1.000	0.269	
		40.00	0.033	0.100	0.080	0.003	29.600	30.500	0.900	0.299	
		40.00	0.033	0.100	0.080	0.003	29.600	30.600	1.000	0.269	
		40.00	0.033	0.100	0.080	0.003	29.700	31.800	2.100	0.128	
		Average									0.283
	b	40.00	0.033	0.100	0.080	0.003	30.000	30.400	0.400	0.673	
		40.00	0.033	0.100	0.080	0.003	30.100	31.600	1.500	0.180	
		40.00	0.033	0.100	0.080	0.003	30.200	32.000	1.800	0.150	
		40.00	0.033	0.100	0.080	0.003	30.300	32.100	1.800	0.150	
40.00		0.033	0.100	0.080	0.003	30.400	32.200	1.800	0.150		
40.00		0.033	0.100	0.080	0.003	30.400	32.200	1.800	0.150		
40.00		0.033	0.100	0.080	0.003	30.300	32.700	2.400	0.112		
	Average									0.223	
c	40.00	0.080	0.100	0.033	0.008	29.100	29.800	0.700	0.065		
	40.00	0.080	0.100	0.033	0.008	29.300	30.300	1.000	0.046		
	40.00	0.080	0.100	0.033	0.008	29.500	30.500	1.000	0.046		
	40.00	0.080	0.100	0.033	0.008	29.600	30.600	1.000	0.046		
	40.00	0.080	0.100	0.033	0.008	29.800	30.800	1.000	0.046		
	40.00	0.080	0.100	0.033	0.008	29.900	30.900	1.000	0.046		
	40.00	0.080	0.100	0.033	0.008	30.200	31.100	0.900	0.051		
		Average									0.049
d	40.00	0.080	0.100	0.033	0.008	30.500	30.800	0.300	0.153		
	40.00	0.080	0.100	0.033	0.008	30.900	31.300	0.400	0.115		
	40.00	0.080	0.100	0.033	0.008	31.000	31.500	0.500	0.092		
	40.00	0.080	0.100	0.033	0.008	31.300	31.800	0.500	0.092		
	40.00	0.080	0.100	0.033	0.008	31.500	32.200	0.700	0.065		
	40.00	0.080	0.100	0.033	0.008	31.700	32.400	0.700	0.065		
	40.00	0.080	0.100	0.033	0.008	31.900	32.700	0.800	0.057		
		Average									0.091

## FINAL YEAR PROJECT II

Final Report

WAN MOHD SHAFIE BIN WAN IBRAHIM (11736)

SAMPLE	PHASE	Q	H	W	L	A	T1	T2	$\Delta T$	k
		watts	m	m	m	m <sup>2</sup>	°C	°C	°C	
#2 (marble)	a	40.00	0.100	0.077	0.100	0.008	29.100	37.600	8.500	0.017
		40.00	0.100	0.077	0.100	0.008	29.300	37.000	7.700	0.019
		40.00	0.100	0.077	0.100	0.008	29.300	36.800	7.500	0.019
		40.00	0.100	0.077	0.100	0.008	29.400	37.600	8.200	0.018
		40.00	0.100	0.077	0.100	0.008	29.400	38.800	9.400	0.015
		40.00	0.100	0.077	0.100	0.008	29.500	39.600	10.100	0.014
		40.00	0.100	0.077	0.100	0.008	29.700	40.400	10.700	0.013
		Average								
	b	40.00	0.077	0.100	0.100	0.008	29.500	30.300	0.800	0.180
		40.00	0.077	0.100	0.100	0.008	29.700	30.600	0.900	0.160
		40.00	0.077	0.100	0.100	0.008	29.700	30.700	1.000	0.144
		40.00	0.077	0.100	0.100	0.008	29.800	31.000	1.200	0.120
		40.00	0.077	0.100	0.100	0.008	29.900	31.100	1.200	0.120
		40.00	0.077	0.100	0.100	0.008	29.900	31.300	1.400	0.103
		40.00	0.077	0.100	0.100	0.008	29.900	31.300	1.400	0.103
		Average								
	c	40.00	0.100	0.100	0.077	0.010	30.200	33.100	2.900	0.030
		40.00	0.100	0.100	0.077	0.010	30.400	33.600	3.200	0.027
		40.00	0.100	0.100	0.077	0.010	30.500	34.100	3.600	0.024
		40.00	0.100	0.100	0.077	0.010	30.500	34.300	3.800	0.023
		40.00	0.100	0.100	0.077	0.010	30.600	34.600	4.000	0.021
		40.00	0.100	0.100	0.077	0.010	30.600	35.000	4.400	0.019
		40.00	0.100	0.100	0.077	0.010	30.700	35.300	4.600	0.019
		Average								
	d	40.00	0.077	0.100	0.100	0.008	29.800	30.400	0.600	0.241
		40.00	0.077	0.100	0.100	0.008	29.800	30.500	0.700	0.206
		40.00	0.077	0.100	0.100	0.008	29.900	30.600	0.700	0.206
		40.00	0.077	0.100	0.100	0.008	29.900	30.700	0.800	0.180
		40.00	0.077	0.100	0.100	0.008	30.000	30.800	0.800	0.180
		40.00	0.077	0.100	0.100	0.008	30.000	30.800	0.800	0.180
		40.00	0.077	0.100	0.100	0.008	30.100	30.900	0.800	0.180
		Average								

SAMPLE	PHASE	Q	H	W	L	A	T1	T2	ΔT	k
		watts	m	m	m	m <sup>2</sup>	°C	°C	°C	
#3 (marble)	a	40.00	0.100	0.080	0.085	0.008	30.400	36.600	6.200	0.019
		40.00	0.100	0.080	0.085	0.008	30.500	37.200	6.700	0.018
		40.00	0.100	0.080	0.085	0.008	30.500	37.700	7.200	0.016
		40.00	0.100	0.080	0.085	0.008	30.600	37.900	7.300	0.016
		40.00	0.100	0.080	0.085	0.008	31.600	38.500	6.900	0.017
		40.00	0.100	0.080	0.085	0.008	32.100	37.800	5.700	0.021
		40.00	0.100	0.080	0.085	0.008	33.900	36.200	2.300	0.051
		Average								
	b	40.00	0.080	0.085	0.100	0.007	30.800	31.500	0.700	0.233
		40.00	0.080	0.085	0.100	0.007	31.000	31.900	0.900	0.182
		40.00	0.080	0.085	0.100	0.007	31.100	32.100	1.000	0.163
		40.00	0.080	0.085	0.100	0.007	31.300	32.300	1.000	0.163
		40.00	0.080	0.085	0.100	0.007	31.400	32.400	1.000	0.163
		40.00	0.080	0.085	0.100	0.007	31.400	32.700	1.300	0.126
		40.00	0.080	0.085	0.100	0.007	31.500	32.900	1.400	0.117
		Average								
	c	40.00	0.085	0.100	0.080	0.009	30.700	34.500	3.800	0.028
		40.00	0.085	0.100	0.080	0.009	30.900	34.800	3.900	0.027
		40.00	0.085	0.100	0.080	0.009	30.900	35.200	4.300	0.024
		40.00	0.085	0.100	0.080	0.009	31.000	35.600	4.600	0.023
		40.00	0.085	0.100	0.080	0.009	31.100	36.200	5.100	0.021
		40.00	0.085	0.100	0.080	0.009	31.200	36.800	5.600	0.019
		40.00	0.085	0.100	0.080	0.009	31.300	37.000	5.700	0.018
		Average								
	d	40.00	0.080	0.085	0.100	0.007	30.700	31.500	0.800	0.204
		40.00	0.080	0.085	0.100	0.007	30.900	31.900	1.000	0.163
		40.00	0.080	0.085	0.100	0.007	30.900	32.100	1.200	0.136
		40.00	0.080	0.085	0.100	0.007	31.000	32.300	1.300	0.126
		40.00	0.080	0.085	0.100	0.007	31.100	32.400	1.300	0.126
		40.00	0.080	0.085	0.100	0.007	31.200	32.700	1.500	0.109
		40.00	0.080	0.085	0.100	0.007	31.300	32.900	1.600	0.102
		Average								