

**INVESTIGATION OF GEOPOLYMER DEGRADATION IN CONTROL  
RELEASE FERTILIZER (CRF) APPLICATION**

By

**NUR AMIRA BINTI ABDULLAH HALIM**

**FINAL PROJECT REPORT**

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

Universiti Teknologi PETRONAS

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Perak Darul Ridzuan

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# CERTIFICATION OF APPROVAL

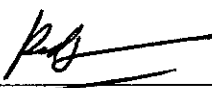
## INVESTIGATION OF GEOPOLYMER DEGRADATION KINETICS IN CONTROL RELEASE FERTILIZER (CRF) APPLICATION

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A project dissertation submitted to the  
Department of Chemical Engineering  
Universiti Teknologi PETRONAS  
in partial fulfilment of the requirement for the  
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(Chemical Engineering)

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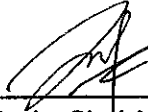
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TRONOH, PERAK

May 2012

## **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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Nur Amira Binti Abdullah Halim

## ABSTRACT

Geopolymer is one of the new control release fertilizer (CRF) that will have the potential to replace the current CRFs as it is eco-friendly and manufactured through sustainable production processes. Degradation of the geopolymer is being studied as the geopolymer needs to be degraded in order to be used as urea coating material. Inefficient of conventional fertilizer that makes nutrient lost to the environment that will cause serious environmental problems as well as the polymer coating material that will leave undesired residue of synthetic polymer material that lead to the contamination of soil, lead to the research of developing a new control release fertilizer using geopolymer. With the improvement in the performance due to the controllable structure, lower manufacturing cost due to proprietary process and low cost raw materials, and superior eco-friendly characteristics, geopolymer-based CRFs seem to provide a novel fertilizer and associated manufacturing technology solution with improved performance. Geopolymer degradation study is important to see whether geopolymer can degrade or not and suitable to be used as urea coating material. The effect of pH towards geopolymer degradation also being studied to see what is the effect of changing the pH towards the degradation of geopolymer. Amylase enzyme is used in this research to help the degradation of geopolymer. To study the geopolymer degradation, Fourier Transform Infrared (FTIR) is used as FTIR can show provide info on analyte chemical structure like bonding and functional group. Analysis of the infrared spectra showed a slight decreased of certain specific peaks of geopolymer. This showed that geopolymer can degrade, but it takes time to fully degrade. Some modification for the amylase enzyme concentration and geopolymer can be made for a better result for geopolymer degradation.

## **ACKNOWLEDGEMENTS**

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# CHAPTER 1

## INTRODUCTION

### 1.1 BACKGROUND OF STUDY

Control release fertilizer (CRF) that are based on eco-friendly geopolymer and manufactured through sustainable production processes is the new technology that is being developed nowadays as geopolymer is clean to the environment and also low cost of the production. Development of CRFs is very important due to its impact to the environment such as soil and water acidification, contamination of surface and groundwater resources, increased ozone depletion and greenhouse gas levels, and loss of biodiversity (Plant Management Network International , 2008).

It is found that geopolymer coating have the potential to be developed as urea coating. Geopolymer, which is basically refer as artificial cement-like aluminosilicates, seems to have the potential to be develop as urea coating material. The main properties to produce geopolymer are rice husk ash or fly ash, aside from another material such as Sodium Hydroxide and Sodium Silicate. Studies have proved that geopolymer is clean to the environment as the chemicals that use to make geopolymer are in low concentration, aside from the rice husk ash that is already proven clean to the environment as it doesn't has any hazardous properties inside it.

In order to make the geopolymer as fertilizer coating material, geopolymer needs to be degradable. Method for the geopolymer degradation has been studied and it was found that  $\alpha$ -amylase enzyme can be used to help studying the degradation of geopolymer. FTIR can show the pattern of the changing for certain peaks of geopolymer to show the degradation of geopolymer after adding some enzyme to the geopolymer.

## **1.2 PROBLEM STATEMENT**

The fertilizer industry faces a continuing challenge to improve its products as they need to increase the efficiency of the fertilizer usage. This is due to the nutrient loss to environment that will make the plant uptake nutrient will be reduced. The nutrients lost to the environment will also contaminant the environment such as land, water, and air. In order to solve these problems, many types of Control Release Fertilizer (CRFs) have been introduced, especially by using polymer based coating. To prepare the polymer membrane coated fertilizer, the organic solvents need to be used. The usage of organic solvents will cause serious environmental pollution and also the cost of production also will increase.

Therefore, the recent studies have found that geopolymer can be developing as fertilizer coating. Studies have proved that geopolymer based coating will make less impact to the environment as compared to the polymer based coating fertilizer. This is because the materials that use to make the geopolymer itself is very environmental friendly as rice husk and fly ash are the main material that use to produce the geopolymer.

### 1.3

### OBJECTIVE

This project is a study about the application of geopolymer in Control Release Fertilizer (CRF). CRFs are broadly defined as products that release the nutrients to the soil for plant uptake at pre-determined time and rate. Alpha amylase enzyme has been used in order to study the enzyme activity on a surface bound substrate film.

This research is to study the degradation rate of geopolymer in CRF application. A few methods will be used in order to see whether the geopolymer can degrade or not.

There are some factors that will affect the degradation of geopolymer, such as the pH, the moisture of the soil and also the surrounding and soil temperature. As soil may have different pH, the study will also determine the effect of pH towards the degradation rate of geopolymer.

#### **1.4 SCOPE OF STUDY**

This project will focus on the application of geopolymer in control release fertilizer, where the factors that will affect the nutrient release of fertilizer will be determined. Generally, temperature and sometimes moisture are the main factors affecting nutrient release of CRF (Nano Voltaix, 2007). PH is another factor that will also affect the nutrient release of fertilizer. In this project, the effect of pH to the nutrient release of fertilizer will be the focus, aside from the geopolymer degradation stud itself.

## **1.5 RELEVANCY OF THE PROJECT**

Geopolymer based coating fertilizer is being develop recently as geopolymer seems to be environmental friendly and easy to produce, aside from the low cost of the production due to the main material to produce the geopolymer, which is rice husk and fly ash can be easily found in Malaysia at low cost. Geopolymer seems to be the best replacement for polymer-based coating control release fertilizer as polymer is not an environmental friendly material as it may leave an undesired residue of polymer synthetic material that may lead to undesirable contamination of soil.

## CHAPTER 2

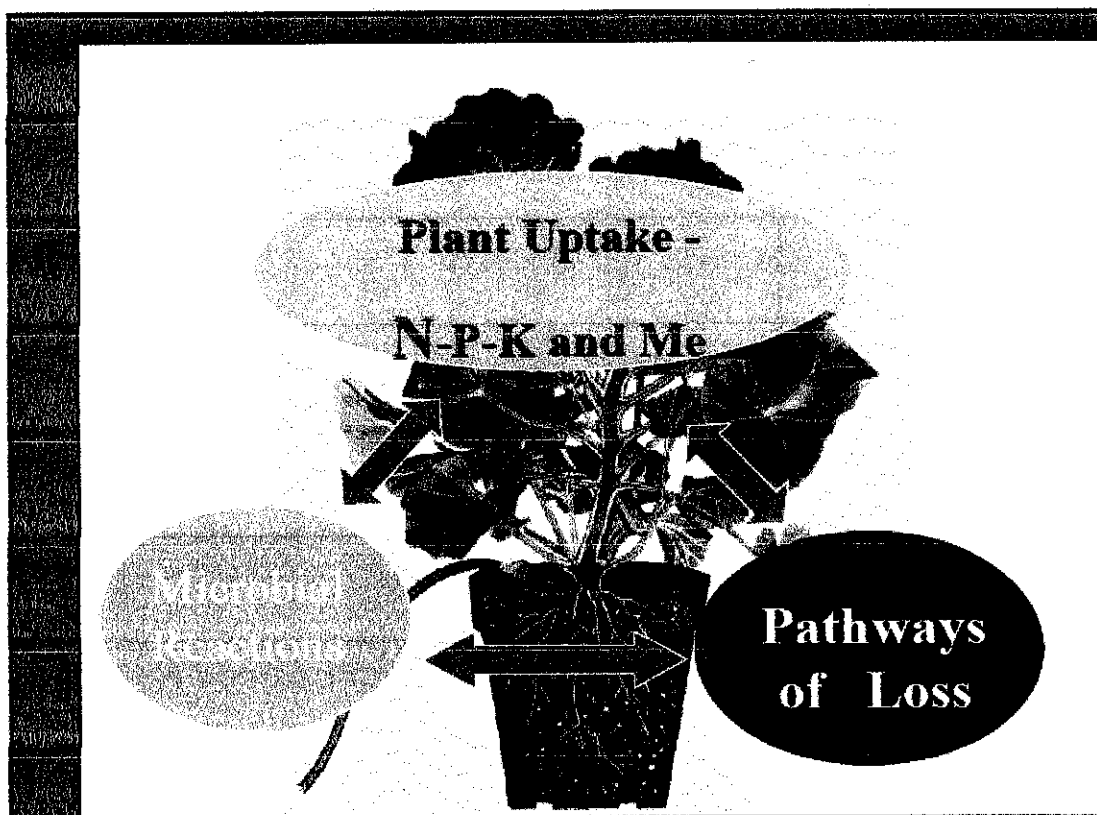
### LITERATURE REVIEW

#### 2.1 CONTROL RELEASE FERTILIZER (CRF)

The *Association of American Plant Food Control Officials (AAPFCO)* defines that control release fertilizer (CRF) is a fertilizer containing a plant nutrient in a form which delays its availability for plant uptake and use after application, or which extends its availability to the plant significantly longer than a reference rapidly available nutrient fertilizer such as ammonium nitrate or urea, ammonium phosphate or potassium chloride. Such delay of initial availability or extended time of continued availability may occur by a variety of mechanisms. These include controlled water solubility of the material by semi-permeable coatings. Occlusion, protein materials, or other chemical forms, by slow hydrolysis of water-soluble low molecular weight compounds, or by other unknown means (Trenkel, 2010).

Recently, the fertilizer industry faces to improve the efficiency of its products. This improvement is done through improvement of fertilizers that already in use or through development of new specific fertilizer types (MAENE, 1995; TRENKEL et al, 1988). Many types of control release fertilizer have been invented such as sulfur and polymer coated control release fertilizer. Sulfur coated control release fertilizer can break easily with sulfur. The amount of sulfur will increase in order to ensure the coating effect. These will resulting in low nutrient content, and high production costs (Lin Fan, 2010).

As for the polymer coated control release fertilizer, the usage of the polymer in order to make the urea will lead to the contamination of soil as some undesirable residue of synthetic polymer material may be release and will be absorbed into the soil and cause serious environmental problems when the usage of polymer coated urea is used widely.



**Figure 1: Cycle in fertilizer usage (Shaviv, 2005)**

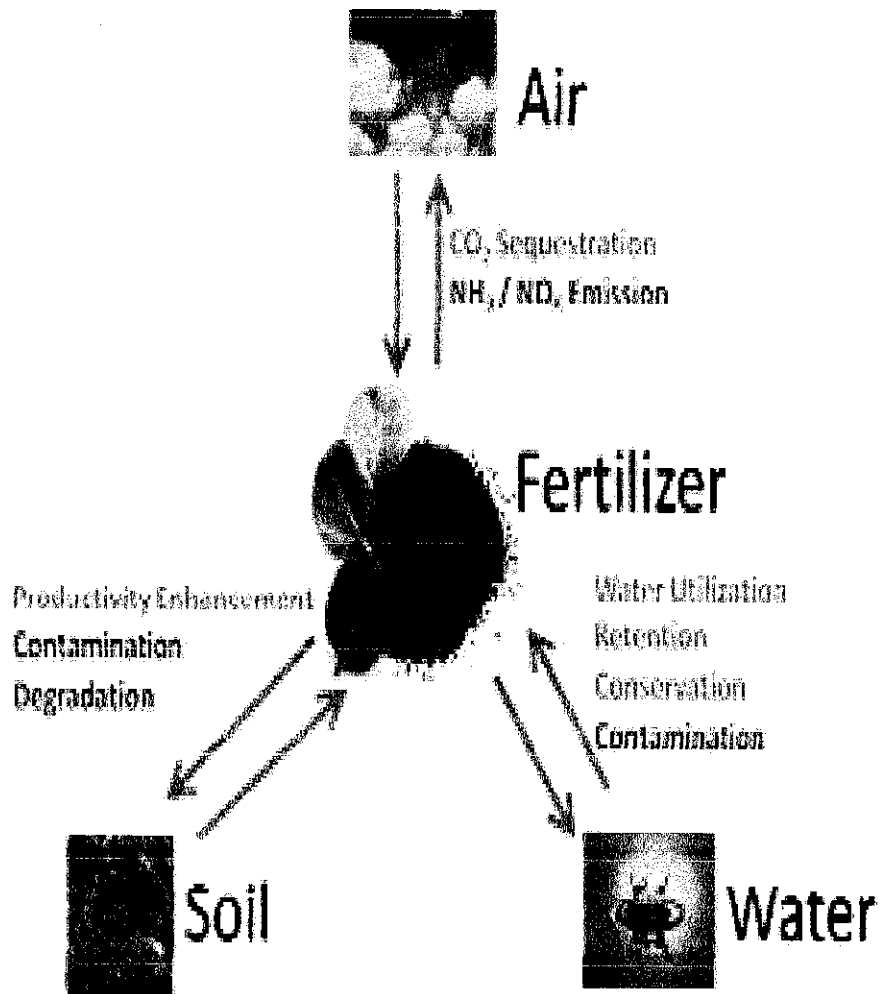


## 2.2 ADVANTAGE OF CRF

Control release fertilizer (CRF) technology is being developed nowadays as today environment is in difficult stage where we need to take serious care towards the environment in order to make sure that future generation can still live in this earth comfortably without serious environmental problems. By developing the technology of CRF to find the most environmental friendly urea, aside from most efficient urea, the environment can be also be saved.

There are some advantages in the CRF that lead to the development of its research nowadays. According to (Trenkel, 2010), by using controlled release fertilizer, it can decrease the nutrient losses to the environment, thus enhances the nutrient use efficiency. The toxicity, that particularly to seedlings, which can result from the application of soluble conventional fertilizers through high ion concentrations inducing osmotic stress and specific damage to plants at different growth stages can be reduce by using the controlled release fertilizer. The lodging and injury from ammonium ions may also reduce that made controlled release fertilizer, especially for those that release nutrient in a sigmoidal pattern, can contribute to improved argonomic safety, permitting co-situs application (Aglukon, 1992, 1993; Grace Sierra, 1993, 1994; Shaviv, 2005; Shaviv and Mikkelsen, 1993a; Shoji, 2005; Sierra, 1991a, 1991b; Tachibana, 2007).

Some other advantages of CRF include that the fertilizer burn is not a problem with CRF even at high rates of application. Also, because fertilizers are released at a slower rate throughout the season; plants are able to take up most of the fertilizers without waste by leaching. By using CRF, it reduced capital and labor outlay in horticultural crop production. CRF can also reduce the seed or seedling damage from high local concentrations of salts and reduce leaf burn from heavy rates of surface-applied fertilizers, besides improving the storage and handling properties of fertilizer materials (Shaviv, 2005).



**Figure 2: Cycle of Fertilizer (Nano Voltaix, 2007)**

### 2.3 TRENDS & TECHNOLOGIES OF CRF

Many types of CRF have been invented nowadays to improve the usage efficiency of fertilizer. CRFs primarily vary in terms of their nutrient formulations, estimated product longevities, and mechanisms of nutrient release. Urea-formaldehyde reaction products, which also known as Nitroform, Ureaform, Methylene Urea, Blue Chip, Nutralene or Methex, represent one of the oldest controlled-release nitrogen technologies, having been first produced in 1936 and commercialized in 1955.

Sulfur-coated fertilizers, which called the Tennessee Valley Authority developed SCU technology in the 1960s and 1970s. Sulfur was chosen as the principal coating material because of its low cost and its value as a secondary nutrient. It is often marketed for use in the turf grass industry.

Polymer-coated fertilizers (PCFs) represent the most technically advanced state of the art in terms of controlling product longevity and providing nutrient efficiency. PCFs were manufactured as early as 1970 in Japan. Polymer coatings can be categorized as either thermoset resins or thermoplastic resins. Because of the relatively high cost of the coatings on most polymer-coated products, their use has been restricted mostly to high-value applications. Many different PCFs are marketed for use in continuous production of forest tree seedlings (Jain, 2007). Nutricote, Osmocote and Polyon are the most common three marketed products types of polymer coated fertilizer.

Multicote products also has been introduce, in the production of multicote products, the fertilizer granules are heated in a rotating pan and treated with fatty acid and metal hydroxide, such as stearic acid and calcium hydroxide. The coating weights of multicote are relatively large if compared to other technologies, but this problem is offset by the relatively low cost of the coating materials. Substrates coated include potassium nitrate, urea, and triple superphosphate. The various coated components are blended together into different grades, which are marketed under the Multicote name.

Polymer/sulfur coated fertilizers (PSCF), which are the hybrid products that utilize a primary coating of sulfur and a secondary polymer coat, were developed to deliver controlled- release performance approaching polymer-coated fertilizers, but at a much reduced cost. Because of its low cost, sulfur is employed as the primary coating. The polymers in this case are chosen to provide a continuous membrane through which water and nutrients must diffuse, unlike the soft wax sealants used to cover imperfections in the sulfur coatings of SCUs. The rate of water diffusion into the particle is controlled by the water permeability characteristic of the polymer. The combination of these two coatings permits a positive cost/benefit value over products with singular coatings of sulfur or polymer.

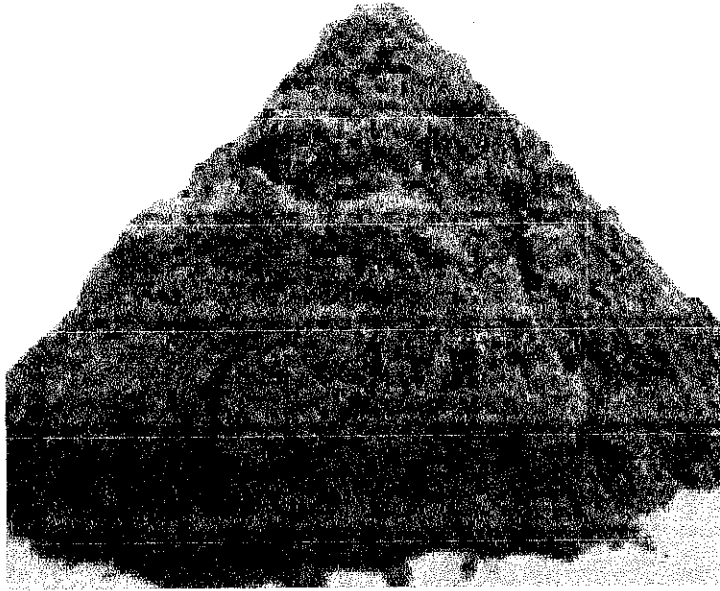
Geopolymer-based control release fertilizer is the newest technology and still under development. Many researches are still studying the application of geopolymer as urea coating material. Geopolymer coated controlled release fertilizer seems to have the potential to replace the current controlled release fertilizer as geopolymer is environmental friendly material. By introducing the geopolymer coated control release fertilizer, the impact of fertilizer usage towards the environment can be reduced.

## **2.4 GEOPOLYMER-COATING CONTROL RELEASE FERTILIZER**

In this research, the usage of geopolymer in the application of urea coating material is conducted. Basically, geopolymer can be synthesized by polycondensation reaction of geopolymeric precursor, and alkali polysilicates, where geopolymer is actually is a type of amorphous aluminosilicate cementitious material. Geopolymer have been increasingly applied in construction, fire protection, thermal insulation, etc.

In the environmental point of view, geopolymer seems to produce less CO<sub>2</sub> emission and consume less energy. This will reduce the effect of global warming and help to save our environment. Geopolymer is said to be environmental friendly also due to the factor that geopolymer is made from environmental friendly material, which is rice husk or rice husk ash. Other chemicals that use in preparation of geopolymer, which are sodium silicate and sodium hydroxide also are in low concentration. So, it does not contaminate the soil and harm the environment.

Geopolymer also is easy in the manufacturing process as the main materials that use to produce geopolymer are fly ash and rice husk ash, which can be easily find in Malaysia, aside from other chemicals such as sodium silicate and sodium hydroxide (Zongjin Li). The manufacturing of geopolymer also seems to be at low cost raw materials, due to the availability of the raw material in Malaysia with huge quantity.



**Figure 3: Fly ash**



**Figure 4: Rice husk ash**

## 2.5 EFFECT OF PH TOWARD DEGRADATION OF GEOPOLYMER

In studying the degradation of geopolymer, the things that should be kept in mind is that there are some factors that will affect the degradation rate of the geopolymer. The most influence factors that affecting the degradation of geopolymer are pH, temperature, and moisture content in the soil. In this study, pH effect toward the degradation rate of geopolymer is being studied.

In order to investigate the effect of the temperature and pH to the degradation of geopolymer coating,  $\alpha$ -amylase enzyme will be used. In general,  $\alpha$ -amylase is an enzyme that hydrolyses alpha-bonds of large alpha-linked polysaccharides, such as starch, and glycogen, yielding glucose and maltose. Also present in seeds containing starch as a food reserve, and is secreted by many fungi,  $\alpha$ -amylase enzyme is the major form of amylase found in humans and other mammals (Mystrica, 2012). As  $\alpha$ -amylase is relatively sensitive to temperature and pH conditions, investigating the effect of both temperature and pH is appropriate.

The effect of pH is very important to investigate as there will be certain range of pH that will allow the degradation of the geopolymer coating urea to be optimum. If the pH is not suitable, then the degradation of the geopolymer cannot occur.

Fourier Transform Infrared (FTIR) will be used in the research to see the change in the molecular structure of geopolymer after addition of pH buffer solution and also after the addition of amylase enzyme.

## **2.6 ALPHA AMYLASE ENZYMES**

A-amylase, which is the major form of amylase found in humans and other mammals, hydrolyses alpha bonds of large, alpha-linked polysaccharides, such as starch and glycogen, yielding glucose and maltose. Alpha amylase is the major form of amylase that found in the humans and other mammals. Also present in seeds containing starch as a food reserve, amylase is secreted by many fungi.

Amylase is the most prominent in pancreatic juice and saliva, although it can be found in many tissues. Each of it has its own isoform of human antibodies. It can be separated in testing by using monoclonal antibodies and also behave differently on isoelectric focusing.



## 2.6 FOURIER TRANSFORM INFRARED (FTIR)

FTIR stands for Fourier Transform Infrared, is the preferred method of infrared spectroscopy to obtain an infrared spectrum of absorption, emission, photoconductivity or Raman scattering of a solid, liquid or gas. Generally, the functions of FTIR are as follow:

- To identify the unknown materials
- To determine the quality or consistency of a sample
- To determine the amount of component in a mixture

FTIR is useful in identifying the chemicals that are either organic or inorganic. During the measurement, the infrared radiation is passed via the sample. Some of the infrared radiation is absorbed by the sample whilst some of it is transmitted through the sample. This leads to the producing of a spectrum which represents the molecular absorption and transmission. There is no two molecular structures can produce the same infrared. This advantage and its ability to collect spectral data in a wide spectral range make the infrared spectroscopy useful for several types of analysis.

Infrared (IR) light is known as an electromagnetic radiation with a wavelength longer than the visible light, measured from the nominal edge of visible red light at  $0.74\mu\text{m}$ , and extending conventionally to  $300\mu\text{m}$ . Microscopically, IR light emits or absorbs by molecules when they change the rotational-vibrational movements.

Infrared is useful in industrial, scientific, and medical applications. The applications of IR light include:

- Night-vision devices using infrared illumination allow people or animals to be observed without the observer being detected;
- In astronomy, imaging at infrared wavelengths allows observation of objects obscured by interstellar dust;
- Infrared imaging cameras to detect heat loss in insulated systems;
- Observing changing blood flow in the skin;
- study the degradation of organic and inorganic materials;
- Observing overheating of electrical apparatus, etc.

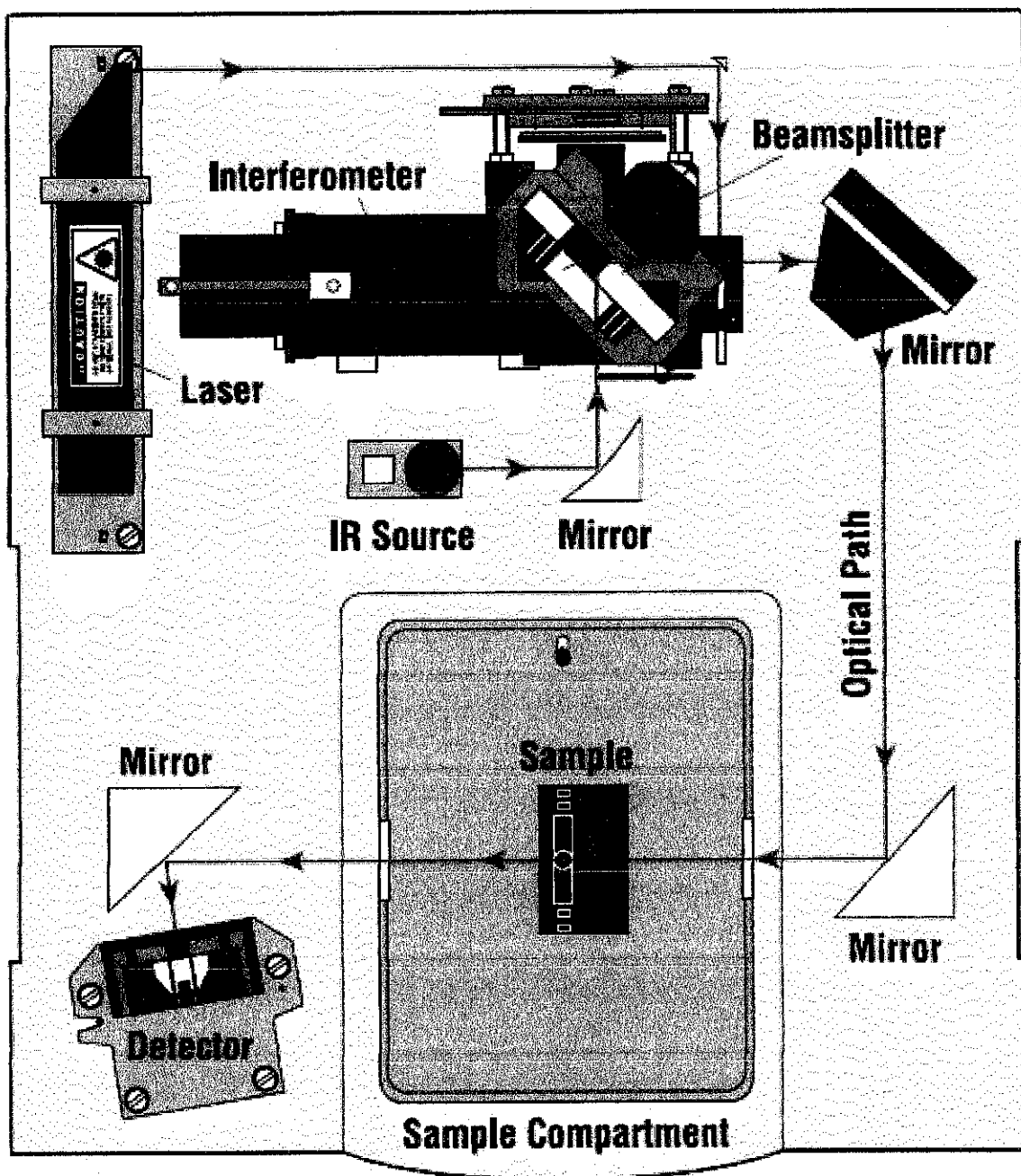


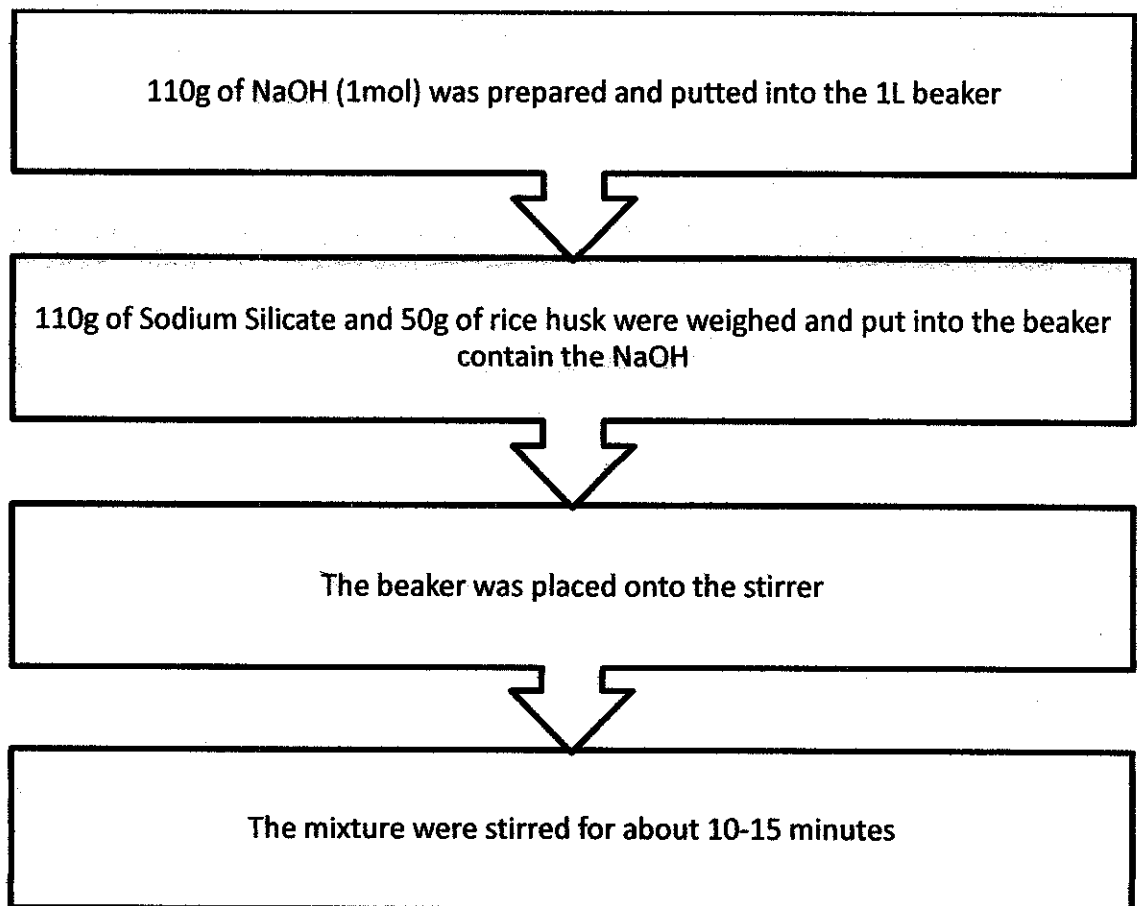
Figure 5: A Simple FTIR Spectrometer Layout

## CHAPTER 3

### METHODOLOGY

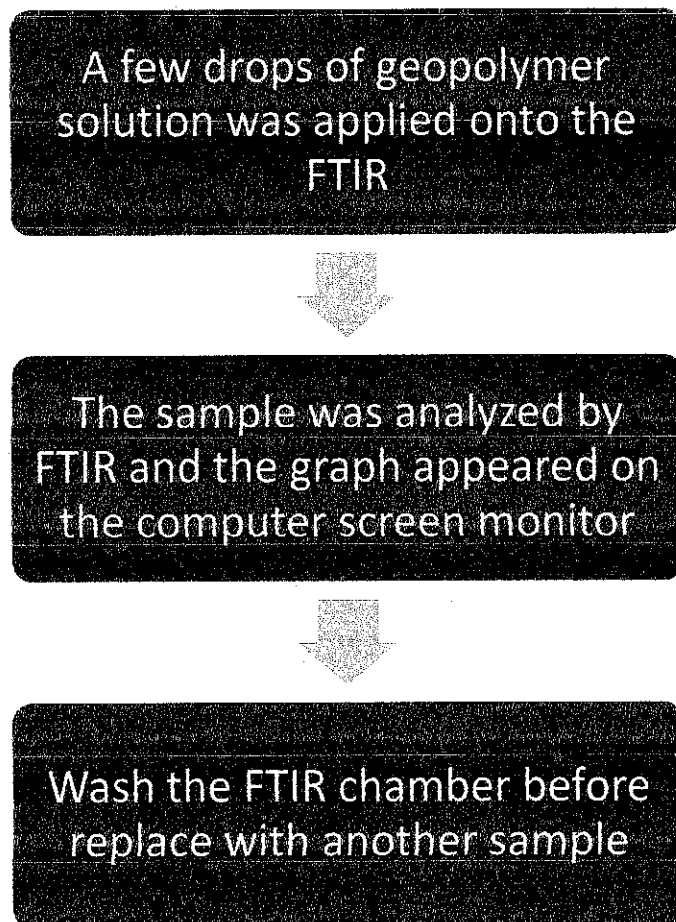
#### 3.1 RESEARCH METHODOLOGY

##### 3.1.1 Preparation of geopolymer solution



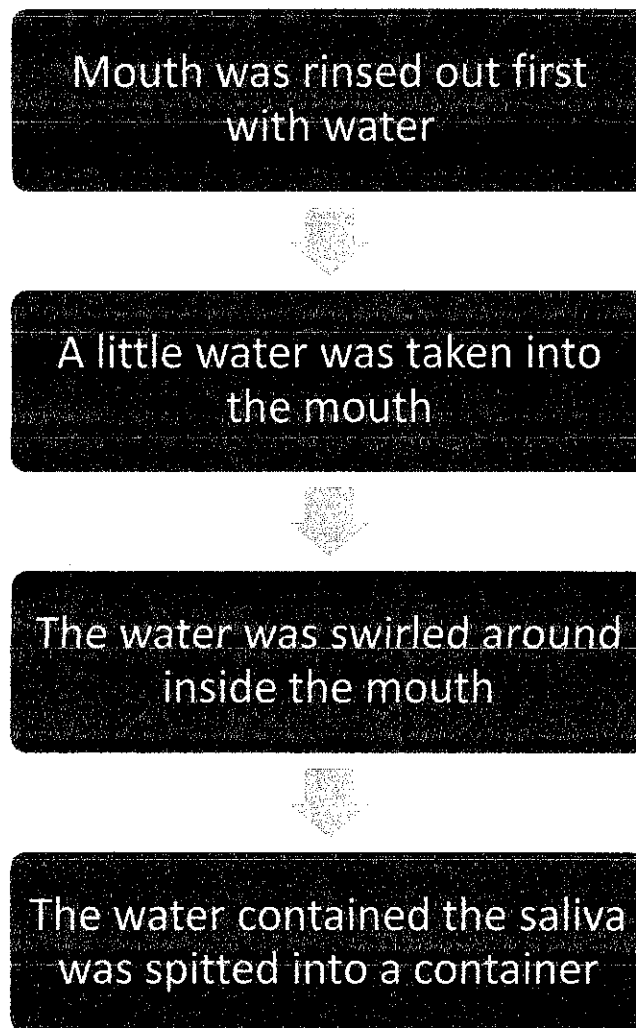
**Figure 6: Preparation of Geopolymer Solution**

### 3.1.2 Geopolymer Degradation Study For Different Sodium Hydroxide Concentration



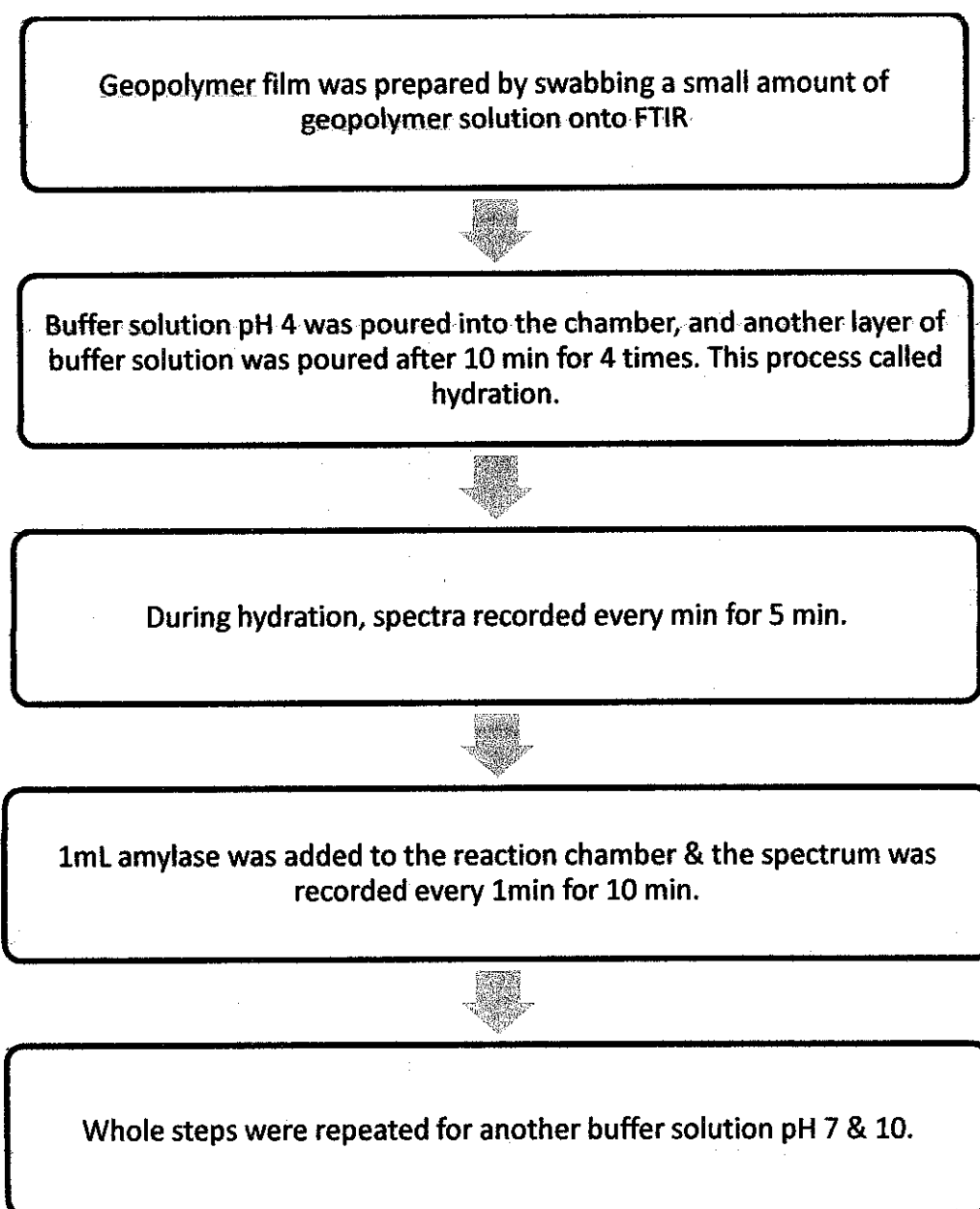
**Figure 7: Method for Geopolymer Degradation Study**

### 3.1.2 Preparation of $\alpha$ -amylase enzyme



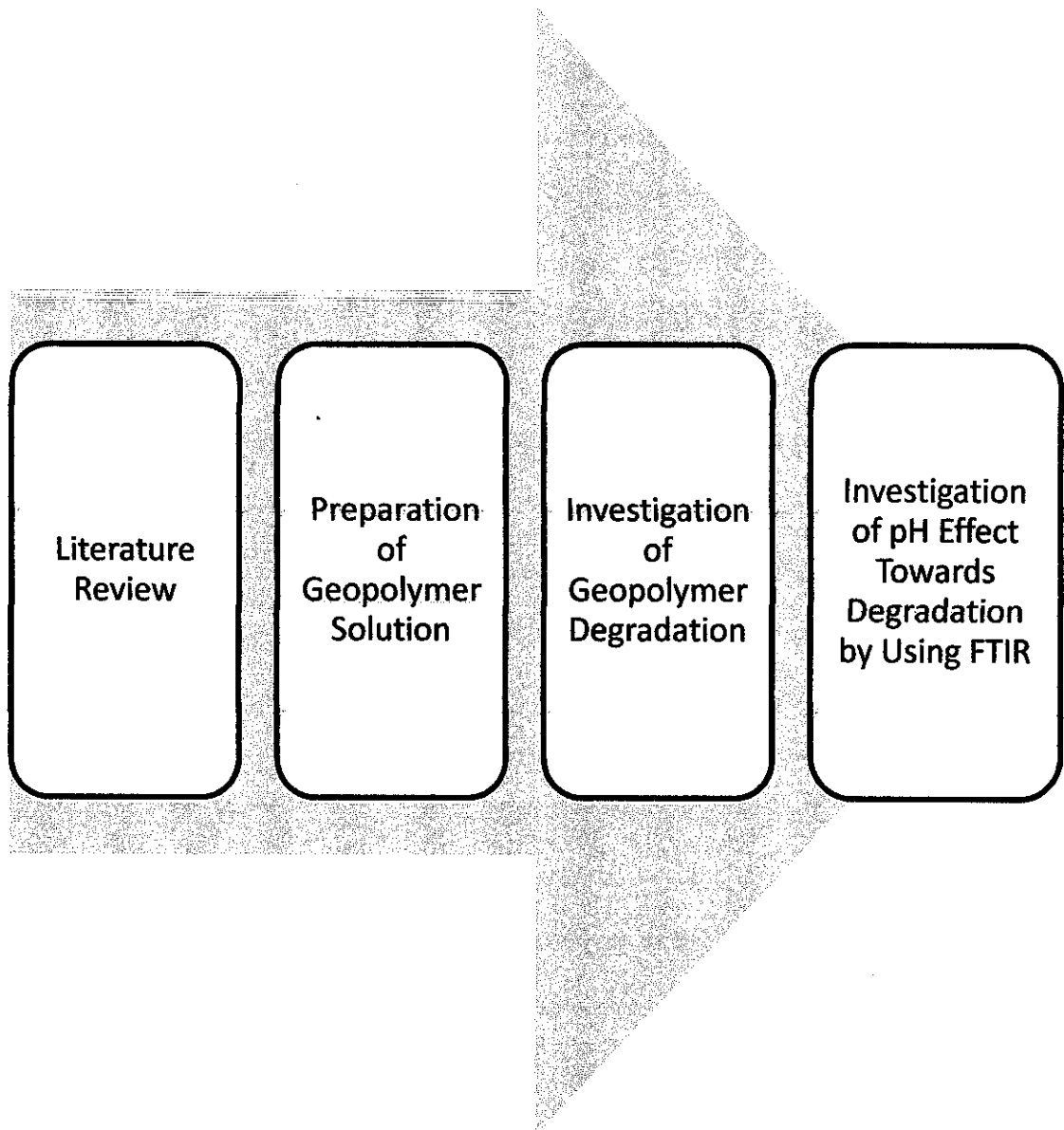
**Figure 8: Alpha amylase enzyme preparation**

### 3.1.3 Effect of pH towards degradation of rice husk ash geopolymer

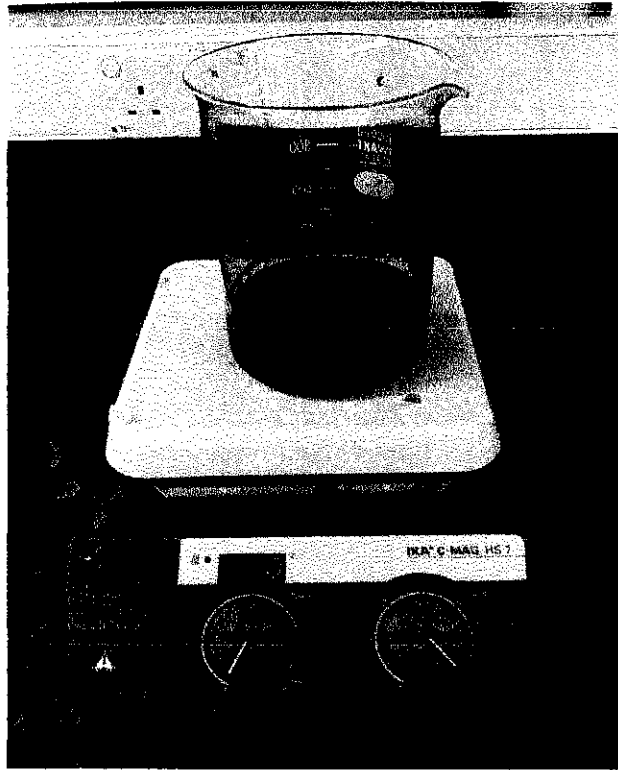


**Figure 9: Preparation of rice husk ash geopolymer mixture and FTIR test**

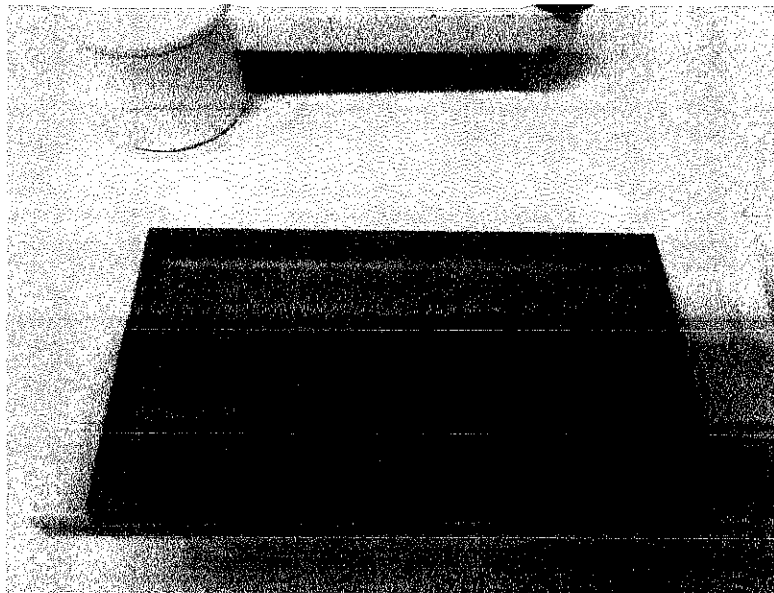
## 3.2 PROJECT ACTIVITIES



**Figure 10: Project Activities**

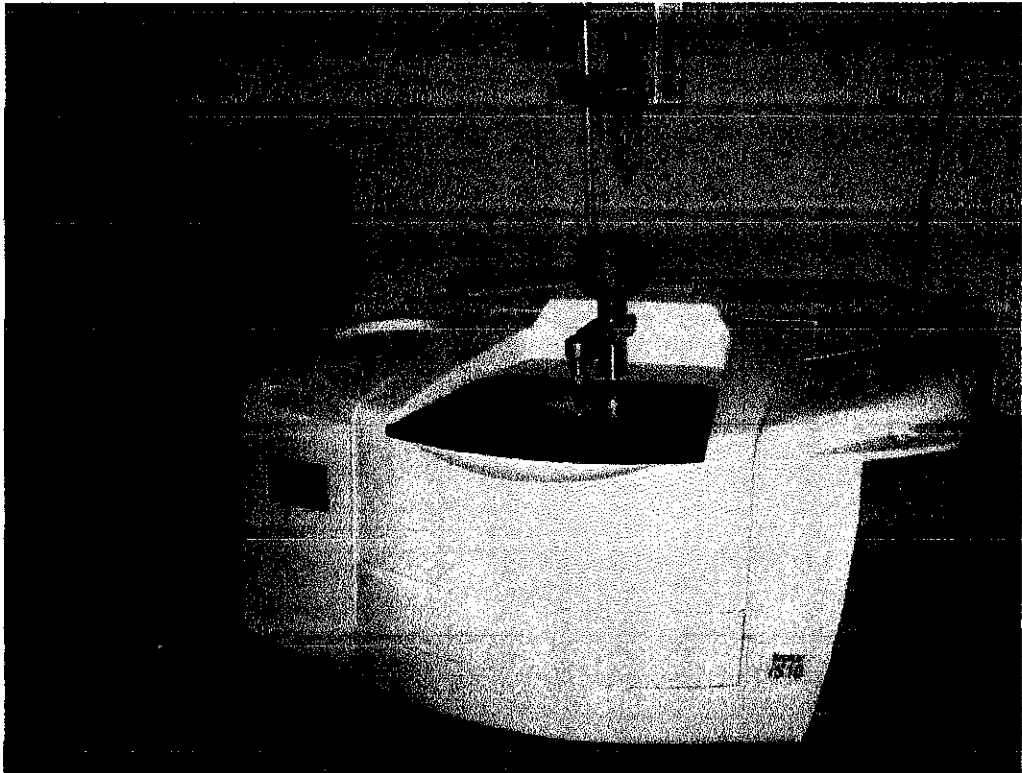


**Figure 11: Preparation of geopolymer solution**

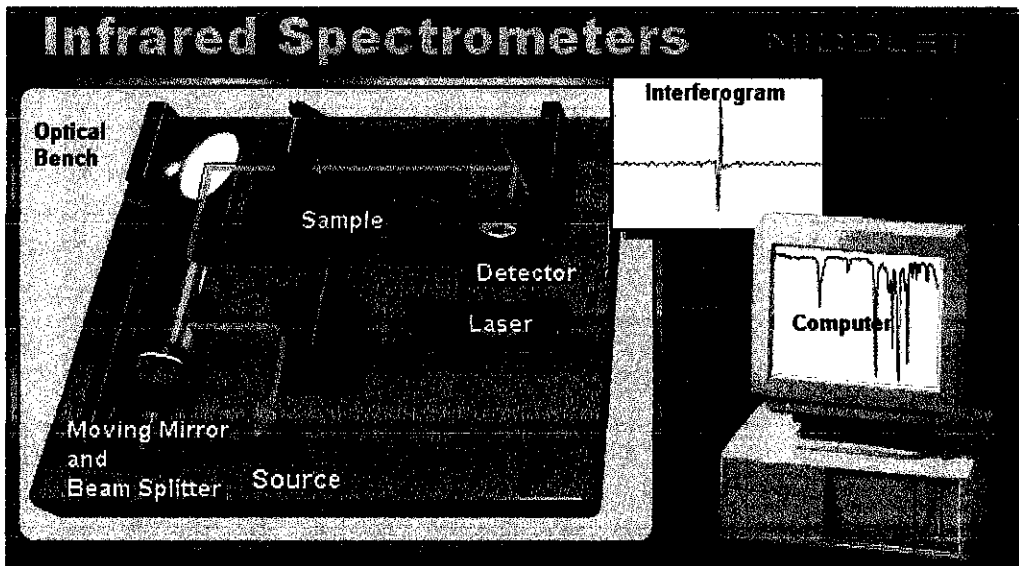


**Figure 12: FTIR crystal**





**Figure 13: FTIR equipment**



**Figure 14: How FTIR works**

### 3.3 GANTT CHART/ KEY MILESTONE

No	Detail/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	Find the journal and article regarding the methods to study the degradation of rice husk ash geopolymer.	■	■													
2	Book the Fourier Transform Infrared (FTIR) equipment.				■											
3	Preparation of geopolymer solution using rice husk ash.					■										
4	Preparation of progress report						■									
5	Submission of progress report								■							
6	Use FTIR to study the degradation of geopolymer									■						
7	Interpret the data from FTIR										■					
8	Preparation of poster											■				
9	Poster presentation												■			
10	Use FTIR in order to study the effect of pH towards the degradation of rice husk ash													■		

MID SEM BREAK



### **3.4 EQUIPMENTS AND SOFTWARE REQUIRED**

Below are some of the equipments and software that use in this research:

- Equipments: Fourier Transform Infrared (FTIR), oven, stirrer.
- Software: Microsoft Word, Microsoft Excel.

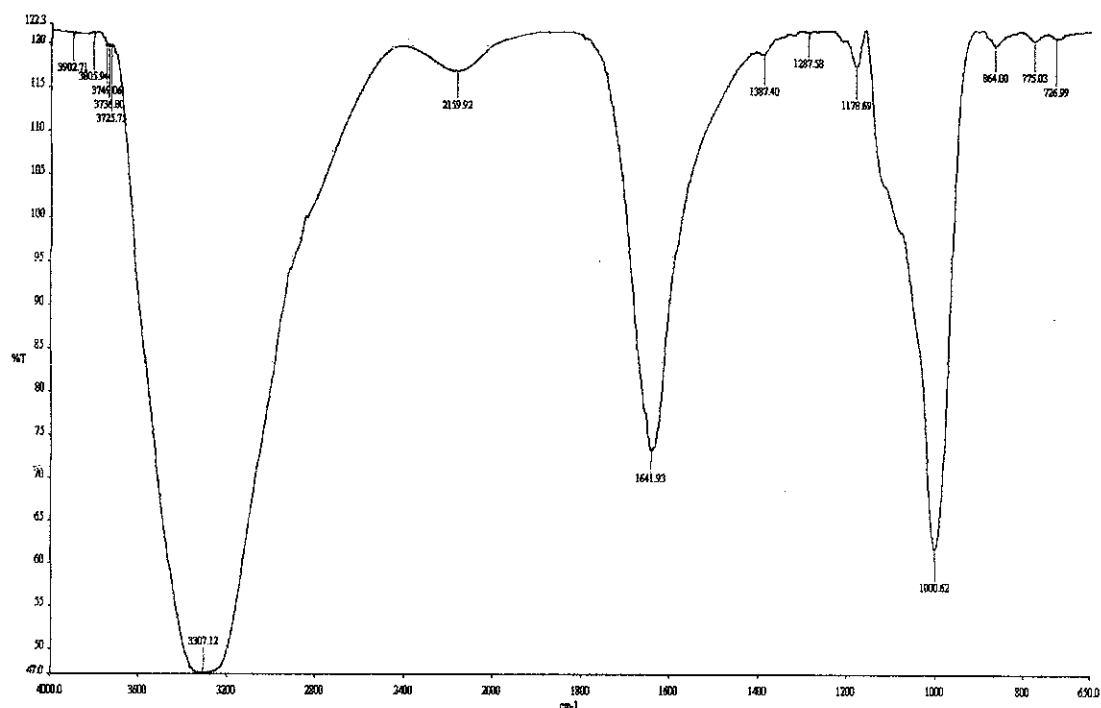
## CHAPTER 4 RESULTS AND DISCUSSION

### 4.1 RESULTS

#### 4.1.1 Fourier Transform Infrared (FTIR) Analysis

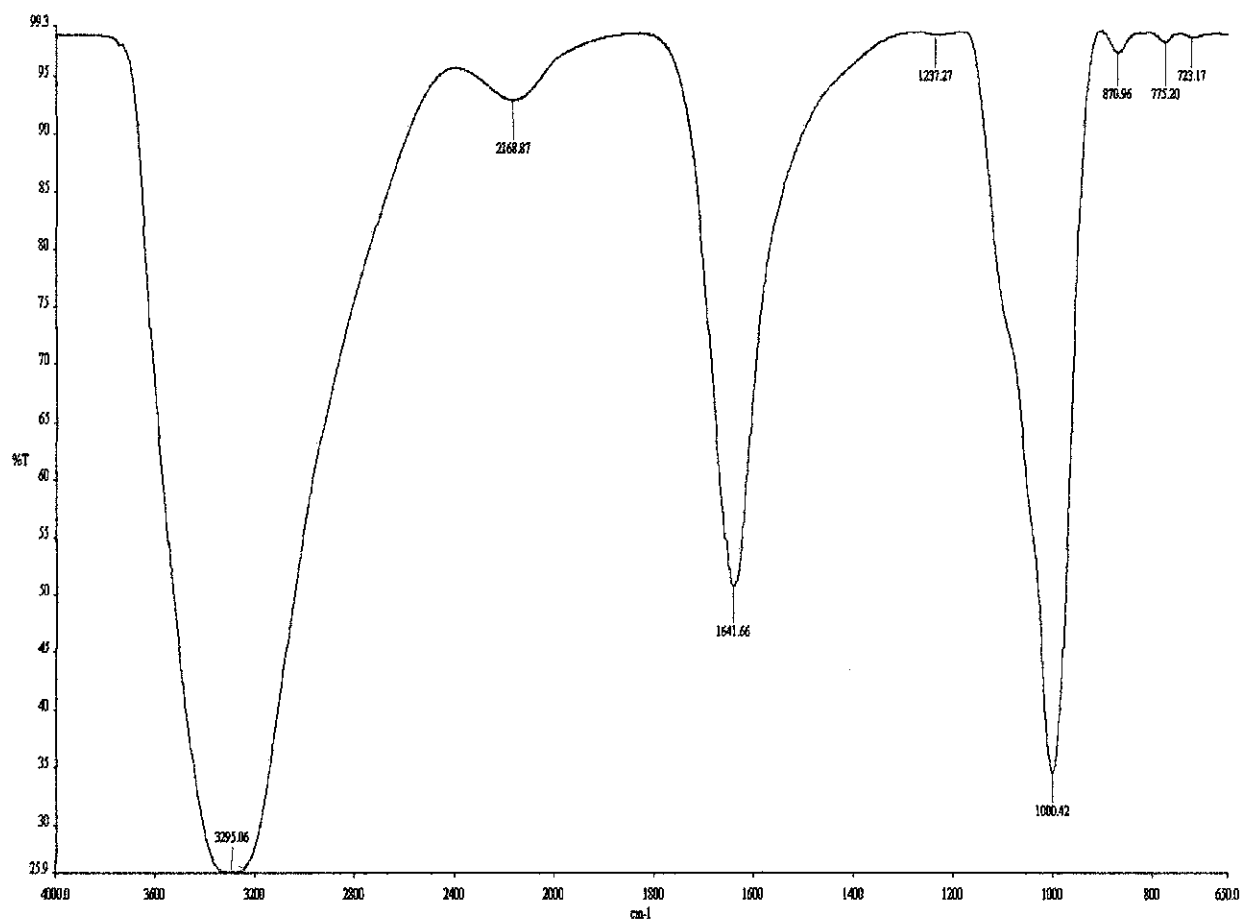
##### 4.1.1.1 Geopolymer Degradation Study With Different Concentration of Sodium Hydroxide

##### 1M Sodium Hydroxide before adding amylase enzyme



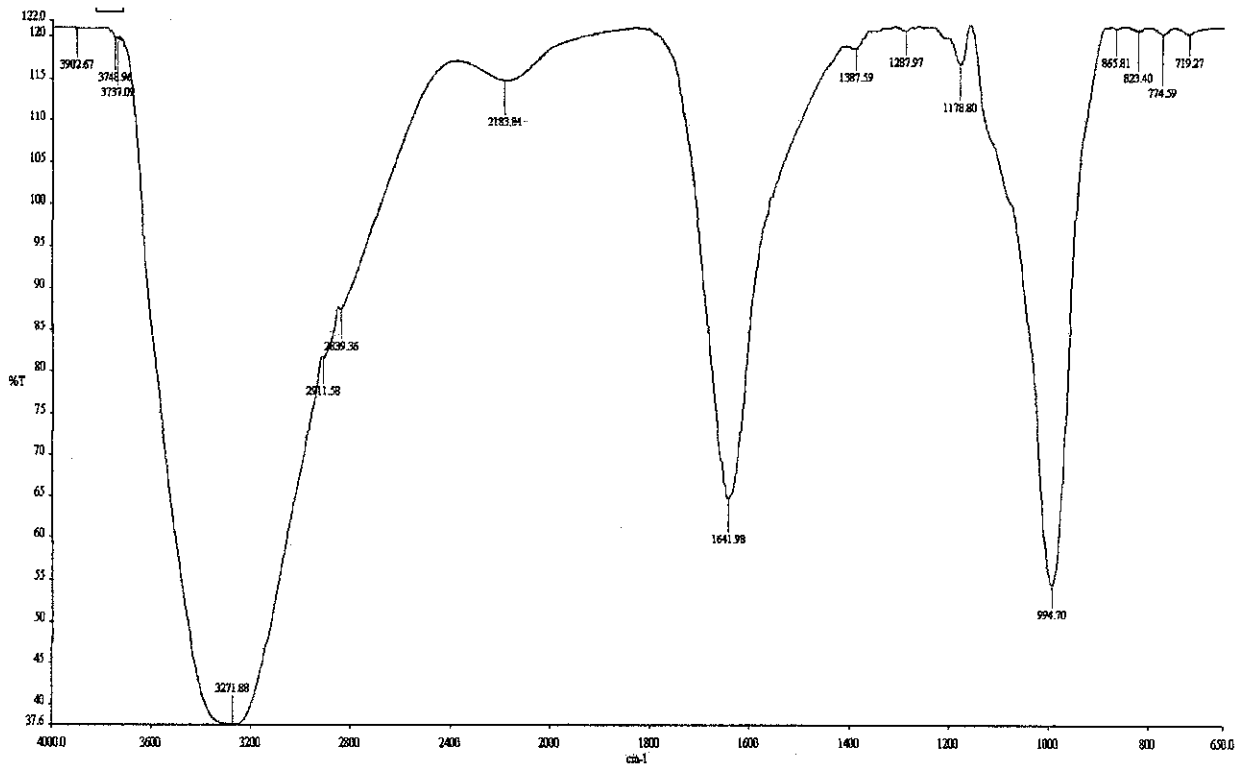
**Figure 16: Transmission vs. absorption frequency graph from FTIR analysis of geopolymer with concentration of 1M before adding amylase enzyme**

## 1M Sodium Hydroxide after adding amylase enzyme



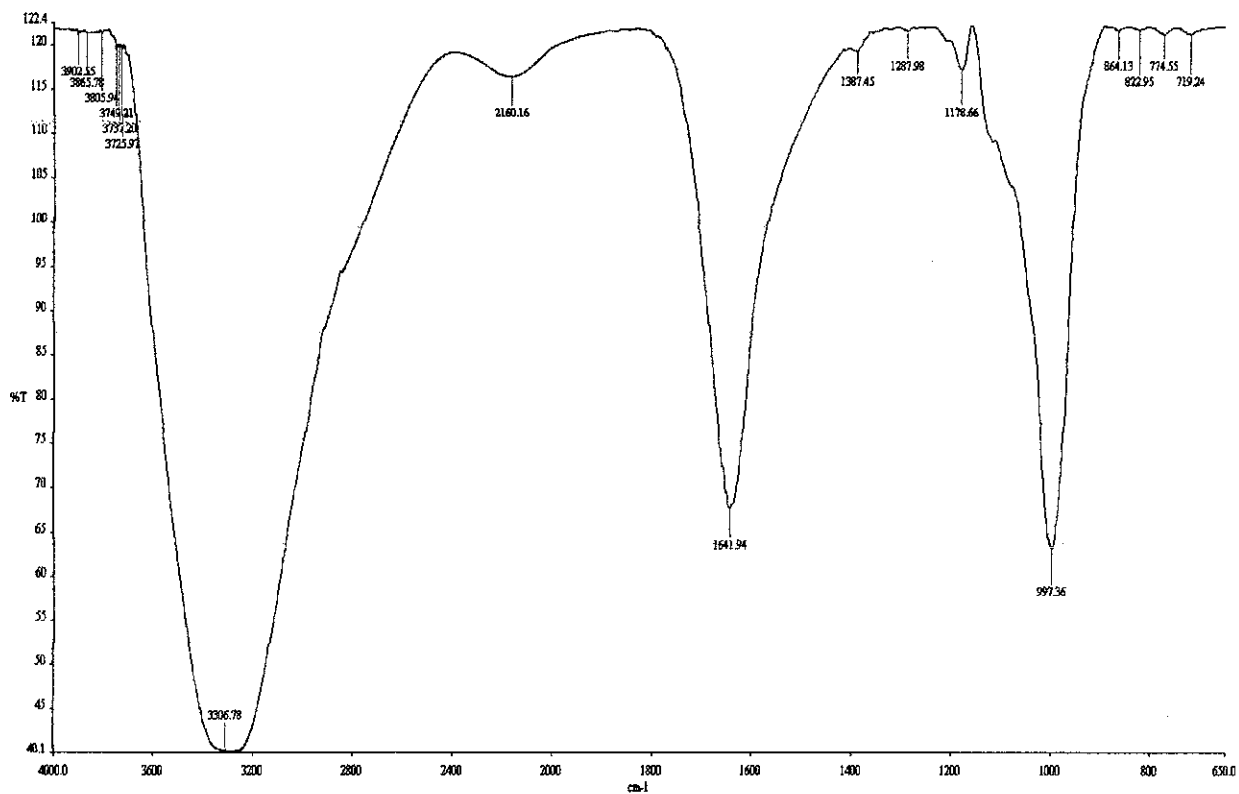
**Figure 17: Transmission vs. absorption frequency graph from FTIR analysis of geopolymer with concentration of 1M after adding amylase enzyme**

## 2M Sodium Hydroxide before adding amylase enzyme



**Figure 18: Transmission vs. absorption frequency graph from FTIR analysis of geopolymer with concentration of 2M before adding amylase enzyme**

## 2M Sodium Hydroxide after adding amylase enzyme

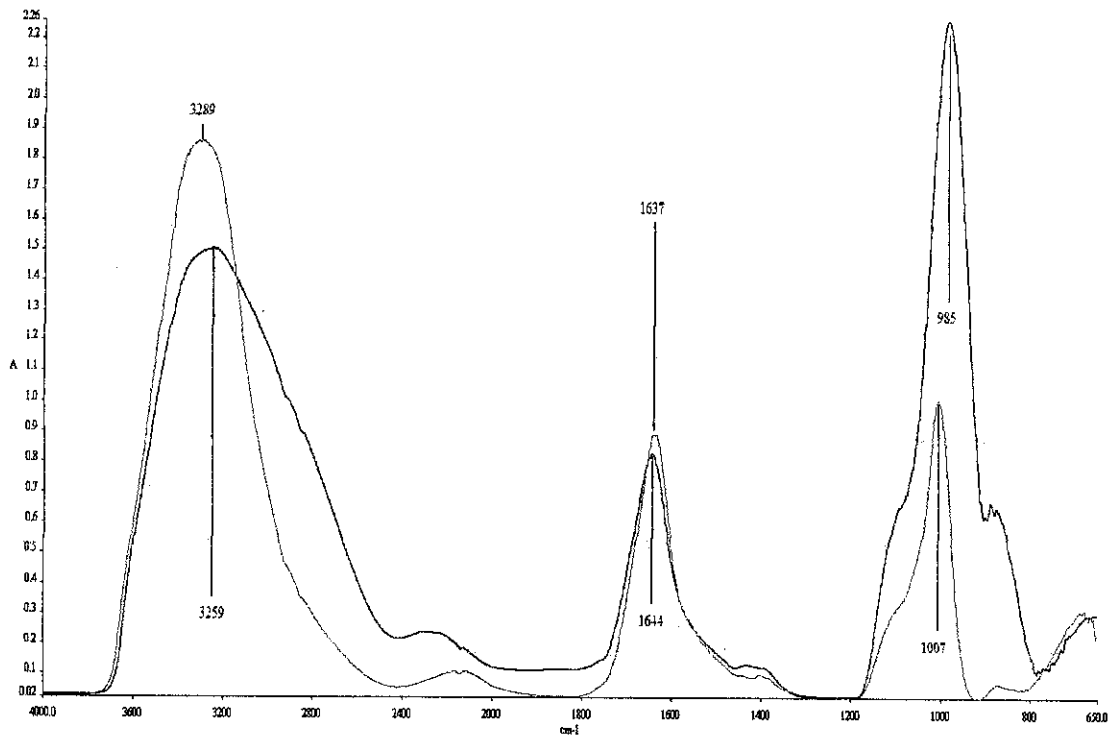


**Figure 19: Transmission vs. absorption frequency graph from FTIR analysis of geopolymer with concentration of 2M after adding amylase enzyme**



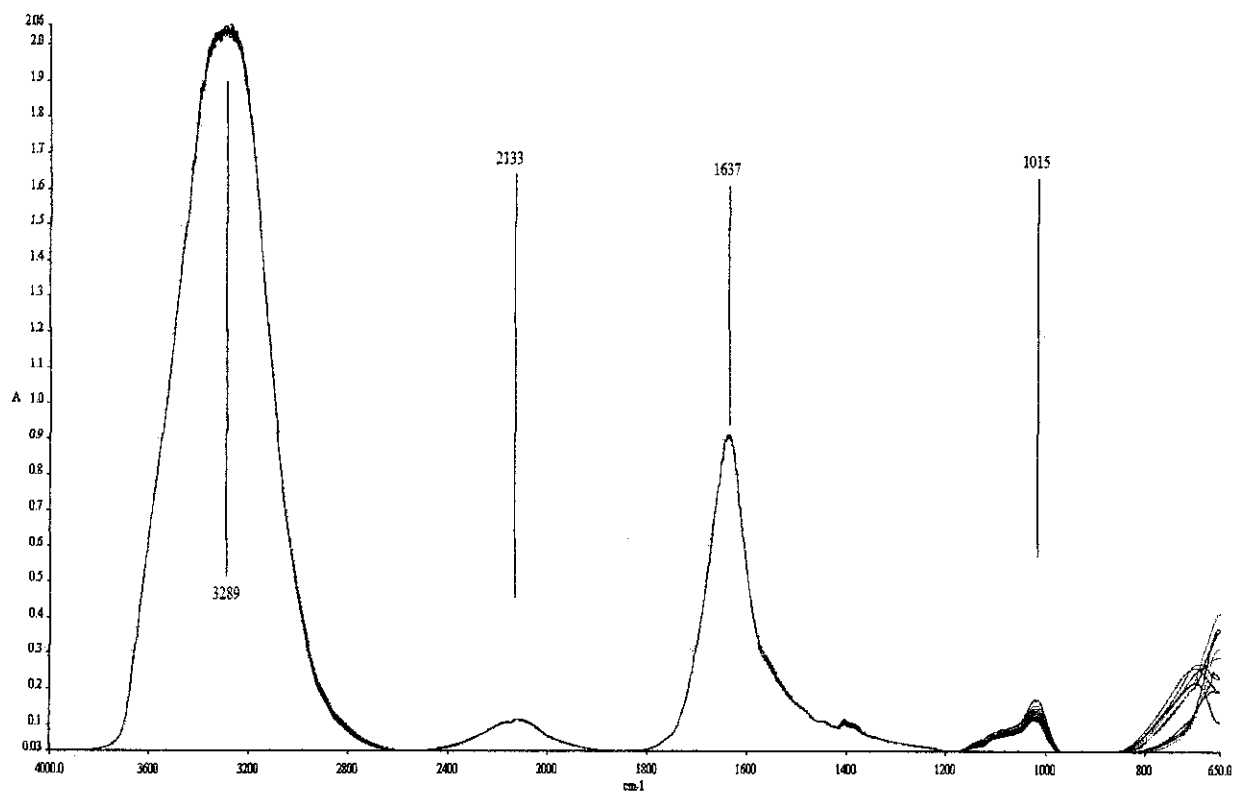
### 4.1.1.2 Geopolymer Degradation Study With Difference Ph of Buffer Solution

#### PH 4 Before and After Adding Buffer Solution



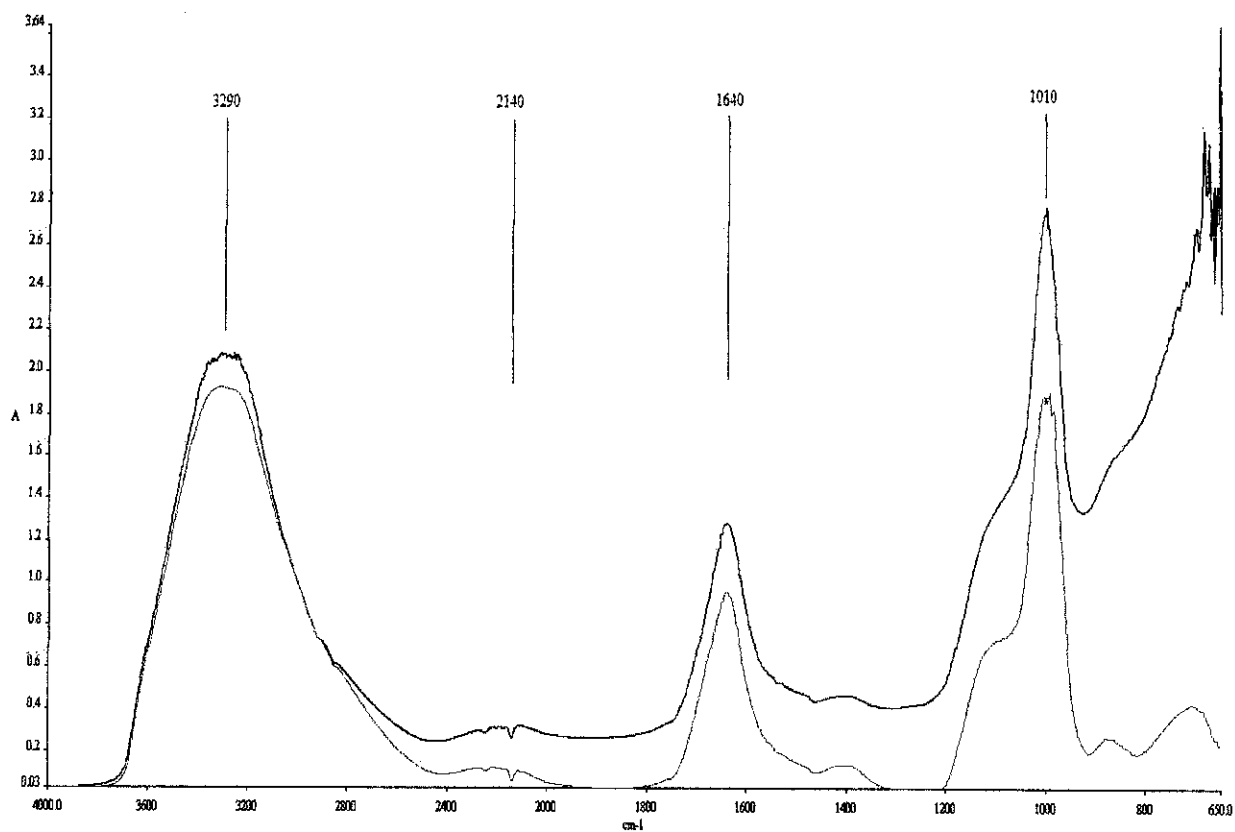
**Figure 20: Infrared spectrum of the geopolymer film on the FTIR without buffer (black line), and the same geopolymer film hydrated with buffer solution pH 4.0 (blue line).**

## PH 4 After Adding Amylase Enzyme



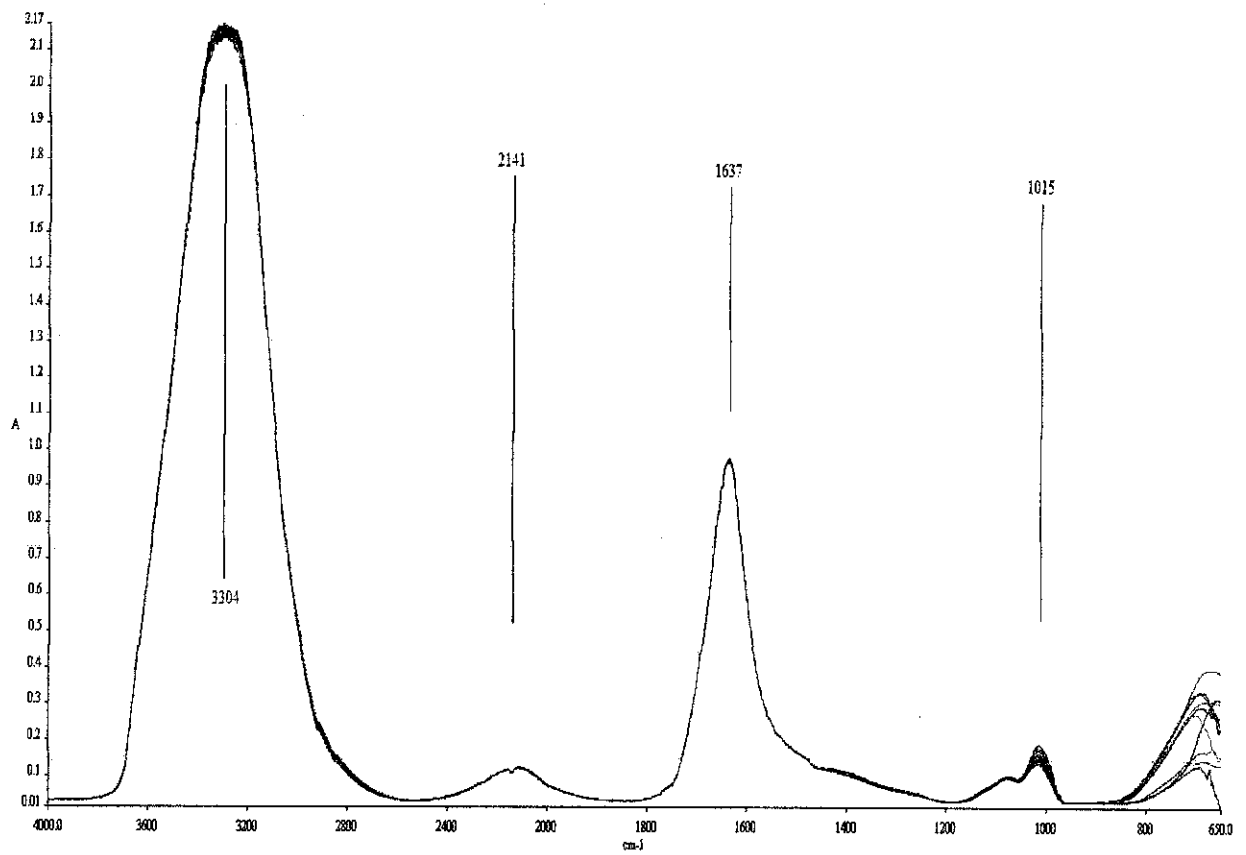
**Figure 21: FTIR difference spectra recorded 1-10 min after addition of amylase enzyme with  $\Delta t = 1$  min.**

## PH 7 Before and After Adding Buffer Solution



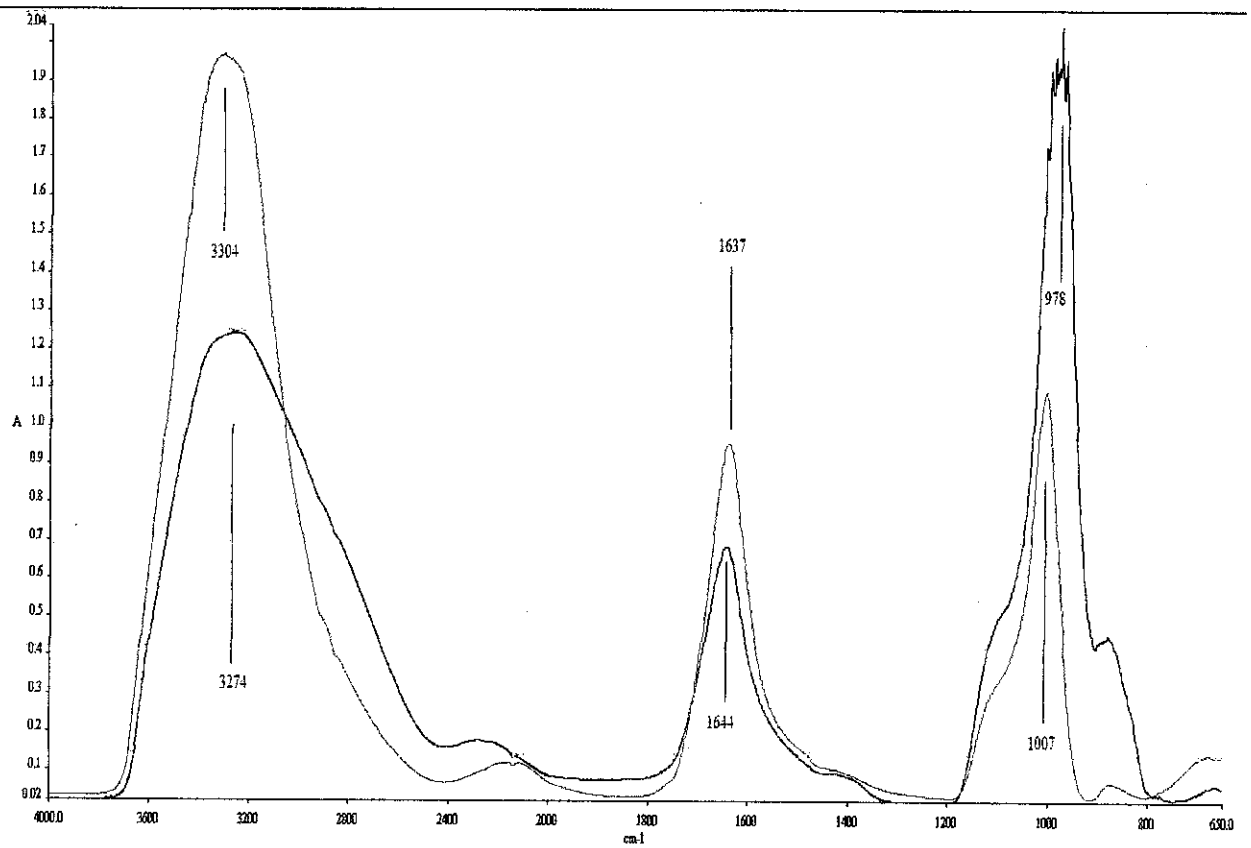
**Figure 22: FTIR spectrum of the geopolymer film before adding buffer (blue line) and after adding the buffer (black line) solution at pH 7.0.**

## PH 7 After Adding Amylase Enzyme



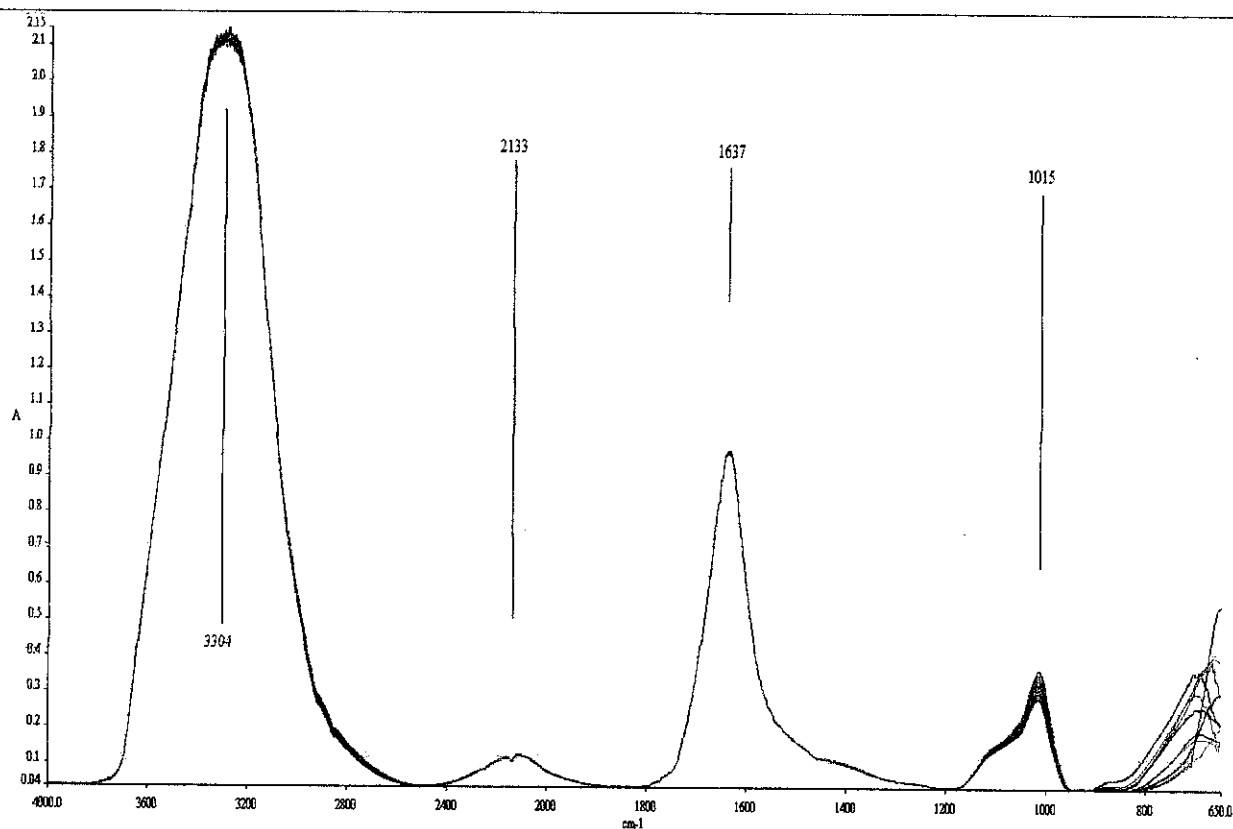
**Figure 23: FTIR difference spectra recorded 1-10 min after addition of amylase enzyme with  $\Delta t = 1$  min.**

## PH 10 Before and After Adding Buffer Solution



**Figure 24: FTIR spectrum of the geopolymer film before adding buffer (black line) and after adding the buffer (blue line) solution at pH 7.0**

## PH 10 After Adding Amylase Enzyme



**Figure 25: FTIR difference spectra recorded 1-10 min after addition of amylase enzyme with  $\Delta t = 1$  min.**

## 4.2 DISCUSSION

### 4.2.1 Geopolymer Degradation Study With Different Concentration of Sodium Hydroxide

Infrared is probably the most appropriate technique for studying the bonds degradation of rice husk ash geopolymer as it is sensitive to structures with short range structural orders. From the spectrum produced from the FTIR, we are able to analyze the wavelength to identify the functional groups in the unknown sample.

The Infrared spectra of all samples showed the presence of typical stretching and deformation vibrations of the O - H bond of water, centered at 3307, 3295, 3271, and 3306  $\text{cm}^{-1}$ . The other functional group that presence in the sample is the C = C bonds at 1641, and 1642. The sharp and strong peak centered at 1000, 994 and 997 in rice husk ash sample indicates that under high alkaline environment the  $\text{SiO}_4$  tetrahedra in rice husk ashes were dissolved and some  $\text{Na}^+$  was introduced into -Si-O<sup>-</sup> network (Hanna 1964). When Al was introduced into above reaction system on the hand, some of the Si+4 are isomorphously replaced by  $\text{Al}^{+3}$  ions, resulting in  $\text{AlO}_4$ -tetrahedra. The absorption frequencies associated with Si-O-Si linkages would be expected to shift lower wave numbers, since the Al-O bond vibrates at a lower frequency than the Si-O-Si bond (Kolesova, 1990).

From the FTIR spectra of rice husk ash geopolymer with concentration of 1 molar before and after adding the enzyme, there is a slight different between the pattern of the graph and also the value of specific peaks. The specific peaks shifted from 3307 to 3295 where the broad and strong OH stretch change to very broad signal.

As for the 2 molar concentration of rice husk ash geopolymer, the FTIR spectra for before and after adding the enzyme is quite the same with 1 molar concentration, where a very broad signal to broad strong absorption of OH with a slight shift of the peaks from 3271 to 3306. This shows that for both 1 molar and

2 molar concentration do not really effect the degradation of geopolymer. After the addition of amylase enzyme, for both 1 and 2 molar concentration of geopolymer solution, there are slight changes in the specific peaks that show the geopolymer is being degraded, but not fully degrade. In order for the geopolymer to fully degrade, some modification needs to be made such as change the concentration of enzyme and also the concentration of sodium hydroxide. The concentration of sodium hydroxide can be made lower than 1 molar while the concentration of amylase enzyme should be increase in order for the rice husk ash geopolymer to degrade.



#### 4.2.1 Geopolymer Degradation Study With Different PH

For pH 4, the FTIR spectra of the rice husk ash geopolymer film showed that the major geopolymer specific peaks were at 3259, 1644, and 985  $\text{cm}^{-1}$ . After the addition of buffer solution, the specific peaks of geopolymer shift to 3289, 1637, and 1007. The peak at 1637 for after the addition of buffer is assigned to  $\text{H}_2\text{O}$  showed an increasing tendency, indicating water penetration into the film as the buffer solution is added onto the geopolymer film. After the addition of  $\alpha$ -amylase enzyme, where the difference spectra is recorded every minute for 10 min, showed that the geopolymer specific peaks at 3289, 2133, 1637, and 1015. The specific peak centered at 2590 is decreased after addition of amylase enzyme. The three specific peaks at 3289, 1637, and 1007  $\text{cm}^{-1}$  constitute a unique pattern for the geopolymer film.

For the second experiment, where buffer solution with pH 7 is used, there are no changes in the specific peaks of geopolymer before and after the addition of buffer solution. This may due to the fact that pH 7 is neutral and it did not affect the specific peaks of geopolymer. After addition of enzyme, the specific peaks change from 3290, 2140, 1640, and 1010 to 3304, 2141, 1037, and 1015. Very broad signal of OH band centered at 3290 shifted to 3304 which is broad and strong absorption.

As for the pH 10, before adding the buffer solution, the specific peaks centered at 3274, 1644, and 978. These specific peaks shifted to 3304, 1637, and 1007 after addition of buffer solution. This is due to the water that may enter the film after addition of buffer solution, thus diluting the geopolymer concentration in the film. After addition of amylase, the geopolymer specific peak that centered at 1015 is decreased slightly over the time.

For all the pH, after the addition of amylase enzyme, the specific peak that centered at 1015 showed a slight decrease over the time. The specific peak at

1015 indicates the strong absorption. The decreasing of specific peak showed that the geopolymer is slightly degraded. As the time is increase, the geopolymer degradation rate also increases, although just a slight degradation that occurred. In order for the geopolymer to fully degrade, it may take more time. The concentration of amylase enzyme can be modified so that it will be suitable for the degradation of geopolymer.

From all the graphs for the pH effect towards degradation, it can be seen that pH 10 shows a greater decreasing of specific peak at 1015 as compared to pH 4 and pH 7. This show that pH 10 is the most suitable pH for the degradation of rice husk ash geopolymer. Although the geopolymer do not fully degrade, some modification can be made so that it can fully degrade in order for it to be used as urea coating material.

## **CHAPTER 5**

### **CONCLUSION & RECOMMENDATIONS**

Geopolymer is recently being developed in the application of control release fertilizer (CRF). It is proven that geopolymer is eco-friendly as it gives less impact to the environment. Also it is manufactured through sustainable production processes. The production of geopolymer also at low cost as the main material to produce the geopolymer, which is rice husk can be easily found in Malaysia at low cost. From the experiment, it is found that the geopolymer can degrade as the geopolymer is made from rice husk ash, which is inorganic and degradable material. The factor that mainly affects the degradation of geopolymer is pH. The effect of pH towards rice husk ash geopolymer can be test by using FTIR with the addition of enzyme onto the geopolymer film. In order for the geopolymer to fully degradade, some modifications need to be made such as the concentration of amylase enzyme and sodium hydroxide. Amylasē enzyme also can be replaced with better enzyme, not using human saliva. Development of CRFs need to be study and improved in order to produce better CRFs for agriculture usage.

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