SOLID CATALYST FROM CHICKEN BONE FOR TRANSESTERIFICATION

By

SARIYAH PUTEH

Progress report submitted in partial fulfillment of

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Bachelor of Engineering (Hons)

(Chemical Engineering)

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
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Universiti Teknologi PETRONAS
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Approved by,

____
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Project Supervisor

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
SEPTEMBER 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.

SARIYAH PUTEH

ABSTRACT

Biodiesel is produced by the transesterification of triglycerides with an alcohol in the presence of a homogeneous or heterogeneous catalyst. The reaction results in the production of ester group called biodiesel and glycerol as a byproduct. In this research project, study the feasibility of heterogeneous catalysts prepared from chicken bone. Using the calcination method to convert dried chicken bone into active catalyst at different temperature: 800˚C, 900˚C and 1000˚C. Catalyst characterized by various methods: X-Ray Diffraction (XRD), Field-emission scanning electron microscopy (FESEM), Energy Dispersive X-ray (EDX) and Fourier transform infrared spectroscopy (FTIR) analysis. Lastly, gas chromatographic instrument used to investigate the catalitic performance in the reaction.

ACKNOWLEDGEMENTS

In the Name of Allah, The Most Merciful and Compassionate, praise to Allah, He is the Almighty. Eternal blessings and peace upon the Glory of the Universe, our Beloved Prophet Muhammad (S.A.W), his family and companions.

This Final Year Project: “Solid Catalyst from Chicken Bone for Transesterification” has involves many parties in order to bring it successfully. It is a golden opportunity to learn, practice and apply engineering project throughout the study.

I would like to take this opportunity to express my sincere thanks and appreciation to the following persons and organizations who have directly or indirectly given generous contributions towards the success of this project. Firstly, special appreciated gratitude shall go to the project’s supervisor, Dr. Norhayati Bt. Mellon, for her tireless efforts and ongoing support as well as advice and encouragement throughout this project. Deepest gratitude shall be given to Dr. Anita Ramli, co-supervisor, for her willing helps, who is another important person that fully support me to complete this project. This project would not be able to complete in time without them. Yet not to be forgotten to Mr. Farooq Khattak, a PhD student who is Dr. Anita assistance, for the guidance, assistance and always support. Moreover, thank you to all technicians for the support as well as all people who helped in gaining wonderful experience along this final year project in completing the job assignment. All the help and support is highly appreciated in which has enabled well performance of the project.

Lastly, I hope that this project gives the readers some insight as to the maze of activities associated with biodiesel industrial improvement.

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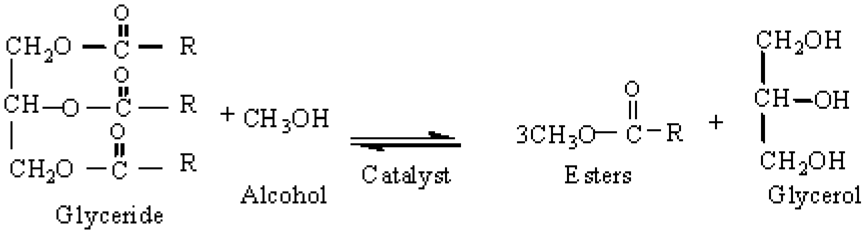
CHAPTER 1: INTRODUCTION

1.1 BACKGROUND

Biodiesel is a clean energy as an alternative diesel fuel. Nowadays it becomes very famous fuel due to clean burning, biodegradable, nontoxic and essentially free of sulfur and aromatics (Mueanmas et al., 2010) whereas the increasing worldwide concern for the conservation of nonrenewable natural resources and the environmental protection. It is necessary to develop an alternatives fuel due to the volume of petroleum discovered worldwide is decreasing with a higher demand by approximately 2% each year (Gunnar L., 2003) and the trend was identified that petroleum resources tend to be shortage in the next few years (Besti S., 2010). The attractiveness of biodiesel is its physical and chemical properties are similar to petrodiesel, allowing used either mixed with petrodiesel or directly use in any diesel engine without requiring any modifications moreover, biodiesel properties are able to enhance engine yield and extend engine life (P.T. Vasudevan & M. Briggs, 2008).

The process used to convert fatty acid (triglyceride) and primary alcohol in the presence of catalyst to produce alkyl esters (biodiesel) and glycerol as called transesterification reaction. Furthermore, both transesterification and esterification reaction are the primary routes of biodiesel production. The methods differ in the catalyst and oil source used.



Source: http://www.esru.strath.ac.uk/EandE/Web_sites/02-03/biofuels/what_biodiesel.htm

Figure 1: Transesterification of Triglyceride

Transesterification is general term used to describe the importance of biodisiel formation where the triglyceride is reacted with the alcohol group as shown in Figure 1. The alcohol reacts with the fatty acids to form the mono-alky ester or biodiesel and glycerol. Most of biodiesel industries used methanol and ethanol but H.D. Hanh et al. (2009) has proved that methanol is the best alcohol for transesterification process due to lower price and providing very high ester conversion because of having a shorter chain of alcohol group, forming less number of carbon, which is easily separate the ester group.

The presence of catalyst accelerates considerably the adjustment of the equilibrium in order to achieve high yield of the ester (R. Sercheli et al., 1997). In industry, the using of catalyst is either acid or base else new technology method is developed the utilization of enzymes as a catalyst rather than acid or base catalyst but it is not economic. Both homogenous catalyst and heterogeneous catalyst can be used in transesterification reaction. The common type employed in indusrty is homogeneous catalyst due to most economical concern (Singh V. et al, 1991). Vidya S. et al., (2006) conclude that alkali catalysts (base catalyst) such as NaOH, CH3ONa, and KOH give very high conversion. However, heterogenous catalyst has more attracting attention with its advantage due to high activity, withstand in high temperature and requires neither catalyst recovery nor aqueous treatment (A. Refaat, 2010).

1.2 PROBLEM STATEMENT

In industry, homogenous catalyst is widely used. It gave low grade of production due to difficult to separate the product with catalyst and also caused potentially environmentally hazardous waste is regenerated (Y.C. Sharma & B. Singh, 2009). Many researcher attampt to overcome this problem by replacing homogenous catalyst to heterogeneous catalyst. Heterogeneous catalyst has easier simplifying product of biodiesel and reusability which acts as medium of reaction (Karen W. & Adam F. L., 2012). Several types heterogeneous catalysts have been employed in the biodiesel production, for example alkali metal salt, MgO, CaO, and hydrotalcites (N. Viriya-empikul et al, 2010) but it is more expensive if compare with homogeneous catalyst (Singh V. et al). For this project, the author interested in extracting chicken bone and convert into catalyst which suspected to contain CaO as its main component. In malaysia as well as UTP, it is found that most of malay dishes in daily meals use chicken as the main ingredient such as chicken rice, chicken curry, chicken tandoori and fried chicken. Using waste of chicken bone as a raw material for catalyst synthesis is such an attracted alternative resource for low-cost biodiesel production catalyst.

1.3 OBJECTIVE

The aim of this study is to conduct the feasibility study of converting chicken bone as a catalyst for biodiesel production.

1.4 SCOPE OF STUDY

- The bone derives throughout calcination method in different temperature of thermal treatment.
- The characteristics of the catalysts have been performed throughout X-Ray Diffraction (XRD), Field-emission scanning electron microscopy (FESEM), Energy Dispersive X-ray (EDX) and Fourier transform infrared spectroscopy (FTIR) analysis.
- The characteristic of catalyst activities have investigated in transesterification reaction of triglyceride and methanol and analyze by Gas Chromatography-Mass Spectroscopy (GC-MS) tool.

CHAPTER 2: LITERATURE REVIEW

The usage of vegetable oil as fuel dates back to a century ago when the diesel engine was first invented by Rudolph Diesel. In the earlier years of the diesel engine, vegetable oil was directly used as fuel. However, extensive engine testing proved that it was not suitable fuel for the engine due to several issues encountered. By year 1920, petroleum industry had developed petrodiesel in more suitable quality the engine and cheaper (Manzanera et al., 2008). One solution to poor fuel property of vegetable oil was to convert it into monoalkyl esters, biodiesel, throughout the process of transesterification and resulted very similar to petrodiesel properties (Knothe G., 2010). The development of biodiesel has been increased since the shortage of petrodiesel keeps increasing due to the limited resources and the market price would be higher globally. Thus, currently biodiesel considered as one of the best alternatives as nowadays has lower price than petrodiesel and environmental friendly is the most attractive. The process of biodiesel synthesis is much simpler and easier to extract the raw material which usually extracted from vegetable oils or animal fat. The process of Transesterification is produced under the presence of catalyst, traditionally used homogeneous alkali catalyst. However, the homogeneous catalyst suffers from the expensive downstream separation and the toxicity waste produced. In recent years, the development of heterogeneous catalyst has begun and achieved high yields while reducing production cost. In the following sections, a comprehensive review of biodiesel development is presented.

2.1 BIODIESEL PRODUCTION

Biodiesel is defined by ASTM International as a fuel composed of monoalkyl esters of long-chain fatty acids derived from renewable vegetable oils or animal fats meeting the requirements of ASTM D6751. Biodiesel is biodegradable, environmentally friendly and produced lower emissions. Moreover, biodiesel can reduce unburned emission that cause global warming as a recent world concerned.

2.1.1 Biodiesel Properties and Applications

The physical properties of biodiesel are very similar to petrodiesel fuel. Usage of biodiesel in a conventional diesel engine substantially reduce emissions of unburned hydrocarbons, carbon monoxide, sulfates, polycyclic aromatic hydrocarbons, nitrated polycyclic aromtic hydrocarbons, and particulate matter (Mahfusah M, 2009). In fact the diesel engine was originally designed to run on vegetable oil rather than fossil fuel.

Biodiesel can be used in any diesel engine by directly use or mixing with petrodiesel. In some manufacturers cover their diesel engines under warranty for 100% biodiesel use. However, the majority of vehicle manufacturers' limit their recommendations are 15% of biodiesel blended with petrodiesel. In many European countries use 5% biodiesel blend called B5 that is widely used and available at thousands stations. Blending is possible to contain biodiesel up to 20% biodiesel called B20 can be used in all diesel powered equipment. For pure biodiesel, B100 can be used in many engines with little or no modification. It is compatible with most storage and distribution equipment, but special handling is required.

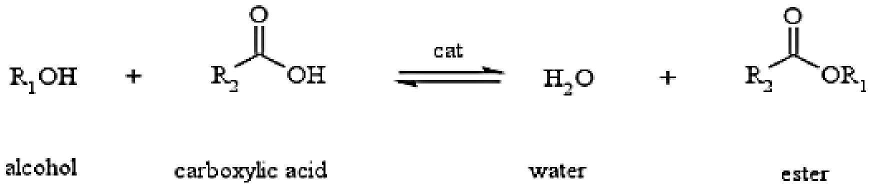
2.1.2 Biodiesel Emission

Biodiesel is the first and only alternative fuel to have a complete evaluation of emission results and potential health effects submitted to the U.S. Environmental Protection Agency (EPA) under the Clean Air Act Section 211(b).

Biodiesel reduces the health risks associated with petroleum diesel. One major benefit of biodiesel is lower emissions. The use of biodiesel reduces emission of carbon monoxide and other hydrocarbons by 20 to 40%. Biodiesel burns up to 75% cleaner than conventional petroleum diesel fuel. In Health Effects testing, polycyclic aromatic hydrocarbons (PAH) compounds were reduced by 75 to 85 percent, with the exception of benzoanthracene, which was reduced by roughly 50 percent. Biodiesel emission impact depens on the source of fatty and amount of blending.

2.2 CHEMICAL REACTION OF BIODESIEL

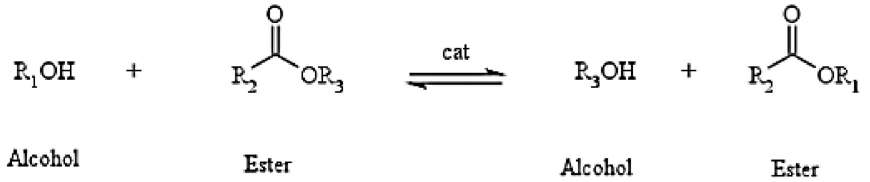
There are a number of chemistry terms that used to describe the process of biodiesel. Esterification and tranesterification reaction are known as primary route of production. Esterification is the general name for a chemical reaction in which two reactants form an ester as the reaction product.



Source: Nourredine A., 2010. *Sulfate and Hydroxide Supported on Zirconium Oxide Catalysts for Biodiesel Production,* Blacksburg, Virginia

Figure 2: General Term of Esterification Reaction

Transesterification is the process of exchanging the alkoxy group of an ester compound with another alcohol. These reactions are often catalyzed by the addition of an acid or base.

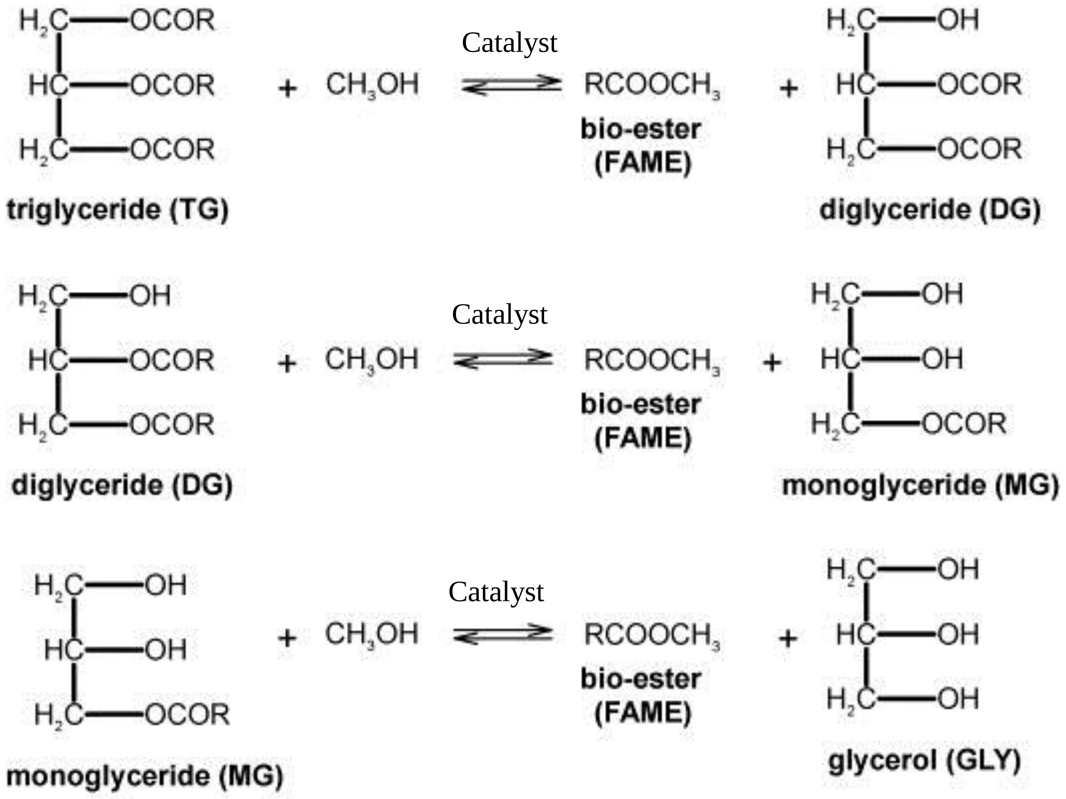


Source: Nourredine A., 2010. *Sulfate and Hydroxide Supported on Zirconium Oxide Catalysts for Biodiesel Production,* Blacksburg, Virginia

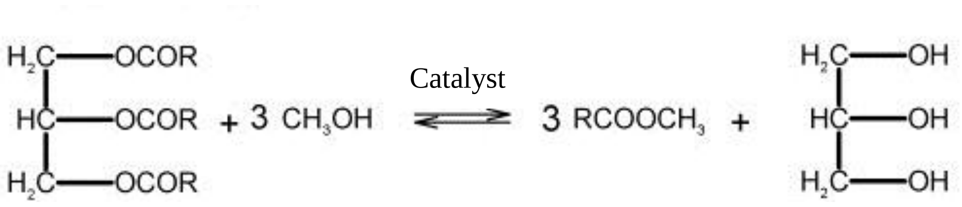
Figure 3: General Term of Transesterification Reaction

The methods differ in oil source and catalyst used. Transesterification is catalyzed by base or acid catalysts but esterification, however, is only catalyzed by acid catalysts. In this patent, the author described the kinetics transesterification of triolein converted to methyl oleate.

Generally, the reaction is reversible since triolein is highly non-polar and alcohol is polar, the organic solvent or catalyst is usually used to make reaction occur in irreversible. The following is a model to explain the kinetics of triglyceride tranesterification reaction:



Overall reaction:



Triglyceride + Methanol $\overset{\text{Catalyst}}{\Longleftrightarrow}$ methyl acetate + glycerol

Source: A. Zieba et al., 2010. "Transesterification of triglycerides in the presence of Ag-doped $H_3PW_{12}O_{40}$," *Science Direct*, *General* **316**: 30-44.

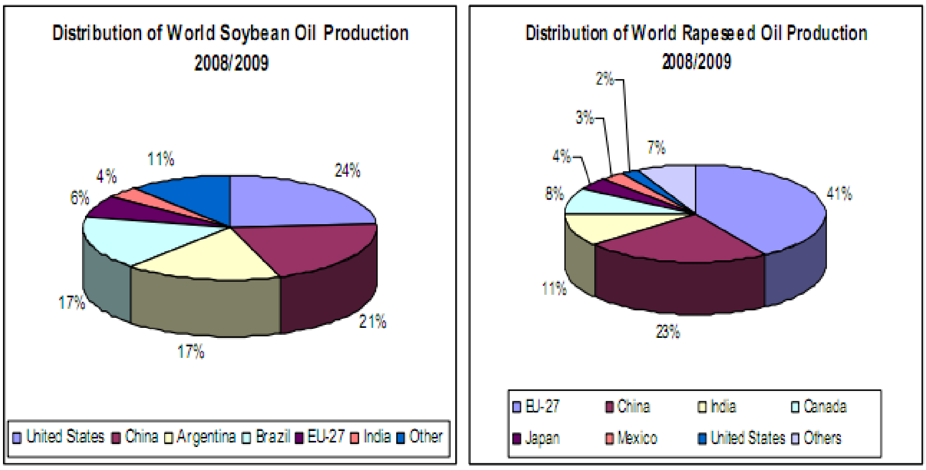
Figure 4: The Kinetics of Transesterification reaction of Triolein

The transesterification is an equilibrium reaction and the transformation occurs essentially by mixing the reactants (C. Mueanmas et al., 2010). Triglyceride react with methanol under presenting of catalyst produce bioester and dioglyceride. Dioglyceride react with methanol produce also bioester and monoglyceride. Under the same condition, monoglyceride react with methanol, it produce again bioester and glycerol.

The overall process is a sequencing of three consecutive and reversible reactions, in which diglycerides and monoglycerides are formed as intermediates (Freedman, B. et al., 1986). The stoichiometric reaction requires a mole of a triglyceride and three moles of the alcohol. However, in three steps of mechanism reaction is all required alcohol group. Thus, the used of alcohol in reaction should be excess in order to increase the yields of the alkyl esters and to allow its phase separation from the glycerol formed (R. Sercheli et al., 1997).

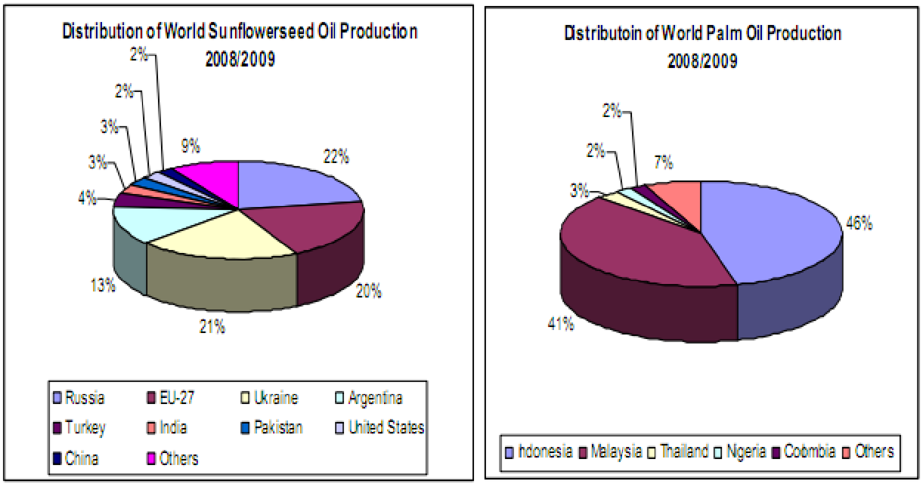
2.3 FEED STOCK

Alkyl fatty acid esters can be prepared from any fatty acid sources and as such biodiesel has been prepared from various feedstocks. The most popular type of feedstock for biodiesel production are edible refined vegetable oils such as soybean, rapeseed, sunflower, palm, coconut and linseed (Singh and Singh, 2010). However, the choice of feedstock is dictated by the availability of oil crops in a particular region (Figure 5 and Figure 6).



Source: Nourredine A., 2010. *Sulfate and Hydroxide Supported on Zirconium Oxide Catalysts for Biodiesel Production*, Blacksburg, Virginia

Figure 5: World production of soybean and rapeseed oil. Data retrieved from SDA Foreign Agricultural Services PSD Online database (USDA, 2010)



Source: Nourredine A., 2010. *Sulfate and Hydroxide Supported on Zirconium Oxide Catalysts for Biodiesel Production*, Blacksburg, Virginia

Figure 6: World production of sunflowerseed oil and palm oil. Data retrieved from USDA Foreign Agricultural Services PSD Online database (USDA, 2010).

Feedstock selection is a crucial aspect to the success of biodiesel manufacturing. According to Figure 6, Malaysia is second biggest world palm oil distribution. Thus the selection of feedstock for this project is palm oil as easy to find and especially for Malaysia’s future delopment.

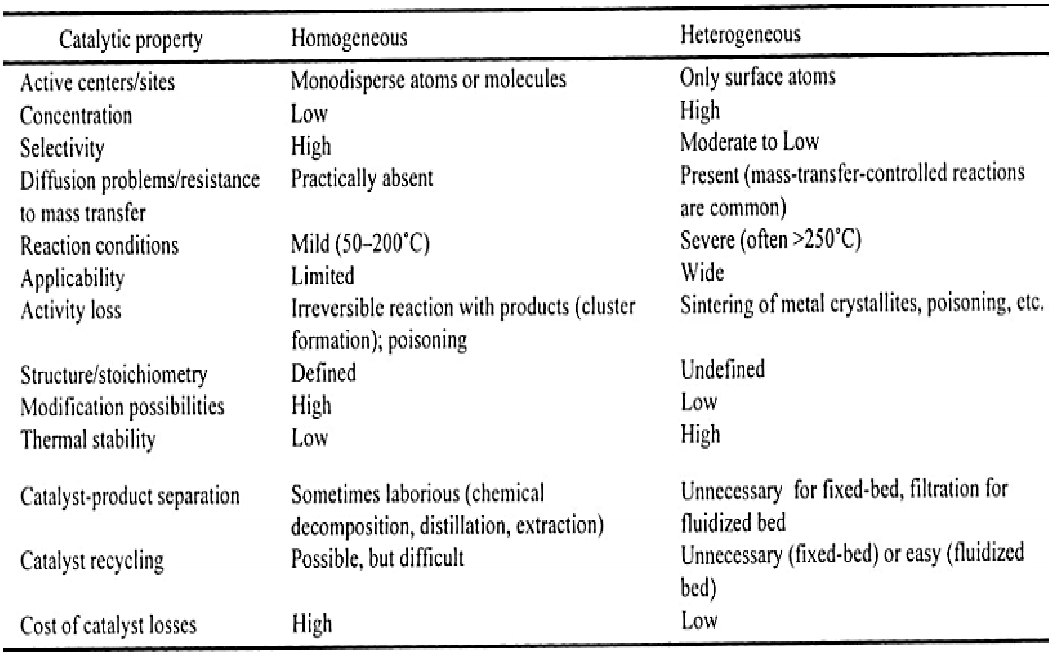
2.4 ALCOHOL FOR BIODIESEL

Alcohol is another one of main chemical reactant for biodiesel reaction that essentially effect to conversion of the ester group. The alcohol reacts with the fatty acids to form the mono-alky ester or biodiesel and glycerol. In most production, methanol and ethanol are commonly used in the industry but H.D. Hanh et al. (2009) have proved that methanol is the best alcohol for transesterification process because of giving very high ester conversion and lower price. Methanol is able to give higher conversion with the reason of shorter chain of alcohol group, forming less number of carbons, which can possibly break the ester group and form into biodiesel production easier.

2.5 CATALYST FOR BIODIESEL

The foundamental of using catalyst is chemical reaction as an accelerator. The presence of catalyst in biodiesel production become a major factor to the reaction. Commonly, catalyst used in biodiesel process is ether acid or base. It is widely used base catalyst to catalyze the reaction which is safer and lower cost if compared to acid catalyst. Most often used alkali metal. Moreover, new technology was developed to utilize enzyme as a catalyst and perform more effective and good for safety concerns.

Transesterification reaction can be catalyzed both homogenous and heterogeneous catalitic. The common type employed industrially is homogeneous catalyst due to lower cost. Lately, many researchers attempt to replace homogeneous catalyst by heterogenous catalyst (Nezahat B. & Miray K., 2009) in order to overcome the problem of separation product from catalyst that required an expensive equipment.

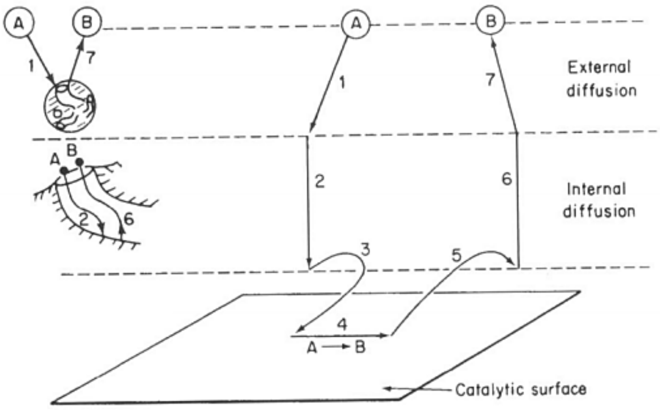


Catalytic property	Homogeneous	Heterogeneous
Active centers/sites	Monodisperse atoms or molecules	Only surface atoms
Concentration	Low	High
Selectivity	High	Moderate to Low
Diffusion problems/resistance to mass transfer	Practically absent	Present (mass-transfer-controlled reactions are common)
Reaction conditions	Mild (50–200°C)	Severe (often >250°C)
Applicability	Limited	Wide
Activity loss	Irreversible reaction with products (cluster formation); poisoning	Sintering of metal crystallites, poisoning, etc.
Structure/stoichiometry	Defined	Undefined
Modification possibilities	High	Low
Thermal stability	Low	High
Catalyst-product separation	Sometimes laborious (chemical decomposition, distillation, extraction)	Unnecessary for fixed-bed, filtration for fluidized bed
Catalyst recycling	Possible, but difficult	Unnecessary (fixed-bed) or easy (fluidized bed)
Cost of catalyst losses	High	Low

Source: C. H. Bertholomew et al.,2006. Fundamentals of Industrial Catalytic Processes 2nd ed.,Wiley Interscience

Table 1: Comparison of Homogeneous and Heterogeneous Catalyst

The main objective of using heterogenous catalyst is to simplify the product separation from catalyst and glycerol by-product (Karen W. & Adam F. L., 2012). It is also able to regenerate and avoid any neutralization step after the reaction and reduce soap formation. In the other hand it has very high sensitivity to CO_2, H_2O and O_2 (Vidya S.et al., 2006).



Source: H. S. Fogler, 2006. Elements of Chemical Reaction Engineering 4th ed., Pearson Education

Figure 7: Heterogenous Catalytic Activity

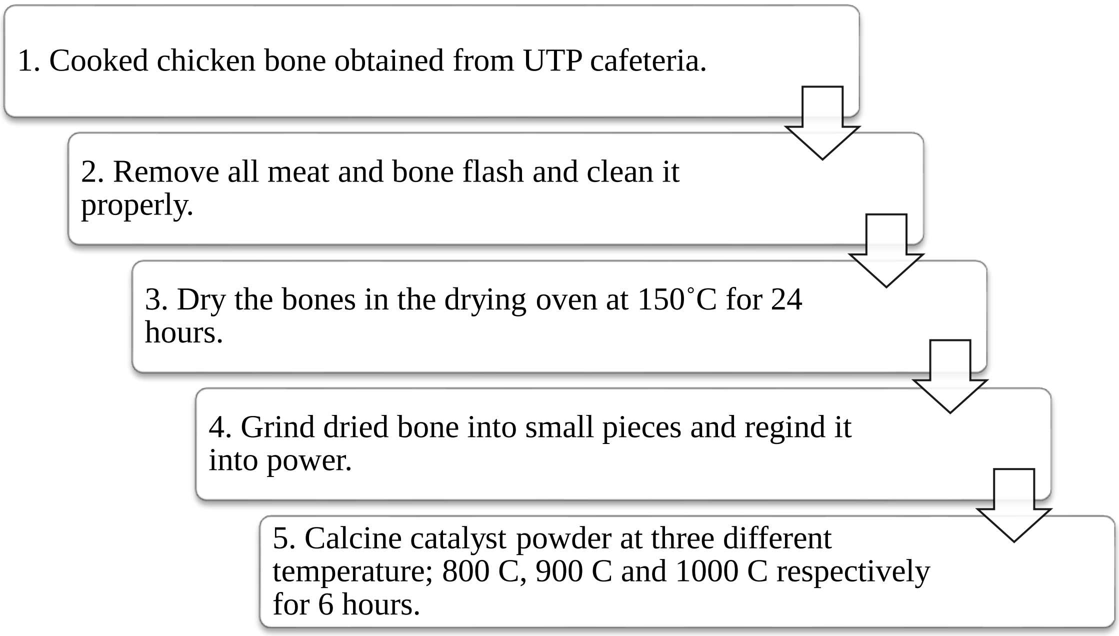
Figure 7 shows the activity of heterogeneous catalyst. The reaction occurs at the active site surface, catalytic surface. Smith G. V. and Notheisz F. (2006) conclude that the effeciency of heterogeneous catalyst depends on several factors such as specific surface area, pore size, pore volumn and active site concentration.

There are several types of heterogeneous base catalyst has been used for biodiesel production such as alkali metal salt, MgO, CaO, and hydrotalcites (N. Viriya-empikul et al, 2010). It is lack of toxicity and environmental friendly. $Ca(NO_3)_2$, $CaCO_3$, $CaPO_4$ and $Ca(OH)_2$ are raw materials to produce CaO (A. Obadiah et al., 2012). Beside, there are several natural source of calcium that can be extracted such as eggshell, mollusk shell, oyster shell, shrimp shell, mud crab shell and animal bone were successfully used for biodiesel production (R. Chakraborty et al, 2011). Animal bone known as the most contain of calcium, the author expected to found a mixture of calcium oxide in treated chicken.

CHAPTER 3: METHODOLOGY

This section presents the overview of the methods to be used in this feasibility study. Areas covered the method of catalyst preparation, catalyst characterization and catalyst testing.

3.1 CATALYST PREPARATION



1. Cooked chicken bone obtained from UTP cafeteria.

2. Remove all meat and bone flash and clean it properly.

3. Dry the bones in the drying oven at 150˚C for 24 hours.

4. Grind dried bone into small pieces and regind it into power.

5. Calcine catalyst powder at three different temperature; 800 C, 900 C and 1000 C respectively for 6 hours.

3.2 CATALYST CHARACTERIZATION

The calcined catalyst were characterized by varios analytical techniques, there are X-Ray Diffraction (XRD), Brunauer, Emmett and Teller (BET), Temperature programmed desorption (TPD), Fourier transform infrared spectroscopy (FTIR) and Field-emission scanning electron microscopy (FESEM).

3.3 CATALYST TESTING

Reactor type for catalyst testing is condensed reactor.

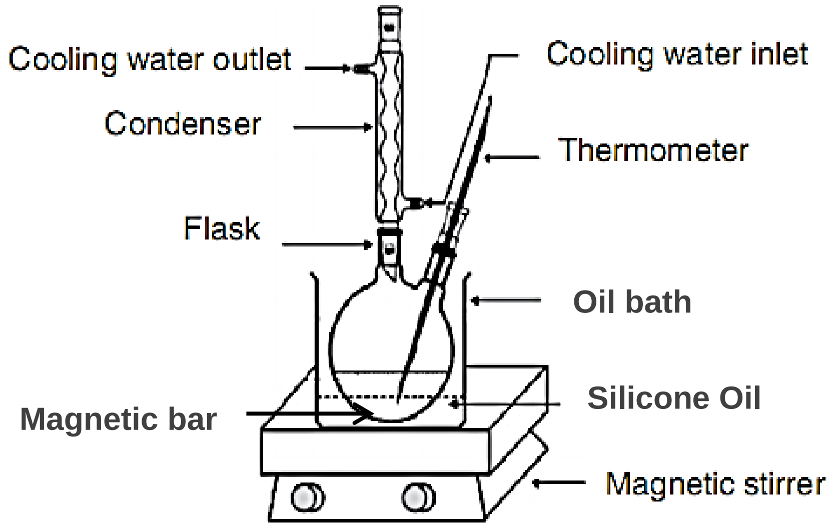


Figure 8: The experimental setup for the biodiesel production

<u>Activated Catalyst Step:</u>

- Use 2.5 g of 800˚C catalyst.
- Remove moisure of catalyst by drying at 100˚C for 1 hour.
- Do the same activated catalyst for 900˚C and 1000˚C.

<u>Reactor Conditions:</u>

- 65˚C and 1100 rpm of stirred rate.
- In order to keep temperature of reactor at 65˚C, using hot plate at 115˚C and put reactor in oil bath.

<u>Catalyst Testing Step:</u>

- Setup reactor as shown in Figure 9.
- Mix 800˚C activated catalyst with 40mL of methanol in reactor for 30 mins.

- Remove moisure of 40g cooking oil at 100˚C for 15 mins and mix with activated catalyst.
- Keep running experiment for 4 hous.
- First step of separation is separate catalyst by using folter paper.
- Second step is separation biodiesel and glycerol by funnel separator; top layer is biodiesel and bottom layer is glycerol.
- Purify biodiesel product by heating at 40˚C to remove methanol.

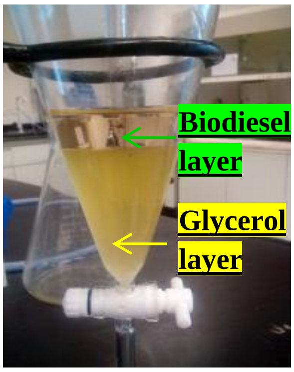


Figure 9: Biodiesel Separation

Biodiesel Product Analysis

According to D.Y.C. Leung & Y. Guo, 2006, the yield of the product can be calculated by using Eq. (1)

$$\%\text{Yield} = \frac{\text{weight of product (g)}}{\text{weight of raw oil (g)}} * 100 \qquad (1)$$

The methyl ester content in the product was analyzed by Gas Chromatography-Mass Spectroscopy instrument that used to monitor the reaction progress. The methyl ester peaks identified by comparing their retention times to that of authentic standards obtained from Restek (Restek, Bellefonte, PA).

3.4 PROJECT PLAN

No	Detail/Work	1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	First meeting with supervisor	■	■						Mid-semester break							
2	Preliminary Research Work		■	■	■	■										
3	Submission of Extended Proposal						◎									
4	Proposal Defense									■	■					
5	Project Research Continue											■	■	■		
6	Submission of Interim Draft Report														◎	
7	Submission of Interim Report															◎

Table 2: Gantt Chart for the First Semester Plan

No	Detail/Work	1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	Experimental Work	■	■	■	■	■	■	■	**Mid-semester break**							
	- Catalyst Preparation	■	■													
	- Experiment			■	■	■	■									
	- Analytical data							■		■						
2	Continuing Report										■	■	■	■		
3	Submission progress report										■					
4	Pre- EDX												■			
5	Submission of Draft Report													■		
6	Submission of Dissertation (soft bound)														■	
7	Submission of Technical Paper														■	
8	Oral Presentation															■
9	Submission of Dissertation (hard bound)															■

Table 3: Gantt Chart for the Second Semester Plan

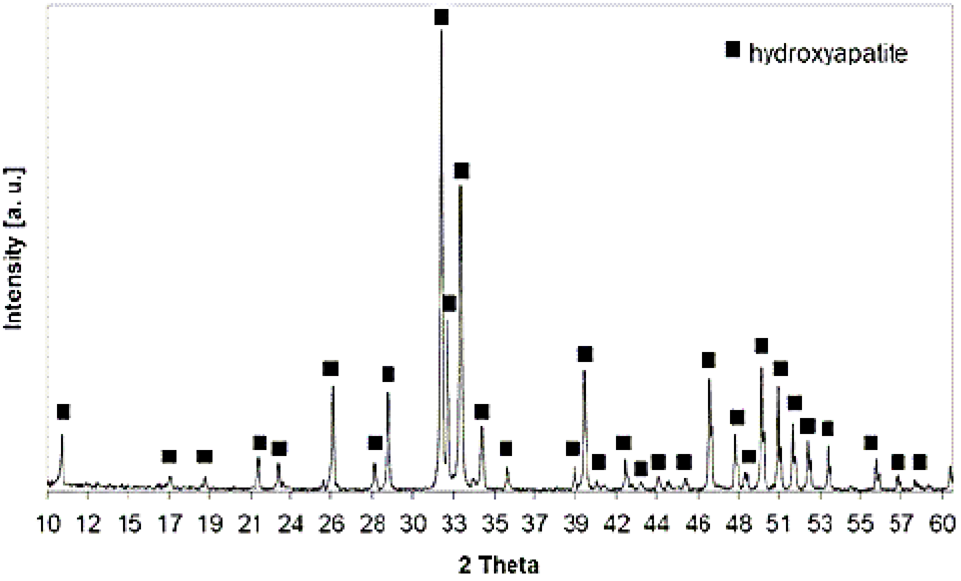
CHAPTER 4: RESULTS AND DISCUSSION

After calcination, we can clearly see that the color of bone catalyst change from light yellow brown in white color. For 1000˚C of calcination gives the most fine particle follow by 900˚C and 800˚C respectively. This is because the traces of organic matter, which are not present in the brighter sample obtained at higher temperature.

Figure 10: Uncalcined Catalyst (left) and Calcined Catalyst (right)

4.1 XRD (X-Ray Diffraction)

XRD method used to identify the chemical composition and crystallographic structure of natural and manufactured materials. The method characterized by sharply defined wavelength that closely similar to the spacing if the planes of standard mineral crystals.



Source: Agnieszka S. et. al, 2009. *Preparation of hydroxyapatite from animal bones,* Cracow University of Technology, Poland

Figure 11: XRD Pattern of Hydoxylapatite

Figure 12 is the XRD result. All three calcined catalyst shows similar XRD pattern. The results show that it is most similar to hydoxylapatite component (formula $Ca_5(PO_4)_3(OH)$ or usually write as $Ca_{10}(PO_4)_6(OH)_2$), natural of calcium apatite, usually found in teeth and bones. Higher calcination temperature behave clearer characteristic by overlapping line of lower calcination temperature and gives a higher peak.

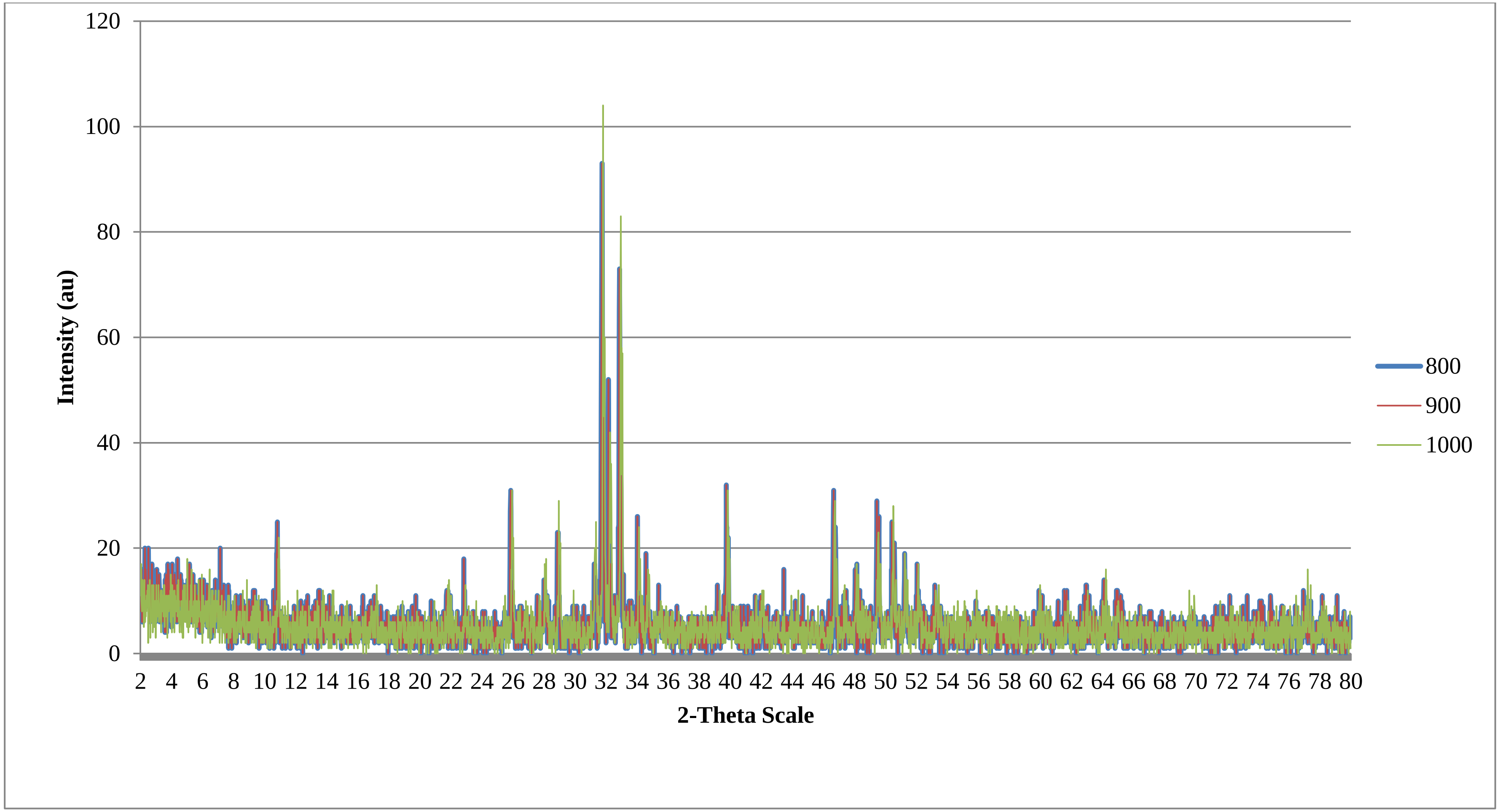


Figure 12: XRD Result Summary

4.2 FESEM (Field-emission scanning electron microscopy)

FESEM is another tool for characterization the catalyst by performing image of sample surface by raster scanning over it with a high-energy beam of electrons at length scales from millimeters up to 10 nanometers.

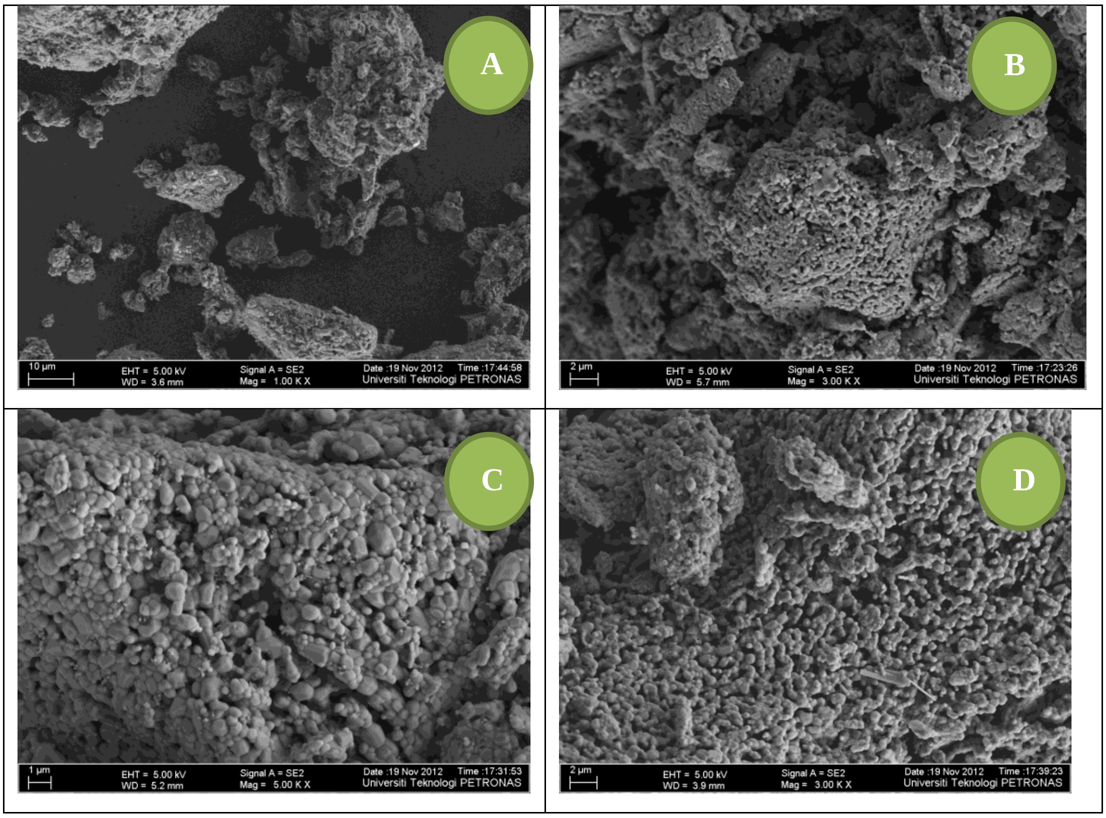


Figure 13: FESEM image of Catalyst

(A = uncalcined, B = 800˚C calcined, C = 900˚C calcined, D = 1000˚C calcined)

In Figure 13 shows microstructure of the same powders of uncalcined and calcine catalyst. Their crystallization behaves as a function of calcination temperature. We can see that 1000˚C has very good uniform and gives higher surface area while comparing with uncalcined catalyst that has very poor uniform of particle. At the same time SEM analysis has reported that possible element found in each sample with Energy Dispersive X-ray analysis (EDX).

4.3 EDX (Energy Dispersive X-ray analysis)

Energy Dispersive X-ray analysis is another x-ray technique used to identify the elemental composition of material. The results reported as below.

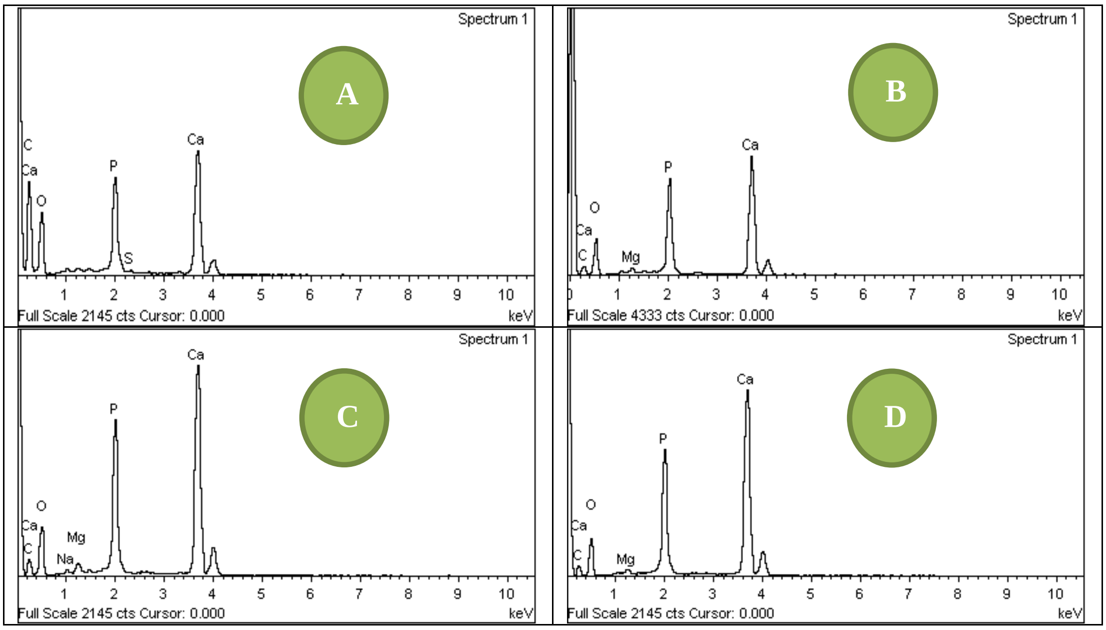


Figure 14: EDX Result

(A = uncalcined, B = 800˚C calcined, C = 900˚C calcined, D = 1000˚C calcined)

Element	Weight%			
	Uncalcine	Calcine 800 C	Calcine 900 C	Calcine 1000 C
C	42.71	15.67	17.50	14.48
O	36.99	43.40	39.30	39.00
S	0.26	0.00	0.00	0.00
Na	0.00	0.00	0.55	0.00
Mg	0.00	0.79	0.91	0.64
P	6.64	14.07	13.60	14.26
Ca	13.40	26.07	28.14	31.62
Total	**100.00**	**100.00**	**100.00**	**100.00**

Table 4: Element Summary of Catalyst

From EDX result, we can see that higher temperature treated for calcination, higher calcium presented. This is because calcium has higher boiling point if compare to other element, the possibility of weight losing at very high temperature will be lower.

4.4 FTIR (Fourier Transform Infrared Spectroscopy)

FTIR spectroscopy is used primarily for qualitative and quantitative analysis of unknown compounds and determines the chemical structure of the compounds. It is required to convert the raw data into the actual spectrum by measure light transmittance or absorption of sample at each different wavelength like a fingerprint of molecular structure.

Sample	**OH^-**	**H_2O**	**CO_3^{2-}**	**PO_4^{3-}**	**HPO_4^{2-}**	**OH^-**	**PO_4^{3-}**
Standard spectrum (cm^{-1})	3570	2928	1573	1090,1040, 960	983	634	603,565

Table 5: FTIR Standard Spectrum

It observes that in uncalcined catalyst has very high moisture and HPO^{2-} component as shown in Figure 16. This is because during calcination the moisture content was removed and HPO^{2-} was transform into other element such as PO_4^{3-} which is the element of hydroxylapatite component.

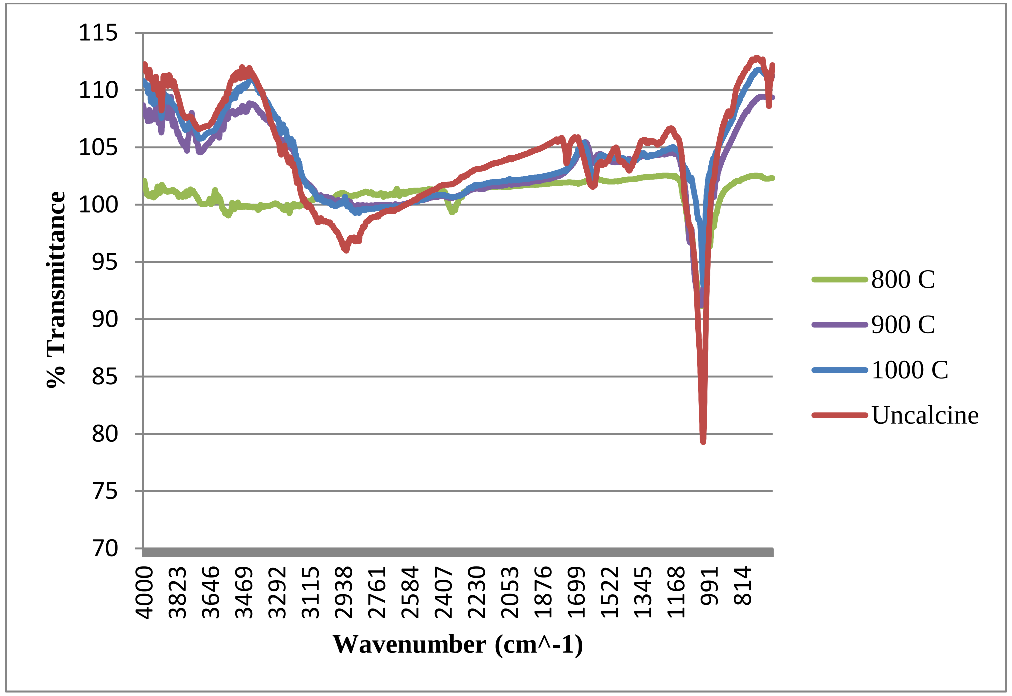


Figure 15: FTIR Result

4.5 BIODIESEL SYNTHESIS

After conducted experiment, the result as showing below

Catalyst Type	Biodiesel weight (g)	% Yield
800˚C	33.58	83.95
900˚C	31.84	79.60
1000˚C	35.73	89.33

Table 6: Biodiesel Synthesis Result

From $$\%\text{Yield} = \frac{\text{weight of product (g)}}{\text{weight of raw oil (g)}} * 100 \tag{1}$$

$$\%\text{Yield of } 800^{\circ}\text{C type} = \frac{33.58\text{ g}}{40\text{ g}} * 100$$

= 83.95%

$$\%\text{Yield of } 900^{\circ}\text{C type} = \frac{31.84\text{ g}}{40\text{ g}} * 100$$

= 79.60%

$$\%\text{Yield of } 1000^{\circ}\text{C type} = \frac{33.58\text{ g}}{40\text{ g}} * 100$$

= 89.33%

We can see that 1000˚C of calcined catalyst gives highest conversion because of smaller particle size that gives higher surface area, higher accelerated reaction as well as the presenting of highest fraction of calcium which is an alkaline metal that sensitive element and high ability to accelerate the reaction and it is commonly use in biodiesel industry.

4.5 GC-MS (Gas Chromatography-Mass Spectroscopy)

GC-MS can identify and quantify volatile organic compounds within a sample by breaking down samples into their components and analyzing each component with a mass spectrometer.

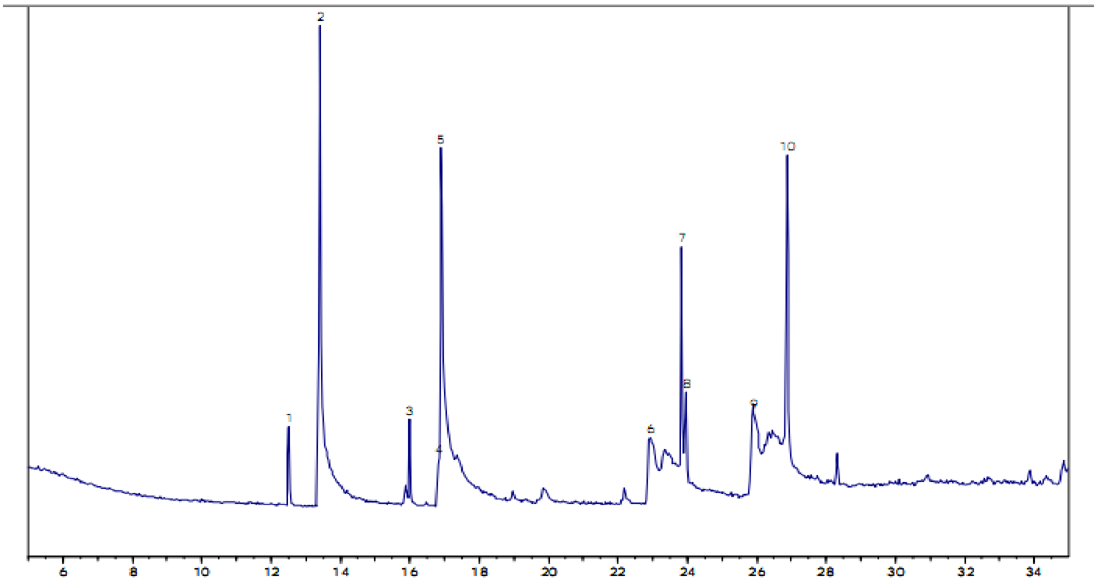


Figure 16: GC-MS Result of Biodiesel

No. Peak	Peak Name	Peak Area
1	methyl ester	352211
2	n-Hexadecanoic acid	3370346
3	methyl ester	406588
4	2-cyclohexenyl methyl ketone	237217
5	9-Octadecenoic acid	3447619
6	methyl ester	375496
7	Palmitic acid	1079514
8	1,2-Benzenedicarboxylic acid	358552
9	1-Dimethyl(chloromethyl)silyloxybutane	1063664
10	Monoolein	1887697
Total		**12578902**

Figure 17: GC-MS Peak Identify Result

GC-MS result can prove that the reaction can produce biodiesel as presenting of methyl ester.

CHAPTER 5: CONCLUSION & RECOMMEDATION

On the basis of the investigation results it could be concluded that the main component of the calcined chicken bone catalyst is hydroxylapatite, as a type of calcium apatite, which can be found in all three different treats.The waste chicken bone has a great potential to be used as a viable and economical biocatalyst for tranesterification. The analysis can also prove that higher temperature of calcination gives better physical and chemical properties of catalyst as giving higher conversion of biodiesel.

Therefore, this feasibility study project can verify that the waste chicken bone can be transformed to catalyst for biodiesel production by calcination treated. As many researchers attempt to find the alternatives catalyst for biodiesel that is effective, environmentally friendly and economic benefit.

Lastly, this project should be perfect if effectiveness of the catalyst and impact of catalyst to kinetics of transesterification were studied. But with the time constrain of this subject, final year project, the author can only complete more on catalyst characterization.

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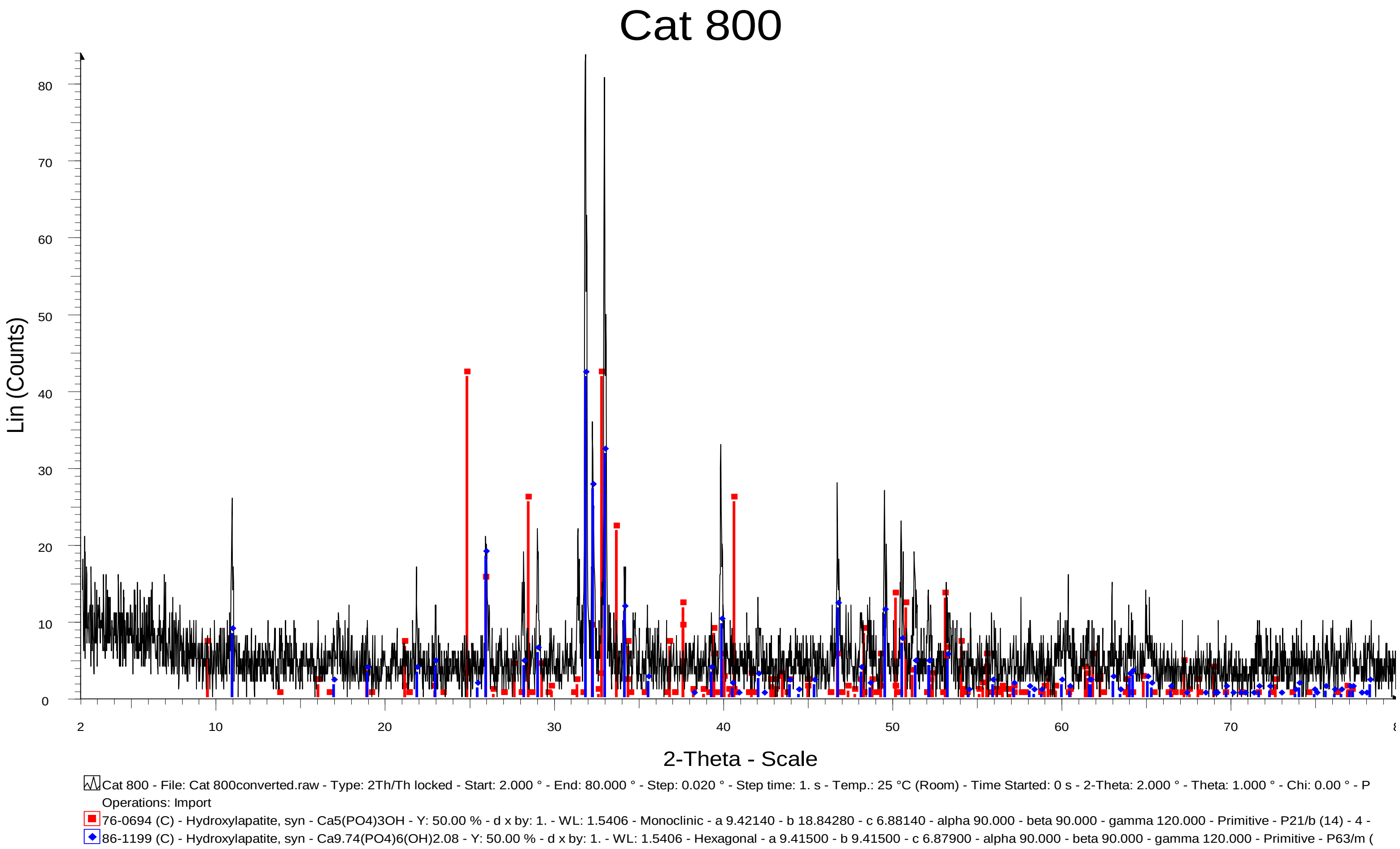
APPENDIX A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60
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20
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50
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70
8
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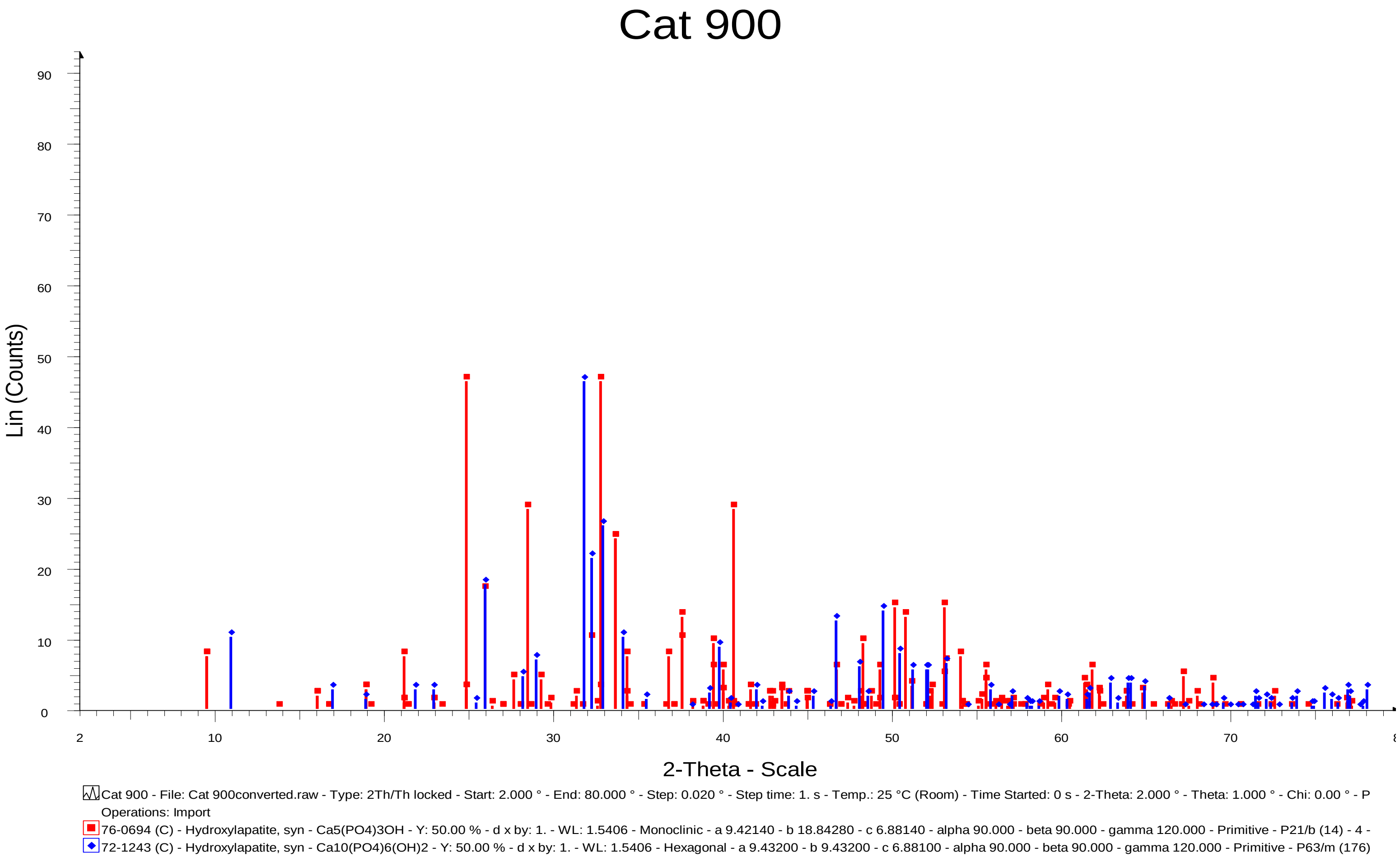
Operations: Import
🟥 76-0694 (C) - Hydroxylapatite, syn - Ca5(PO4)3OH - Y: 50.00 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 9.42140 - b 18.84280 - c 6.88140 - alpha 90.000 - beta 90.000 - gamma 120.000 - Primitive - P21/b (14) - 4 -
◆ 86-1199 (C) - Hydroxylapatite, syn - Ca9.74(PO4)6(OH)2.08 - Y: 50.00 % - d x by: 1. - WL: 1.5406 - Hexagonal - a 9.41500 - b 9.41500 - c 6.87900 - alpha 90.000 - beta 90.000 - gamma 120.000 - Primitive - P63/m (



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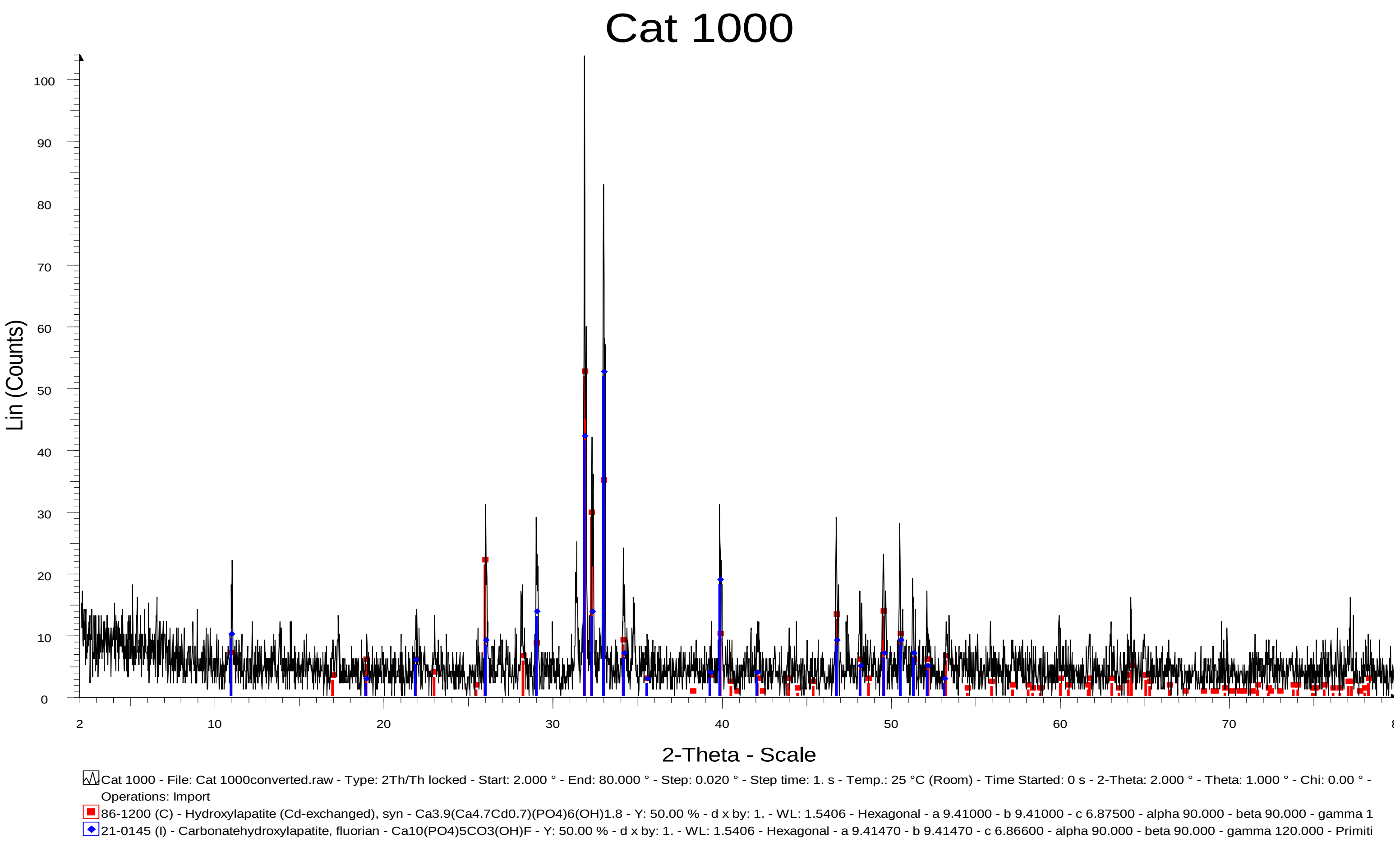
Operations: Import
🟥 76-0694 (C) - Hydroxylapatite, syn - Ca5(PO4)3OH - Y: 50.00 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 9.42140 - b 18.84280 - c 6.88140 - alpha 90.000 - beta 90.000 - gamma 120.000 - Primitive - P21/b (14) - 4 -
◆ 72-1243 (C) - Hydroxylapatite, syn - Ca10(PO4)6(OH)2 - Y: 50.00 % - d x by: 1. - WL: 1.5406 - Hexagonal - a 9.43200 - b 9.43200 - c 6.88100 - alpha 90.000 - beta 90.000 - gamma 120.000 - Primitive - P63/m (176)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2-Theta - Scale
Cat 1000 - File: Cat 1000converted.raw - Type: 2Th/Th locked - Start: 2.000 ° - End: 80.000 ° - Step: 0.020 ° - Step time: 1. s - Temp.: 25 °C (Room) - Time Started: 0 s - 2-Theta: 2.000 ° - Theta: 1.000 ° - Chi: 0.00 ° - Operations: Import
86-1200 (C) - Hydroxylapatite (Cd-exchanged), syn - Ca3.9(Ca4.7Cd0.7)(PO4)6(OH)1.8 - Y: 50.00 % - d x by: 1. - WL: 1.5406 - Hexagonal - a 9.41000 - b 9.41000 - c 6.87500 - alpha 90.000 - beta 90.000 - gamma 1
21-0145 (I) - Carbonatehydroxylapatite, fluorian - Ca10(PO4)5CO3(OH)F - Y: 50.00 % - d x by: 1. - WL: 1.5406 - Hexagonal - a 9.41470 - b 9.41470 - c 6.86600 - alpha 90.000 - beta 90.000 - gamma 120.000 - Primiti

APPENDIX B

	Stretching Vibrations			**Bending Vibrations**		
Functional Class	**Range (cm^{-1})**	**Intensity**	**Assignment**	**Range (cm^{-1})**	**Intensity**	**Assignment**
Alkanes	2850-3000	str	CH_3, CH_2 & CH 2 or 3 bands	1350-1470	med	CH_2 & CH_3 deformation
				1370-1390	med	CH_3 deformation
				720-725	wk	CH_2 rocking
Alkenes	3020-3100	med	=C-H & =CH_2 (usually sharp)	880-995	str	=C-H & =CH_2
	1630-1680	var	C=C (symmetry reduces intensity)	780-850	med	(out-of-plane bending)
	1900-2000	str	C=C asymmetric stretch	675-730	med	cis-RCH=CHR
Alkynes	3300	str	C-H (usually sharp)	600-700	str	C-H deformation
	2100-2250	var	C≡C (symmetry reduces intensity)			
Arenes	3030	var	C-H (may be several bands)	690-900	str-med	C-H bending & ring puckering
	1600 & 1500	med-wk	C=C (in ring) (2 bands) (3 if conjugated)			
Alcohols & Phenols	3580-3650	var	O-H (free), usually sharp	1330-1430	med	O-H bending (in-plane)
	3200-3550	str	O-H (H-bonded), usually broad	650-770	var-wk	O-H bend (out-of-plane)
	970-1250	str	C-O			
Amines	3400-3500 (dil. soln.)	wk	N-H (1°-amines), 2 bands	1550-1650	med-str	NH_2 scissoring (1°-amines)
	3300-3400 (dil. soln.)	wk	N-H (2°-amines)	660-900	var	NH_2 & N-H wagging (shifts on H-bonding)
	1000-1250	med	C-N			
Aldehydes & Ketones	2690-2840(2 bands)	med	C-H (aldehyde C-H)			
	1720-1740	str	C=O (saturated aldehyde)	1350-1360	str	α-CH_3 bending
	1710-1720	str	C=O (saturated ketone)	1400-1450	str	α-CH_2 bending

	1690	str	aryl ketone	1100	med	C-C-C bending
	1675	str	α, β-unsaturation			
	1745	str	cyclopentanone			
	1780	str	cyclobutanone			
Carboxylic Acids & Derivatives	2500-3300 (acids) overlap C-H	str	O-H (very broad)	1395-1440	med	C-O-H bending
	1705-1720 (acids)	str	C=O (H-bonded)			
	1210-1320 (acids)	med-str	O-C (sometimes 2-peaks)			
	1785-1815 (acyl halides)	str	C=O			
	1750 & 1820 (anhydrides)	str	C=O (2-bands)			
	1040-1100	str	O-C			
	1735-1750 (esters)	str	C=O			
	1000-1300	str	O-C (2-bands)	1590-1650	med	N-H (1°-amide) II band
	1630-1695(amides)	str	C=O (amide I band)	1500-1560	med	N-H (2°-amide) II band
Nitriles	2240-2260	med	C≡N (sharp)			
Isocyanates,Isothiocyanates, Diimides, Azides & Ketenes	2100-2270	med	-N=C=O, -N=C=S -N=C=N-, -N_3, C=C=O			