Conversion of Solid Biomass Waste (Oil Palm Shell) Into Potassium Based Fertilizers

by

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Dissertation submitted in partial fulfilment of the requirements for the Bachelor of Engineering (Hons) (Chemical Engineering)

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the Chemical Engineering Programme Universiti Teknologi PETRONAS In partial fulfilment of the requirement for the BACHELOR OF ENGINEERING (Hons) (CHEMICAL ENGINEERING)

Approved by,

(Prof. Dr. Thanabalan Murugesan)

UNIVERSITI TEKNOLOGI PETRONAS TRONOH, PERAK May 2014

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-source or persons.

(MOHD RAZIN BIN ROSELAN)

ABSTRACT

The aim of this project is to study the conversion of solid biomass waste using oil palm shell into potassium based fertilizer using different parameters such as the concentration of the sulphuric acid, the effect of stirring time for dissolving process, the effect of temperature and the effect of healing time. Empty fruit bunch (EFB) were chosen in this project because it can be used as reference for the oil palm shell to produce potassium based fertilizer since people already used empty fruit bunch as their source of fertilizer. Oil palm shell and empty fruit bunch have high content of potassium in their ashes which are 12.1% and 48.3% respectively. The problems arise when solid waste increases annually from the waste of the oil palm industry which can create disposal problems that lead to the environmental problems. The experiment conducted by incinerates the oil palm shell and empty fruit bunch to obtain the ashes that will be added with different concentration of Sulphuric acid. The qualities of the potassium baser fertilizer are determined by the concentration of the fertilizers and its pH value. High Performance Liquid Chromatography (HPLC) will be used to determine the concentration of the components of the fertilizer by calculating the area under peak and the retention time. From the result obtained from the experiments, the best fertilizer from the experiment for oil palm shell is from 20 mL of 0.08M of Sulphuric acid was added with the stirring time of 50 minutes while for empty furit bunch is from 20 mL of 0.2M Sulphuric acid with the stirring time of 50 minutes. The concentration of Potassium and the pH of the fertilizer for empty fruit bunch is 0.5776 mg/L and 8.05 while for oil palm shell is 0.4405 mg/L and 8.12, respectively. The concentration of Potassium could be more accurate if the High Performance Liquid Chromatography (HPLC) can detect the components inside the fertilizers by showing the peak and area under the graph for every components inside the fertilizer. As a recommendation, a future work may be done by studying the effect of Potassium based fertilizer from oil palm shell to the plants and studying on how to increase the Potassium content inside the fertilizer from oil palm shell.

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CHAPTER 1:

INTRODUCTION

1.0 INTRODUCTION

This chapter was about the introduction of this project that consists of the background of study, the problem statements, the objectives of this project and the scope of study. In the background of study, the author wrote about how the project was going to be done and the process used to produce the ash which is incineration. The author also wrote the purpose of this project which is to utilize the solid waste from the oil palm waste which it will be reacted with sulphuric acid to produce potassium based fertilizer under 4 different variables and the quality of the fertilizer will be determine based on the concentration of Potassium inside the fertilizer and its pH value. In the problem statements, the author issued 3 problems which were about the disposal problems of solid waste from oil palm industry that increased annually, the open burning of the wastes that can create environmental impact and the organic fertilizers that easily wash away by rain that can increase the pH value of the surrounding.

There were 3 aims of this project in the objective part which focused on producing the fertilizers in different operating parameters that can be checked its quality by measuring the pH value and concentration of the fertilizers produced. The scope of study will tell us on how to achieve the objectives of this project which is by focusing on 4 operating parameters. The 4 operating parameters were the concentration of sulphuric acid, the effect of stirring time, the effect of temperature and the effect of healing time.

1.1 BACKGROUND OF STUDY

This project is related to solid biomass waste focusing in conversion of oil palm shell which is one of the oil palm waste produced after the seed was obtained in the oil palm industries. Oil palm industries produced solid waste such as oil palm trunks (OPT), oil palm fronds (OPF), empty fruit bunches (EFB) and palm pressed fibers (PPF) and palm shells. Figure 1.1 show the type of oil palm biomass from the palm oil production. The method that will be used for the conversion of solid biomass waste for this project is incineration. Incineration is a waste treatment process that involves the combustion of organic substances contained in waste materials that will converts the waste into ash, flue gas and heat according to the certain temperature (Knox, & Andrew, 2005). The purpose of this project is to utilize the solid waste from the oil palm waste which it will be reacted with sulphuric acid to produce potassium based fertilizer and to determine the effect of several variables to produce potassium sulphate. Based on the presence of potassium in the oil palm waste, potassium oxide will be introduced to increase the amount of potassium in the ashes of oil palm waste. The experiment will be conducted using oil palm waste that will be obtained from the factory that processes the oil palm fruit. There are 4 parameters that will be tested as the variables 1) the concentration of sulphuric acid, 2) the effect of stirring time for dissolving process, and 3) the effect of temperature. The quality and the yield of the fertilizer will be measured based on the final concentration of the fertilizer and the pH value of the sample.



Figure 1.1: The type of oil palm biomass

1.2 PROBLEM STATEMENT

The oil palm industry produced large amounts of solid waste from kernels and fibres, empty fruit bunches (EFB), as well as liquid waste, normally referred to as POME (Palm Oil Mill Effluent) – a liquid waste with a high content of Chemical Oxygen Demand (COD).. If the waste is not fully utilized, the waste will create disposal problem that will cause environmental impact. Figure 1.2 show that the oil palm biomass increased annually and it predicted can be increased up to 110 million tonnes (Innovation Malaysia Agency, 2013).



Figure 1.2: The solid waste produced and its predicted volume

Other than that, to convert the oil palm waste into fertilizer, the waste need to be incinerate first to produce ashes. People traditionally will convert the oil palm shell into ashes by open burning that will cause the release of carbon dioxide that can result to greenhouse effect. Before the era for oil palm shell being used as biomass, the oil palm owner will bury the palm shell underneath the land and let it becomes black colour palm shell compost. However, although it is an organic fertilizer, the hard shell takes much longer time for converting totally to organic fertilizer. Besides that, the gardener and the farmer also use inorganic fertilizer that contains a combination of chemicals and minerals that were produced in a refinery which a more reliable form of plant nourishment because its nutrient levels are calculated to be consistent. However, the immediate availability of nutrients in organic fertilizer results in these nutrients, particularly nitrogen, being "loose". This means that rain and other sources of water can easily wash the nutrients below the root level of the plant and eventually into the surrounding streams, rivers and lakes (Mullin, 2012). As the result, the plants receive no nourishment and high concentrations of nitrogen and other compound enter the ecosystem, with potentially lethal results for plant and animal life due to increase of pH value of the plant and water.

1.3 OBJECTIVE

The aims of the project are:

- To study the conversion of solid biomass waste from oil palm waste into potassium based fertilizer in different operating parameters which are the temperature, concentration of sulphuric acid and stirring time
- To identify the amount of concentration of sulphuric acid needed to produce the best potassium based fertilizer which has the highest potassium content
- To determine the quality of the fertilizer by measuring the pH value and concentration of the fertilizers produced at the end of the experiment

1.4 SCOPE OF STUDY

This project used oil palm waste available such as empty fruit bunches (EFB) and oil palm shell as the solid biomass waste that will be incinerate and reacted with sulphuric acid to produce potassium sulphate. To increase the efficiency of the project by increasing the amount of potassium contained in the ashes, potassium oxide will be added to ashes to react with sulphuric acid. The experiment is repeated for different operating parameters:

- 1. The concentration of sulphuric acid
- 2. The effect of stirring time for dissolving process
- 3. Effect of temperature

1.5 SUMMARY

As a conclusion, this chapter told us on how to produce Potassium sulphate which is the fertilizer in 4 different operating parameters that are the concentration of the sulphuric acid, the effect of stirring time, the effect of temperature and the effect of healing time. The aims of this project was to convert the palm kernel shell into potassium based fertilizer by utilizing the Potassium content inside the palm kernel ashes to produce better or same quality of fertilizer produced from empty fruit bunch. For the next action, the author will discuss about the content of the empty fruit bunch and palm kernel shell and the advantages of Potassium inside the fertilizers for the plant in the literature review.

CHAPTER 2

LITERATURE REVIEW

2.0 INTRODUCTION

This chapter consist of 5 major parts which were about the oil palm shell, the empty fruit bunch, the oil palm ash in potassium based fertilizer, the theoretical basis and the High Performance Liquid Chromatography (HPLC). In the oil palm shell and empty fruit bunch part, the author wrote about the physical and chemical properties of the biomass and the content of the ashes which were rich with Potassium. For example, the oil palm shell has high silicon dioxide and potassium oxide value while for empty fruit bunch, it has high calcium and potassium content. The author also wrote the uses of oil palm shell and empty fruit brunch where mostly it were used to produce charcoal and activated carbon in the filtration or adsorption process from the oil palm shell while for empty fruit brunch, it were used to produce wood-based goods and also to generate electricity.

In the oil palm ash in potassium based fertilizer part, the author wrote about the advantages of Potassium based fertilizer. For example, it can enhance plants defence system and also helps for root development in plants. The chemical reactions inside the fertilizers were explained inside the theoretical basis part and it also explained factors that affect the production of potassium sulphate fertilizer. Among the factors were temperature, stirring speed, longer stirring time and concentration. For the last part, the author explained the uses of High Performance Liquid Chromatography and how to obtain the concentration based on the retention time, area under the peak and the peak height for certain compound.

2.1 OIL PALM SHELL

2.1.1 What is oil palm shell?

Oil palm or it scientific name Elaeis Guineeensis Jacq is a tropical plant belong to the Arecaceae family (Abdullah & Sulaiman, 2013). It can be found in Malaysia, Indonesia as well as in East and central Africa. The oil palm fruit have oily succulent outer layer and a kernel rich in palm oil. Oil palm shells are the shell fractions left after the nut has been removed after crushing in the Palm Oil mill. Oil palm shell can be considered as pellet form because of its nature form that has high grade solid, high calorific value, low ash and low sulphur content. Moisture content in palm shell is quite low compared to other biomass residue. It contains residues of palm oil, which accounts for its slightly higher heating value than average lignocelullosic biomass. If compared with other residues from other industry, it is good quality biomass fuel with easy handling, uniform size distribution, easy crushing and limited biological activity due to low moisture content (Foo & Hameed, 2009). Figure below show the fruit of oil palm and the palm kernel shells (PKS). Table 2.1 shows the chemical composition of oil palm ash. (Fatiha, 2005).



Figure 2.1: The oil palm fruit and the palm Kernel Shell (PKS)

Chemical composition of oil palm	Percentage (%)
ash	
Silicon dioxide, SiO ₂	40.0
Potassium Oxide, K ₂ O	12.1
Calcium Oxide, CaO	10.0
Phosphrous Pentoxide, P ₂ O ₅	8.2
Magnesium Oxide, MgO	6.4
Aluminium Oxide, Al ₂ O ₃	6.1
Carbon, C	5.4
Iron oxide, Fe ₂ O ₃	2.5
Others	2.0
Ignition loss	7.3

Table 2.1: Chemical composition of oil palm ashes

2.1.2 Uses of oil palm shell

According to the Opean Energy company, the oil palm shell are used as boiler fuel source due to their relatively high calorific value, abundance of supply, ease of use and per tonnage cost since Malaysia produce 3.1 million tons of palm kernel shell (Johnson,2011). Other than that, carbonize palm kernel shell can be use as charcoal which can be pressed into bio-fuel briquette, that could be directly sell to consumer such as BBQ or family use. It also can be processed into activated carbon which use in liquid and gaseous phase filtration or adsorption. Besides that, it also can be use as partial replacement for coarse aggregate in asphalt concrete. It also can be converted into mattress fillings, medium density fiber board, molded wares, composite material, fertilizer, and paper and pulp.

2.2 EMPTY FRUIT BUNCHES (EFB)

2.2.1 What is Empty fruit bunches?

EFB is 100% waste or surplus from mills that processes palm fruit to extract the oil. Empty bunches are currently used in many industrial enterprises and agricultural activities. Some are processed into bunch ash fertilizers. However, many empty bunches will be discarded and not used as bunch ash fertilizer. Many plantations use a small amount for the internal use, either biodegrading into compost, or at times burnt to avoid space loss by storage (Goh & Tan, 2010). Therefore, there is a substantial amount of EBB left unused. EFB mainly consist of main stalk (20-25%) and numerous spikelets (75-80%) with sharp spines at their tips. Table 2.2 show the chemical composition of empty fruit bunches. (Udoetok, 2012)



Figure 2.2: The example of empty fruit brunch

Chemical composition of EFB	Percentage (%)
Heavy	Metals
Calcium, Ca	50.7
Potassium, K	48.3
Magnesium, Mg	0.6
Sodium, Na	0.2
Zinc, Zn	0.1
Chromium, Cr	0.03
Others	0.17
Anio	ons
Sulphate, SO ₄	74.8
Chloride, Cl	20.4
Nitrate, NO ₃	3.2
Phosphate, PO ₄	1.6

 Table 2.2: Chemical composition of empty fruit bunches

2.2.2 Uses of empty fruit bunches

Among the applications of empty fruit bunches are production of wood-based goods, paper and pulp production and also can be used to stabilize the soil material. Other than that, it also can be used as fuel to generate electric power output and also can be used as mulch in plantations usually in oil palm plantations. A lot of empty fruit bunch gets rot in the field that will be a source of very good organic fertilizers. It also good sources for fertilizers for crops particularly the supply of material such as muriate of potash. Studies show that every ton of EFB contains 18 Kg of muriate of potash and 3.8 Kg of urea (Johnson, 2011). All this materials are required by all crops, including oil palm trees.

2.3 OIL PALM ASH IN POTASSIUM BASED FERTILIZER

The two highest content of the ash of oil palm shell is Potassium and Calcium (Udoetok, 2012). In N-P-K fertilizers, potassium is responsible for plant metabolism which acts as support for the nutrients nitrogen (N) and phosphorus (P). Potassium aids plants in retaining water, regulates plants internal cation-anion balance and aids protein synthesis to energize plants for healthy growth. It also helps for root development in plants, which is helpful for young plants putting down new root systems. Potassium also enhances plants defence system which thickens cell walls and strengthens stems, stalks and roots to make plants more resistant to disease and better able to tolerate stress.

2.4 THEORETICAL BASIS

By adding the Potassium Oxide (K2O) solution into the ashes, the content of the potassium inside the ashes will be more than Calcium inside the ashes. The potassium oxide then will react with sulphuric acid after sulphuric acid was added into the solution.

$K_2O(I) + H_2SO_4(I) \longrightarrow K_2SO_4(I) + H_2O(I)$

The reaction above is an irreversible reaction which means that the right reaction (product) cannot return to the left (reactants). The mechanism of such reactions is an inorganic reaction, which generally lasts fast inorganic reactions (Vogel, 1979). Inorganic reactions are influenced by the speed of stirring, the time and the temperature.

Since there are other elements in the ashes such as Calcium, Magnesium and Sodium, it also will react with potassium oxide and sulphuric acid. Below are reactions among others:

 $CaO + H_2SO_4 \longrightarrow CaSO_4 + H_2O$ $MgO + H_2SO_4 \longrightarrow MgSO_4 + H_2O$ $Na_2O + H_2SO_4 \longrightarrow NaSO_4 + H_2O$

There are also other elements of content but only in small quantities. Among the factors that affect the production of potassium sulphate fertilizer are:

a. Temperature

Temperature can affect the chemical reactions and the reaction rates. As the temperature increases, the rate of reaction will also increase. The solubility of a solute also will increase with increasing temperature that also will increase the reaction speed.

b. Stirring speed

As the stirring speed increase, the diffusivity of a substance and its solubility will also increase. This is because the particles of matter can move freely in any directions which allow contact between particles so rapidly saturated condition is reached.

c. Longer stirring time

The dissolving process or the solubility also influenced by longer stirring time. The longer the stirring time, the solubility of a substance will be increase.

d. Concentration

The concentration of the sulphuric acid also will affect the solubility and the final concentration of the fertilizer. If the concentration of the sulphuric acid is too high, then mixture will become saturated with sulphuric acid.

2.5 HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

The result of the experiment will be analyzed by checking the concentration of the fertilizer. To determine the concentration of the Potassium and sulphate ion, the author used High Performance Liquid Chromatography (HPLC). In analytical chemistry, HPLC was used to separate the component of the mixture, to identify and to quantify each of the components. The pressurized liquid solvent containing sample mixture will pass through a column filled with a solid adsorbent material that relies on pumps.

As the components of the sample flow out of the column, it will produce different flow rates for the different components since each of the components in the sample reacts differently with the adsorbent material. Many organic compounds absorb UV light of various wavelengths. To get the direct reading of how much the light is absorbed, a beam of UV light will shining through the stream of liquid that coming out of the column and the UV detector on the opposite of the stream will record the reading. Figure 2.3 show the process of UV adsorption.



Figure 2.3: The process of UV adsorption

The use of smaller particle size for the column packing material give advantage which is much greater surface area for interactions between the stationary phase and the molecules flowing pass it. Figure 2.4 below show the flow scheme for HPLC.



Figure 2.4: Flow scheme for HPLC

To interpret the output from the detector, the output will be recorded as a series of peaks that represent a compound in the mixture passing through the detector and absorbing UV light. Analysis made is important to determine the concentration of targeted compounds or species inside the solution. These parameters are:

- i. Retention time
- ii. Area under the peak
- iii. Peak height for certain compound

Retention time t_R is the time taken for a particular compound to travel through the liquid column to the detector. This is the exact time shown by measuring from the time at which the sample is injected to point at which the detector detects the signals and consequently, displaying it in the interface as maximum peak height for that compound. Different compound have different retention times.

The area under the peak is proportional to the amount of sample which has passed the detector, and this area can be calculated directly by the software used while operating the HPLC machine. Figure below shows the example of peak that has certain value of area.



Figure 2.5: Example of area under the Peak

Since the result will contain many peaks of the components in the sample, the author will focus on the target and investigated species which is potassium sulphate. Therefore, the other peak is negligible.

2.6 SUMMARY

As a conclusion, empty fruit bunch has higher Potassium content compared to the palm kernel shell. Besides that, Potassium based fertilizer was better compared to N-P-K fertilizers since it helps for root development system and enhances plant defence system. Other than that, the addition of Sulphuric acid into the ashes will not only produced Potassium Sulphate but also other by-products such as Calcium Sulphate and Magnesium Sulphate. Last but not least, the concentration of Potassium inside the fertilizer can be determined using High Performance Liquid Chromatography (HPLC) from its retention time, area under peak and peak height of the compound. For next chapter which is Methodology, the author will discuss on the project timeline and how to conduct the experiment to achieve its objectives of the project.

CHAPTER 3

METHODOLOGY

3.0 INTRODUCTION

This chapter consist of 3 major parts which were the project activities, the Gantt chart and key milestone and the experiment methodology. In the project activities, it explained on the project planning throughout the project execution which starting from the literature review until the conclusion part. For the Gantt chart and key milestone part, the author described the detailed activity of the project according to the timeline made by the coordinator of the final year project. The purpose of Gantt chart was to avoid late submission of the detailed activity from the due date.

For the experiment methodology, it consist of 6 parts which were the sample preparation, tool and equipment, experiment setup, the substance and chemical used, range of variables and the proposed experimental procedure. In the sample preparation, the author explained on how to produce the ashes from the empty fruit brunch and palm kernel shell obtained. For tools and equipment, the author used the furnace to produce the ashes, the heater with magnetic stirrer and High Performance Liquid Chromatography (HPLC) to obtain the concentration of the Potassium inside the fertilizer. For experiment setup and chemicals part, the author described on how to start the experiment using the chemicals and apparatus that has been ordered from the lab technician. There were 2 variables which were the fixed variables and manipulated variables. The proposed experimental procedure explained on how to conduct the experiment to obtain the result for this project.

3.1 PROJECT ACTIVITES

Literature Review

- Understanding the advantages and process on producing potassium based fertilizer
- More research on how to interpret the HPLC result obtained to calculate the concentration of the fertilizer

Experiment

- Experiment is designed to check the concentration and the pH of the potassium based fertlizer.
- Chemicals and equipment required for the experiment is prepared and currently the experiment is ongoing.

Data Collection

- From the experiment conducted, the sample are prepared for the analysis.
- The sample prepared are analysed using High Performance Liquid Chromatography (HPLC) to obtain the concentration and pH meter to obtain the pH of the fertilizer

Conclusion

- The experiment will be concluded based on the results at the end of the project.
- Report of the experiment will be prepared.

3.2 GANTT CHART AND KEY MILESTONE

No	Detailed Activity Weeks	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	Project work continues															
2	Submission of progress report															
3	Project work continues															
4	Pre-Sedex presentation															
5	Submission of Draft report															
6	Submission of Dissertation (Soft bound)															
7	Submission of															

	Technical Paper								
8	Oral Presentation								
9	Submission of Project								
	Dissertation (Hard								•-
	Bound)								



3.3 EXPERIMENT METHODOLOGY

3.3.1 Oil palm shell and empty fruit bunches sample preparation

For the sample preparation of this project, oil palm shell and empty fruit obtained from the oil palm factory since the factory already separate the waste from the oil palm.For the empty fruit bunch, we cut the samples into smaller pieces before we dried it to ease the process of drying and grinding in the future. Then, the oil palm shell and empty fruit brunches were dried under the sun to remove the moisture from the waste. For better moisture remover, we put the sample into an oven for 24 hours at the temperature of 150 °C. After that, the samples were grinded to the smaller pieces in the grinder to the size of 0.5 mm to ease the incenaration process by increasing the surface area or contact area during the process. To obtain the ashes, the wastes were incenerated in the furnace under the temperature of 900°C for oil palm shell and 800°C for empty fruit brunches. After that, the ashes obtained will be use for the preparation of potassium based fertilizer.



Figure 3.1: The samples that were dried under the sun

3.3.2 Tools and equipment

For the incineration process, the essential part of the equipment is the incinerator. However, adjustment is needed to incinerate two different part of oil palm waste. The incinerator will heat up until 900 °C for oil palm shell and 800 °C for empty fruit bunches. It is because of the different thickness of the shell that need more heat to incinerate it until become ashes compare to the empty fruit bunches.

To study the content or the composition of the ashes, spectrophotometer will be used to check the content of the oil palm shell and empty fruit bunches. The data collected can be used to check the content of potassium inside the waste that can be compare and used to produce potassium based fertilizers.

The magnetic stirrer will be used to kept the constant speed of stirring throughout the experiment. The refractometer will be used to check the pH value of the fertilizer.

To check the concentration of the fertilizer, High Performance Liquid Chromatography (HPLC) will be used in this experiment. The sample will go through the machine to get the area under the peak and the retention time. Some calculation will be done to get the concentration of the fertilizer.

3.3.3 Experiment setup



Figure 3.2: The experiment setup

The samples were placed in the 100mL beaker with the addition of sulphuric acid. It is placed on magnetic stirrer to ensure continous mixing of the solution inside. A thermometer is place inside the beaker to check and regulate the temperature of the fertilizer. A pH meter is used to measure the pH value of the sample every 10 minutes of the experiment. The samples are withdrawn using a 10mL syringe and put into vials for further analysis using High Performance Liquid Chromatography (HPLC).

3.3.4 Substance and Chemicals

List of chemicals needed for this experiment are:

Sulphuric Acid (H₂SO₄) and water (H₂O).

3.3.5 Range of Variables

Fixed Variables

Speed of magnetic stirrer	200 rpm
Extract volume of ash	20 mL
Volume of H ₂ SO ₄	20 mL

Variable Changes

Concentration of	0.08, 0.09, 0.1, 0.2, 0.3 M
sulphuric acid	
Stirring time	30,40,50,60, and 70 minutes
Temperature	50, 60, 70, 80, 90 °C
Sample preparation	• Beaker A: Oil palm ash
	only
	• Beaker B: Empty fruit
	bunches ash only

3.3.6 Proposed Experimental Procedure

The experiment is planned with the starting temperature of 50 C. Taken the previous research work as guidance (Kurniati, 2011), the procedure for conversion of solid biomass waste to potassium based fertilizer are:

- 1. Placed the ash of the oil palm shell into beaker A.
- 2. Dissolved the ashes in water by adding 500 mL of distilled water, and then take the extact by filtering using filter paper.
- Enter a 20 mL of potassium oxide into the 100mL beaker that contain the extrat of the ashes. Then add 20mL sulphuric acid with concentration 0.08 M into the beaker.
- Put the beaker on the magnetic stirrer and heat the solution up to 50 C. Maintain and check the temperature using thermometer.
- 5. After 30 minutes, increase the temperature to 60 C. For every 10 minutes, increase 10 C of the temperature.
- 6. Turn the stirrer for 30, 40, 50, 60 and 70 minutes and set the speed of the stirrer to 200 rpm.
- To determine the concentration and the pH value of the mixture every, a small amount of the sample of the fertilizer will be taken at 30, 40, 50, 60 and 70 minutes.
- 8. The sample then will be go to the HPLC machine to check its retention time and area under the peak that will be obtained from the result to determine its concentration.
- 9. Repeat the experiment by using 0.09, 0.1, 0.2 and 0.3 M of sulphuric acid into the solution.
- 10. Repeat the whole set of experiment by replacing the palm kernel shell with empty fruit bunch in beaker B.

3.4 SUMMARY

As a conclusion, this chapter explained on the project execution and project experiment. The author has executed the experiment according to the procedure and the result has been obtained. The author was able to finish the experiment within the timeline and submit the detailed activity such as the progress report on time to the supervisor and the coordinator. For the next chapter, the author will show the result obtained from the experiment and explained the result obtained in the discussion part.

CHAPTER 4

RESULT AND DISCUSSION

4.0 INTRODUCTION

This chapter will consist of three parts which were the result obtained from the experiment conducted, the discussion part and the problem encountered. For the result obtained, it has divided into which were the result for the palm kernel shell and the result for the empty fruit bunch. The author explained the result in table form that consists of the time conducted, the temperature used, the pH recorded and the concentration of Potassium calculated. The author also describes the relationship between concentration of Potassium and pH value against time conducted. In the discussion part, the author explained the result obtained in three factors which were the effect of temperature, the effect of longer stirring time and the effect of concentration of sulphuric acid. There were two problems encountered during the experiment which were the temperature control and the result of High Performance Liquid Chromatography (HPLC).

4.1 RESULT FOR FERTILIZER USING OIL PALM KERNEL SHELL

The result from the High Performance Liquid Chromatography (HPLC) and the calculation of the concentration of potassium in the fertilizer are attached in the Appendix.

Time	Temperature	pН	Concentration of
(min)	(⁰ C)		Potassium (mg/L)
30	50	8.32	0.0536
40	60	8.18	0.2471
50	70	8.12	0.4405
60	80	8.11	0.3438
70	90	7.84	0.3051

Table 4.1: Concentration of K and pH of fertilizer on the addition of 0.08 M H₂SO₄



Figure 4.1Graph of concentration vs time with 0.08M of Sulphuric acid

Table 4.2: Concentration of K and pH of fertilizer on the addition of 0.09 M H₂SO₄

Time	Temperature	pН	Concentration of
(min)	(⁰ C)		Potassium (mg/L)
30	50	8.48	0.0533
40	60	8.23	0.2462
50	70	8.15	0.4392
60	80	8.10	0.3234
70	90	7.82	0.2655



Figure 4.2 Graph of concentration vs time with 0.09M of Sulphuric acid

Time (min)	Temperature (⁰ C)	рН	Concentration of Potassium (mg/L)
30	50	8.43	0.0531
40	60	8.16	0.2455
50	70	8.14	0.4378
60	80	8.12	0.3032
70	90	7.78	0.2070

Table 4.3: Concentration of K and pH of fertilizer on the addition of 0.10 M H_2SO_4



Figure 4.3 Graph of concentration vs time with 0.10M of Sulphuric acid

Time (min)	Temperature (^o C)	рН	Concentration of Potassium (mg/L)
30	50	8.35	0.0520
40	60	8.20	0.2443
50	70	8.17	0.4256
60	80	8.13	0.3591
70	90	7.72	0.2635

Table 4.4: Concentration of K and pH of fertilizer on the addition of 0.20 M H₂SO₄



Figure 4.4 Graph of concentration vs time with 0.20M of Sulphuric acid

Table 4.5: Concentration of K and pH of fertilizer on the addition of 0.30 M H₂SO₄

Time (min)	Temperature (^o C)	рН	Concentration of Potassium (mg/L)
30	50	8.30	0.0526
40	60	8.18	0.2431
50	70	8.05	0.4335
60	80	8.01	0.3764
70	90	7.68	0.3002



Figure 4.5 Graph of concentration vs time with 0.30M of Sulphuric acid

4.2 RESULT OF THE FERTILIZER USING EMPTY FRUIT BUNCH

Ti	ime	Temperature	pН	Concentration of
(n	nin)	(⁰ C)		Potassium (mg/L)
	30	50	8.39	0.0847
4	40	60	8.16	0.3560
4	50	70	8.13	0.5739
(50	80	8.08	0.4149
7	70	90	8.07	0.3324

Table 4.6: Concentration of K and pH of fertilizer on the addition of 0.08 M H₂SO₄



Figure 4.6 Graph of concentration vs time with 0.08M of Sulphuric acid

Time	Temperature	pН	Concentration of
(min)	(⁰ C)		Potassium (mg/L)
30	50	8.40	0.1452
40	60	8.18	0.3948
50	70	8.04	0.5445
60	80	8.02	0.4447
70	90	7.98	0.2950

Table 4.7: Concentration of K and pH of fertilizer on the addition of 0.09 M H₂SO₄



Figure 4.7 Graph of concentration vs time with 0.09M of Sulphuric acid

Table 4.8: Concentration of K and pH of fertilizer on the addition of 0.10 M H₂SO₄

Time (min)	Temperature (^o C)	рН	Concentration of Potassium (mg/L)
30	50	8.36	0.1425
40	60	8.13	0.3919
50	70	8.05	0.5266
60	80	8.02	0.4342
70	90	7.95	0.2846



Figure 4.8 Graph of concentration vs time with 0.10M of Sulphuric acid

Time (min)	Temperature (⁰ C)	рН	Concentration of Potassium (mg/L)
30	50	8.42	0.0820
40	60	8.18	0.3298
50	70	8.05	0.5776
60	80	7.86	0.4537
70	90	7.83	0.3793

Table 4.9: Concentration of K and pH of fertilizer on the addition of 0.20 M H_2SO_4





Time	Temperature	pН	Concentration of
(min)	(⁰ C)		Potassium (mg/L)
30	50	8.35	0.1365
40	60	8.05	0.3785
50	70	7.88	0.5187
60	80	7.82	0.4069
70	90	7.70	0.2475

Table 4.10: Concentration of K and pH of fertilizer on the addition of 0.30 M H₂SO₄


Figure 4.10 Graph of concentration vs time with 0.30M of Sulphuric acid

4.3 DISCUSSION

4.3.1 Effect of temperature

One of the major factor that affects the rate of chemical reaction is temperature. All the result showed the increase in concentration of Potassium in the fertilizer as the temperature increased. It is because as we increase the temperature, the rate of reaction also will increase.

$K_2O(I) + H_2SO_4(I) \longrightarrow K_2SO_4(I) + H_2O(I)$

The above reaction is inorganic reaction which is influenced by the temperature. The temperature also affect the solubility of the solute and increase the reaction speed. That is why at 50 minutes, all the result achieved the highest concentration of potassium in every set of experiment. However, the temperature need to be increase gradually because the fertilizer will become hardened since the water were evaporated from the fertilizer due to high temperature that will make stirring process become hard.

4.3.2 Effect of longer stirring time

The dissolving process also influenced by stirring time other than the effect of temperature. The longer the stirring time, the solubility of the substance will also increase. This is because the particles of matter can move freely in any direction which allow contact between particles so rapidly until saturated condition is reached.

The result showed the fertilizer was stirred up until 70 minutes and the reading were taken every 10 minutes after the 50°C was reached. Based on the result, the concentration of Potassium increased after 20 minutes of stirring. However, when the stirring time reached 60 minutes, the concentration of Potassium decreased. It is because the fertilizer already achieved its supersaturated condition where almost all of K ion was bound with SO4 ion. The result shows that the empty fruit bunch has higher concentration of Potassium in fertilizer compared to the palm kernel shell. It is because the concent of potassium in empty fruit bunch is higher compared to palm kernel shell.

4.3.3 Effect of concentration of sulphuric acid

Based on the result, the concentration of the sulphuric acid will affect the pH value and the concentration of the fertilizer. If the concentration of the sulphuric acid is too high, the fertilizer will become saturated with sulphuric acid and the fertilizer will become acidic due to decreasing pH value. The result shows that as the concentration of the sulphuric acid increased, the pH value of the fertilizer will become decreased. The pH value obtained from the experiment is alo acceptable based on the Environmental Quality Act 1974 that stated that the pH value of fertilizer must be range between 7.0 to 9.0.

4.4 PROBLEM ENCOUNTERED

4.4.1 Temperature Control

The temperature need to be increased by 10 °C every 10 minutes after the 50°C of temperature is reached. However, due to limitation of the equipment, the temperature show at the heater and the magnetic stirrer does not match with the temperature inside the beaker. We need to use thermometer and increase the temperature a little bit higher to achieved the desired temperature which will affect the outcome of the experiment.

4.4.2 Result of High Performance Liquid Chromatography (HPLC)

The result of the HPLC does not only detect Potassium inside the fertilizer, but it also detect other elements too. The HPLC equipment that we has is not accurate enough to detect the desired element which is the Potassium. Other than that, the calculation of the concentration of the potassium will become harder because we cannot confirm whether the peak that we choose is Potassium. We need to refer to the standard solution that we prepared to know the retention time of the Potassium.

4.5 SUMMARY

As a conclusion, the result showed the highest content of potassium were produced from palm kernel shell ashes when 20 mL of 0.08 H2SO4 was added with the stirring time of 50 minutes while for empty furit bunch is from 20 mL of 0.2 H2SO4 with the striing time of 50 minutes. The concentration of Potassium and the pH of the fertilizer for empty fruit bunch is 0.5776 mg/L and 8.05 while for palm kernel shell is 0.4405 mg/L and 8.12, respectively. The next chapter will conclude the project report starting from the introduction until the discussion part.

CHAPTER 5

CONCLUSION

As a conclusion, this project is vital as it deals with alternative ways of handling solid biomass waste from oil palm industries. Potassium based fertilizers is one of the product that can been produced by the solid biomass waste by utilizing the content of potassium inside the waste to produce the fertilizers. Potassium oxide is added in this experiment to increase the content of the potassium in the ashes and thus increase the quality of the fertilizers. There are many other uses or products can been produced using the solid biomass waste if more research been done regarding the waste products.

From the runs of experiment conducted, it can be said that this porject is showing a good progress and expected results are obtained. Although there are several problems encountered during the experiment and the analysis, it will not cause too much error in the experiment. From the research and the experiment that has been done, the best fertilizer from the experiment for palm kernel shell is from 20 mL of 0.08 H2SO4 was added with the stirring time of 50 minutes while for empty furit bunch is from 20 mL of 0.2 H2SO4 with the striing time of 50 minutes. The concentration of Potassium and the pH of the fertilizer for empty fruit bunch is 0.5776 mg/L and 8.05 while for palm kernel shell is 0.4405 mg/L and 8.12, respectively.

As a recommendation, a future work may be done by studying the effect of Potassium based fertilizer from oil palm shell to the plants and the study on how to increase the Potassium content inside the fertilizer from the palm kernel shell.

CHAPTER 6

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CHAPTER 7 APPENDICES

Examples of results from HPLC

1. Palm Kernel Shell

Concentration of H₂SO₄ = 0.08M

#	RetTime [min]		[min]	Area [mAU*s]	Height [mAU]	Area %
1	4.052	BB	0.1227	72.28306	8.37410	0.1398
2	11.616	BV	0.2277	157.17183	8.66037	0.3041
3	11.984	VV	0.2561	343.00610	16.94871	0.6636
4	13.012	VV	0.2862	2135.92358	94.31099	4.1321
5	13.135	VV	0.1299	2773.38281	289.15643	5.3654
6	13.398	VV	0.1269	1609.32617	175.66751	3.1134
7	13.891	VB	0.2488	4.45993e4	2670.41748	86.2816
Total	s :			5.16904e4	3263.53559	



Concentration of H₂SO₄= 0.09M

Peak #	RetTime [min]	Туре	Width [min]	Area [mAU*s]	Height [mAU]	Area %
		-				
1	4.056	BB	0.1227	321.38910	37.23993	0.6201
2	11.616	BV	0.2290	157.91310	8.65124	0.3047
3	11.983	VV	0.2560	343.24429	16.96618	0.6622
4	13.012	VV	0.2846	2134.75977	94.11210	4.1186
5	13.136	VV	0.1268	2763.68872	290.89819	5.3320
6	13.398	VV	0.1291	1612.37854	175.68633	3.1108
7	13.889	VB	0.2484	4.44990e4	2671.22070	85.8518
Total	ls :			5.18324e4	3294.77467	



Concentration of H₂SO₄= 0.10M

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	90
1	4.058	BB	0.1228	480.22504	55.58565	0.9236
2	11.617	BV	0.2277	157.06805	8.65583	0.3021
3	11.984	VV	0.2558	342.87048	16.96202	0.6594
4	13.011	VV	0.2846	2137.18237	94.23799	4.1104
5	13.136	VV	0.1271	2764.86060	290.36856	5.3176
6	13.398	VV	0.1282	1633.79736	176.23158	3.1422
7	13.888	VB	0.2479	4.44788e4	2675.55493	85.5447
Total	s:			5.19949e4	3317.59657	



Concentration of $H_2SO_4 = 0.20M$

Peak	RetTime Type	Width	Area	Height	Area
#	[min]	[min]	[mAU*s]	[mAU]	00
1	4.052 BB	0.1255	667.69110	75.27631	1.2774
2	11.621 BV	0.2277	157.25026	8.66396	0.3008
3	11.988 VV	0.2557	343.19046	16.98632	0.6566
4	13.015 VV	0.2841	2131.00488	94.11993	4.0770
5	13.139 VV	0.1274	2770.54932	290.05157	5.3006
6	13.400 VV	0.1320	1656.80200	175.76096	3.1698
7	13.898 VB	0.2467	4.45424e4	2669.42236	85.2178
Total	ls :		5.22689e4	3330.28142	



Concentration of H₂SO₄= 0.30M

Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	00
		·				
1	4.070	BB	0.1230	840.08887	97.06429	1.5999
2	11.625	BV	0.3136	221.22455	8.67301	0.4213
3	11.990	VV	0.2550	343.55344	17.05275	0.6543
4	13.016	VV	0.2846	2127.48633	93.81787	4.0516
5	13.139	VV	0.1271	2766.66455	290.39932	5.2688
6	13.400	VV	0.1296	1632.64734	173.72346	3.1092
7	13.903	VB	0.2485	4.45785e4	2673.75146	84.8950
Tota	ls :			5.25101e4	3354.48218	



2. Empty fruit bunch

Concentration of H₂SO₄ = 0.08M



Signal 3: DAD1 C, Sig=223,16 Ref=360,100

Peak	RetTime 1	Гуре		Area	Height	Area
	[min]		[min]		[mAU]	8
		-		I		
1				6.63321		7.823e-3
2	1.783 \			15.38768		0.0181
3	3.324 H			39.20177		
4	5.350 H	BV		835.70209		
5	6.193 \			754.58698		
6	7.662 1	vv		2310.25488		
7	8.093 1			899.50977		
8	8.231 \	vv	0.1116	336.18658	41.91074	0.3965
9	8.634 1	VV	0.3307	1237.20825	46.76016	1.4592
10	8.920 7	vv		818.17206		
11	9.531 1	vv	0.3909	1776.50854	56.18533	2.0953
12	11.342 \	vv	0.8148	8160.00928	121.00116	9.6241
13	11.423 \	vv	0.0771	656.48761	121.28933	0.7743
Peak	RetTime	Type	Width	Area	Height	Area
#				[mAU*s]	-	8
14	11.541	vv	0.0863	742.90179	123.37007	0.8762
15	11.913	vv	0.2434	7990.58252	436.94543	9.4243
16	12.302	vv	0.1143	3851.30322	448.16068	4.5423
17	12.398	vv	0.0974	3603.56616	516.31787	4.2501
18	12.492	vv	0.2232	8004.87744	488.56104	9.4412
19	12.896	vv	0.1300	3061.37207	337.06931	3.6107
20	13.026	vv	0.0718	1715.74744	346.28601	2.0236
21	13.140	vv	0.1505	7185.60059	632.88232	8.4749
22	13.386	vv	0.1804	5267.47266	408.53336	6.2126
23	13.680	vv	0.1180	3310.69067	371.50424	3.9047
24	13.750	vv	0.1342	3672.95044	375.09106	4.3320
25	13.909	vv	0.0783	2031.56323	368.85352	2.3961
26	13.991	vv	0.0586	1531.03662	367.18021	1.8057
27	14.095			7059.87549		
28	14.463			7911.69727		

Totals :

8.47871e4 6400.85505

Concentration of H₂SO₄= 0.09M



Signal 1: DAD1 A, Sig=270,16 Ref=360,100

#	RetTime [min]		Width [min]	Area [mAU*s]	Height [mAU]	90
1	1.779	BB	0.0483	7.91961	2.54432	0.0395
2	7.660	BB	0.1223	11.89918	1.35639	0.0594
3	8.086	BV	0.1376	13.46619	1.29211	0.0672
4	8.918	VB	0.1028	19.59771	2.75985	0.0978
5	9.508	BB	0.1513	24.00967	2.13260	0.1199
6	11.912	BV	0.2015	8245.38086	560.77905	41.1600
7	12.281	VV	0.0777	623.00391	110.64203	3.1100
8	12.372	VV	0.2260	1885.94043	106.82615	9.4144
9	12.723	VV	0.1925	1159.00269	76.61363	5.7856
10	13.145	VV	0.2461	2909.51245	157.10129	14.5239
11	13.688	VV	0.2798	2074.76660	98.47327	10.3570
12	13.999	VV	0.1263	749.31537	77.87499	3.7405
13	14.074	VB	0.3751	2308.70239	77.15457	11.5248

Totals :

2.00325e4 1275.55026

Concentration of H₂SO₄= 0.10M



Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]	[min]	[mAU*s]	[mAU]	%	#
1	1.750	VB	0.0494	9.51610	2.96572	0.0475
2	7.649	BB	0.1190	10.32407	1.19199	0.0511
3	8.080	BV	0.1477	12.46936	1.10464	0.0622
4	8.914	VB	0.0992	19.06817	2.80830	0.0951
5	11.914	BV	0.1947	8139.02393	569.77667	40.6046
6	12.290	VV	0.2936	2614.32422	108.30809	13.0426
7	12.727	VV	0.1014	576.36658	78.74627	2.8754
8	12.881	VV	0.1234	653.67297	75.19101	3.2611
9	13.150	VV	0.2618	2857.05981	143.77905	14.2535
10	13.685	VV	0.2792	2079.32617	98.93729	10.3735
11	14.005	VV	0.1268	754.57159	78.06480	3.7645
12	14.076	VB	0.3751	2318.85498	77.48942	11.5865
TOTALS				2.00446e4	1238.36325	
5						

Concentration of H₂SO₄= 0.20M







Peak	RetTime	Туре	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	1.614	BV	0.102	15.62298	2.46324	0.0754
2	1.724	VB	0.0445	237.21954	85.23232	1.1446
3	1.981	BB	0.1844	57.39159	4.63589	0.2769
4	3.457	BV	0.1502	57.76444	5.09582	0.2787
5	3.752	VB	0.1133	17.17017	2.14592	0.0828
6	6.166	BV	0.1026	101.87486	14.75283	0.4916
7	6.726	BV	0.0963	10.45899	1.64147	0.0505
8	6.862	VB	0.1057	8.48495	1.1286	0.0409
9	7.656	VB	0,1003	92.32351	13.41144	0.4455
10	8.086	BV	0.1249	102.32767	11.16679	0.4938
11	8.223	W	0.0963	44.5663	6.47535	0.215
12	8.356	W	0.0856	17.87307	2.99571	0.0862
13	8.631	W	0,1045	60,16069	8,3053	0.2903
14	8.923	VB	0.1	112.76742	16.45283	0.5441
15	9.513	BB	0.1017	17.74825	2.53501	0.0856
16	11.921	BV	0.2011	8171.56396	563,74139	39.4294
17	12.293	W	0.2916	2590.66064	108.08749	12,5004
18	12,729	W	0,1069	599.12756	76.90359	2.8909
19	12,885	W	0.107	554,12067	72.67582	2.6737
20	13.156	W	0.2639	2827.64038	141.02623	13.6439
21	13.691	W	0,2809	2063.82178	96.73959	9,9583
22	14.004	VB	0,4656	2963.8501	76.23892	14.3012
Totals				2.07E+04	1313.851	54

Calculation of the concentration of the Potassium

Since the HPLC equipment that we used can't show the result based on the components, we need to compare the retention time of the pure Potassium with other results of the fertilizer. Based on the standard solution that we analysed using HPLC equipment, it show that the retention time of the pure Potassium is at 13.13 min. Using the retention time of pure Potassium, we can find the area under the curve of Potassium in the fertilizer. The calculation is shown below.

For example,

Using Palm Kernel Shell with 0.08 Sulphuric acid and at 50°C, use the area under the curve at 13.13 min to get the concentration of Potassium:

Concentration of Potassium

2773.38281 72.2830 + 157.1718 + 343.0061 + 2135.92358 + 2773.38281 + 1609.32617 + 44599.3 = 0.0536 mg/L

Percentage of Potassium inside the fertilizer

 $= 0.0536 \times 100$ = 5.4%

Peak RetTime Type	Width	Area	Height	Area		
# [min]	[min]	[mAU*s]	[mAU]	90		
1 4.052 BB	0.1227	72.28306	8.37410	0.1398		
2 11.616 BV	0.2277	157.17183	8.66037	0.3041		
3 11.984 VV	0.2561	343.00610	16.94871	0.6636		
4 13.012 VV	0.2862	2135.92358	94.31099	4.1321		
5 13.135 VV	0.1299	2773.38281	289.15643	5.3654		
6 13.398 VV	0.1269	1609.32617	175.66751	3.1134		
7 13.891 VB	0.2488	4.45993e4	2670.41748	86.2816		
	_		\sim			
Totals :	Ļ	5.16904e4	3263.53559			
		,				
	Total area		Area und	ler the peak for		
Retention time based	under the peak			otassium		
on pure Potassium						
45						