

# **Impact of Fermentation Duration to Pyrolysis Process**

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# **CERTIFICATION OF APPROVAL**

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Approved by,

.....  
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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.

.....

(NURUL AIN BINTI R ASMAN)

## **ACKNOWLEDGEMENT**

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## **ABSTRACT**

Diminishing of fossil fuels cause the price of it becomes highly expensive and due to that it becomes less attractive to be the source of fuels especially in transportation sector. Thus most study is focus on searching the alternatives as the demands of it is also increasing day by day. The second generation of fuels production is biomass. However, the cost of raw biomass is also expensive that make the products also expensive. Now most researcher moving to third generation of fuels production that is lignocellulosic biomass. It is non value materials which is the residue of forestry has possibility to be one of the alternatives to the diminishing of fossil fuels. In this study,it will be discussed the lignocellulosic biomass, the conversion process of lignocellulosic biomass, pre treatment process and the modification on the pre treatment in order to produce low cost pre treatment and less time consuming.

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## CHAPTER 1

### INTRODUCTION

#### 1.1 BACKGROUND STUDY

Nowadays most research in chemistry, engineering, agriculture, and environmental mainly focus on clean technologies that operate on a sustainably produced feed stocks to the largest extent possible. Jean Paul Lange (2007) says that “governments across the world are stimulating the utilization of renewable energies and resources such as solar, wind, hydroelectricity and biomass.”

This study basically significant to transportation fuel sector which is strongly dependent on petroleum, the non renewable source of carbon. Furthermore, as worldwide supply of fossil fuels is diminishes the price is very expensive hence is becomes less attractive as carbon source. Biomass is one of the alternatives of sustainable feed stocks that can replace the diminishing fossil fuels for the production energy.

Based on the journal of Catalytic Conversion of Biomass to Bio-fuels by David Martin Alonso, Jesse Q. Bond and James A. Dumesic(2010)

The overall strategy in the production of hydrocarbon fuels from biomass is (i) to reduce the substantial oxygen content of the parent feed stock to improve energy density and (ii) to create C-C bonds between biomass derived intermediates to increase the molecular weight of the final hydrocarbon product.

There are three general classes of feed stocks derived from biomass which are starchy feed stocks (sugars), triglyceride feed stocks and lignocellulosic feed stocks. Dr. Eid MA MAGEED and Dr.Hisham FM ALY (2008) say that “out of these lignocellulosic materials are composed from carbohydrate polymers (cellulose and hemicelluloses) and the most abundant in the globe.”

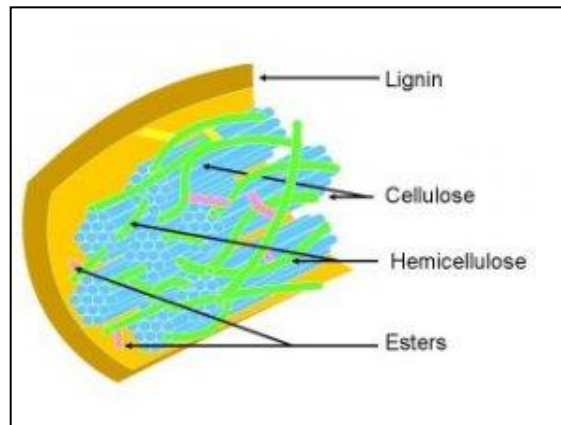


Figure 1

(Source: What is Lignocellulose ,2010)

Lignocellulosic biomass can be classified into four main categories which are agricultural residue, dedicated energy crops, forestry residues and municipal organic. However, from this four groups agricultural and forestry residue have major advantages as they have infrastructure in place for collection; transport and processing together with their abundance represent promising low cost feed stocks for the production of bio based chemicals.

## 1.2 PROBLEM STATEMENT

The uses of biomass as a renewable source of carbon for production of transportation fuels are promising alternative. There are three classes of biomass which are starchy feed stocks; triglyceride feed stocks and lignocellulosic feed stocks. Lignocellulosic biomass has potential to be one of the alternatives to replace the diminishing fuels due to its abundant represents and low cost feed stocks. Main challenges in converting the lignocellulosic biomass is the pre treatment process as it has very complex structure that makes it difficult to be access.

## 1.3 OBJECTIVE

Objective of this study is as follows:

- 1.3.1 To study the impact of fermentation duration towards pyrolysis process

**CHAPTER 2**

**LITERATURE REVIEW**

**2.1 LIGNOCELLULOSIC BIOMASS**

Lignocellulosic biomass is attractive as an inexpensive and most abundant feed stocks compared to two other biomass classification. Generally, it is consists of 40-50% of cellulose, 25-35% of hemicelluloses and 15-20% of lignin. Sun JX, Sun XF, Zhao H, Sun RC (2004) stated that “In lignocellulosic materials, cellulose is ordered into fibrils which are surrounded by a matrix and hemicelluloses.”

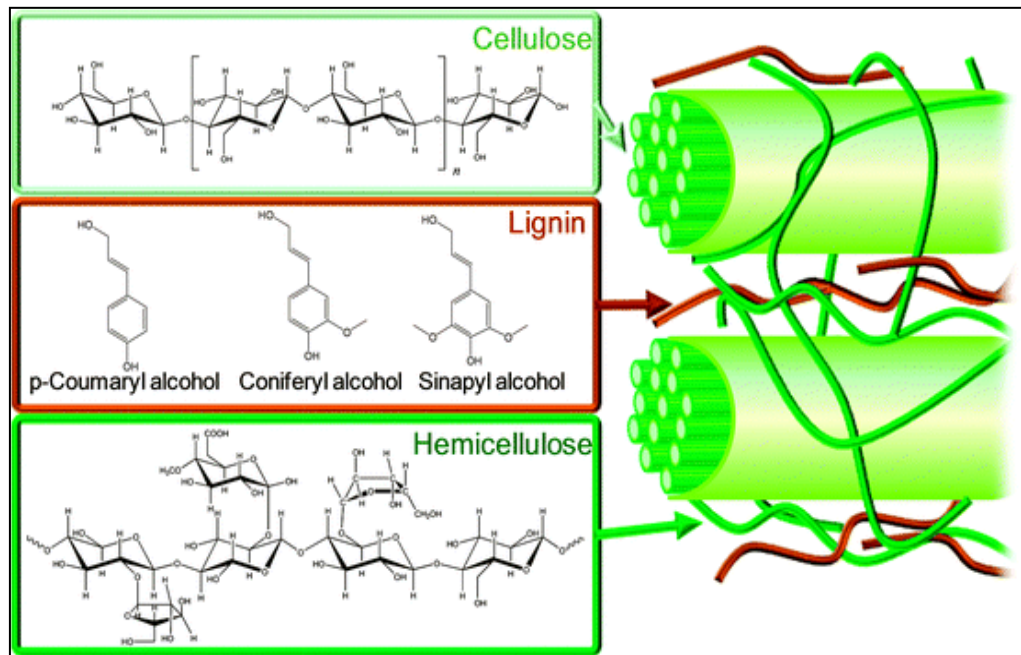


Figure 2

(Source: Bimetallic Catalyst for Upgrading of Biomass to fuels and Chemicals, 2012)

According to Jean Paul Lange (2007)

Lignocelluloses are much more difficult to convert than sugars, starches and oils. It is the fibrous material that forms the cell walls of the plants *architecture* that is consisted of three major components.

The components of lignocellulosic biomass can be summarized as follow:

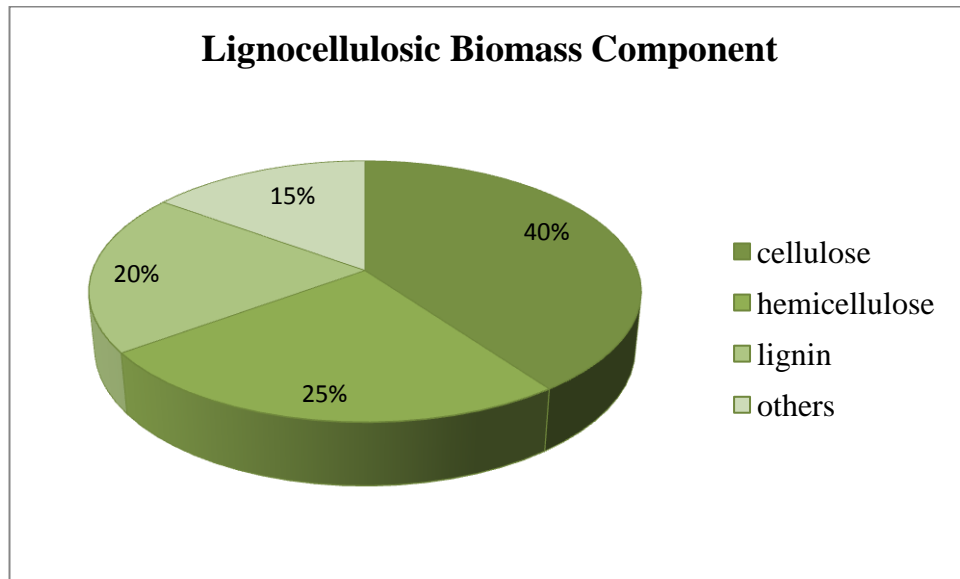


Figure 3

Most industrial processes have considered lignocellulosic biomass as a promising alternative due to its advantageous and by concerning current demands. Compared to other renewable sources, lignocellulosic biomass feed stocks cost will be less due to its abundance represents. Besides, it will not affect food supplies as lignocellulosic biomass often a waste material of food and forestry. Utilizing it is considered as clean technology as burning it with coal power plant will not add carbon to the environment that brings major environmental impact. Lucia (2008) says that “It is not feasible to use a non-lignocellulosic, corn, to make a significant dent in our national or global demands.”

Jean Paul Lange (2007) clearly state that

The world demand for energy is expected to double between years 2000 to 2050. Ways to overcome this is sought in the utilization of lignocellulosic materials such as residues from agriculture and forestry or energy crops.

However, lignocellulose is quiet difficult to be converted due to its fibrous materials that form the cell walls of the plants. Darryn W. Rackemann and William OS Doherty (2011) says that “As a result of the architectural of lignocellulosics, one of the challenges of our time is to develop cost effective fractionation procedures for the release of sugars from lignocellulosics.”

	Cellulose (wt%)	Hemicellulose (wt%)	Lignin (wt%)
Agricultural residues	35–55	25–35	15–30
Energy crops	30–50	20–40	10–20
Forestry residues	40–50	25–35	20–30

Table 1  
(Source: The conversion of Lignocellulosics to Levulinic Acid, 2011)

## 2.2 BIOMASS CONVERSION PROCESS

According to David Martin Alonso, Jesse Q. Bond and James A. Dumesic (2010),

The overall goal of converting lignocellulosic biomass to hydrocarbon fuels is the removal of oxygen, combined with the formation of C-C bonds to control the molecular weight of the final hydrocarbons, and requiring the least amount of hydrogen from an external source such as steam reforming of petroleum.

Klass DL (1998) says that “A variety of chemistries and process can be applied to convert lignocellulosic materials to valuable fuels and chemicals.”

There are two main strategies for conversion of lignocellulosic biomass to liquid biofuels. They are thermochemical platform which consists of gasification and pyrolysis and secondly is biochemical platform or also known as hydrolysis. Thermochemical are exploited in the pyrolysis to produce charcoal, oil and gases. Then, its gasification is to synthesis gas and hydrogen. Besides that, lignocelluloses can also be hydrolysed to liberate the lignin and depolymerised the polysaccharides to sugar. Basically all these conversion involve complex reactions of carbohydrate, the lignin and between carbohydrates and lignin.

According to Jean Paul Lange (2007)

Lignocellulose can be converted via three major routes:

- Pyrolysis to a complex and unstable oil with some gas and char
- Gasification and subsequent conversion to electricity or to liquid products such as alkanes or methanol
- Hydrolysis to sugars with subsequent transformation to fuels and chemicals via chemical conversion or fermentation.

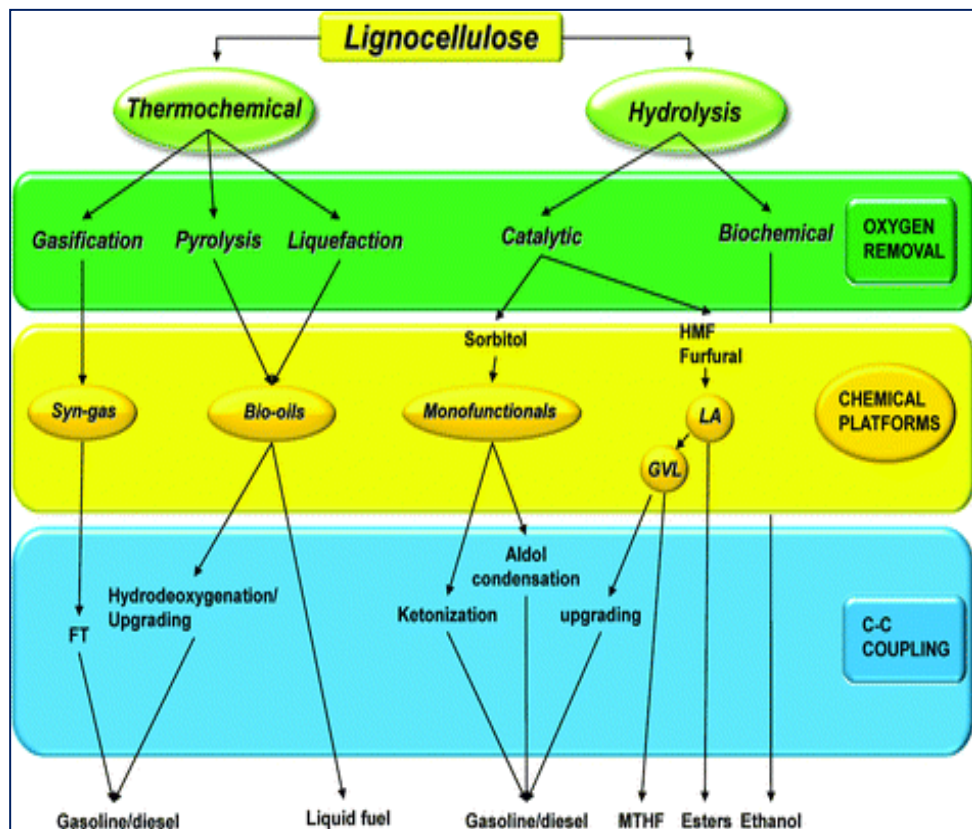


Figure 4

(Source: Catalytic Conversion of Biomass to Biofuels, 2010)

### 2.2.1 Biochemical (Hydrolysis) Platform

Thomas D. Foust, Andy Aden and Abhijit Dutta (2008) says that “Biochemical conversion routes rely on biocatalyst such as enzymes and microbial cells, in addition to heat and chemicals to convert biomass first to an intermediate mixed sugars streams and then to ethanol or other fermentation produced biofuel.”

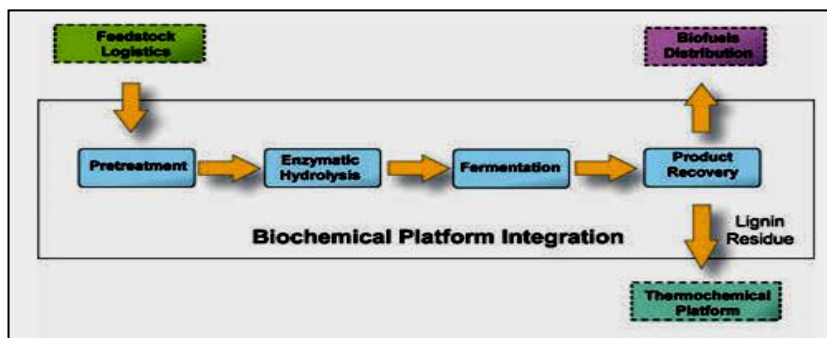


Figure 5  
(Source: Biochemical Conversion Process, 2008)

**2.2.2 Thermochemical Platform**

In the other hand, thermochemical platform depends on heat or catalyst to be converted to an intermediate gas or liquid. Then followed by conversion step of that intermediate to biofuel. Both gasification and pyrolysis require higher temperature in order to convert lignocelluloses to biofuel. However, compared to gasification pyrolysis might needed lower temperature.

According to Thermochemical Conversion (2012)

It is effectively applied to any biomass feed stocks and complement biochemical work by converting lignin rich non-fermeable material left over from high starch feed stocks conversion. It also will enhance fuel yields in integrated bio refineries by combining conversion types with heat and power efficiencies to produce fuel and products.

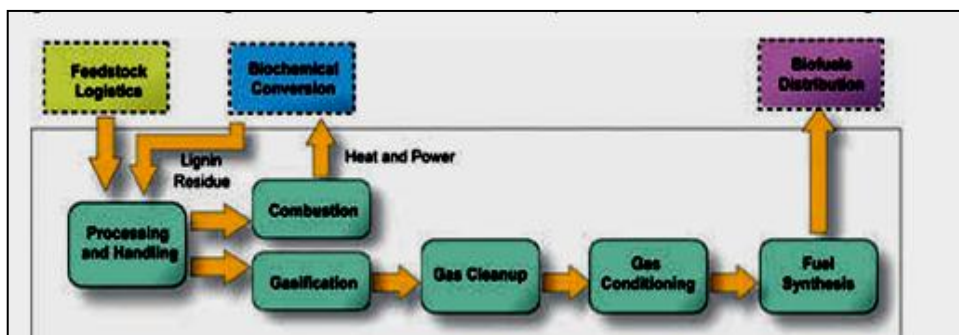


Figure 6  
(Source: Thermochemical Conversion Process, 2011)

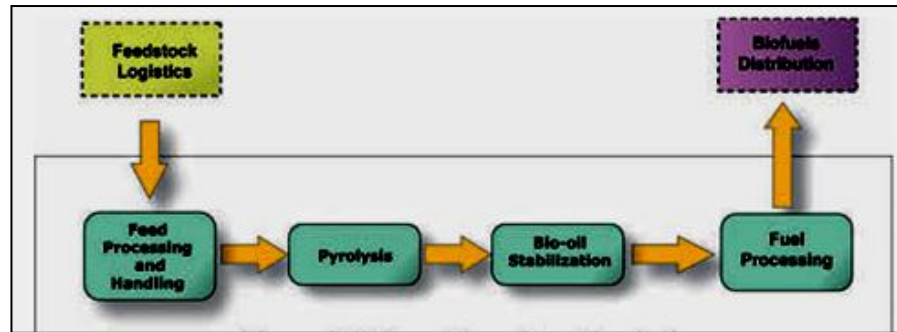


Figure 7

(Source: Thermochemical Conversion Process, 2011)



## 2.2.3 Comparison for All Methods

	Bio Chemical	Thermochemical	
		Gasification	Pyrolysis
Details of the Process	Appropriates for lignocelluloses process if higher selectivity is desired in biomass utilization.	Carried out through partial combustion of biomass to produce syngas. Then, it can be upgraded to liquid fuels such as diesel and gasoline by Fischer Tropsch (FT) synthesis.	Takes place through thermal, anaerobic decomposition of biomass.
Temperature		>1000K	650K- 800K
Pressure	Lower	Lower	Lower
Advantages	Decreased process time. Separate spontaneously the water so do not require distillation process to purify the final product.	Not constrained to a particular plant	Inexpensive and allow utilization of lignocellulosic biomass. Do not require extensive pre treatment steps.
Disadvantages	Complex process. Expensive on order to isolate the sugar monomers.	End products contains lot of water and impurities	End products not suit for transportation fuel as it is extensively corrosive

Table 2

After comparing, all the methods, pyrolysis process is the best choice for this study as it needs lower temperature instead of other methods. Besides it is much easier and simpler compared to other three methods.

For this study, the pyrolysis process is took place in Universiti Teknologi Mara (UiTM) Shah Alam and the pyrolysis reactor is customized by the university. Basically, the furnace will heat the temperature with the optimum temperature of pyrolysis process which is 773 K. Then, the gas will form and transferred to condenser to be condensed. The bio fuel is collected as the final product of the process.

Following is the flowchart of the pyrolysis process:

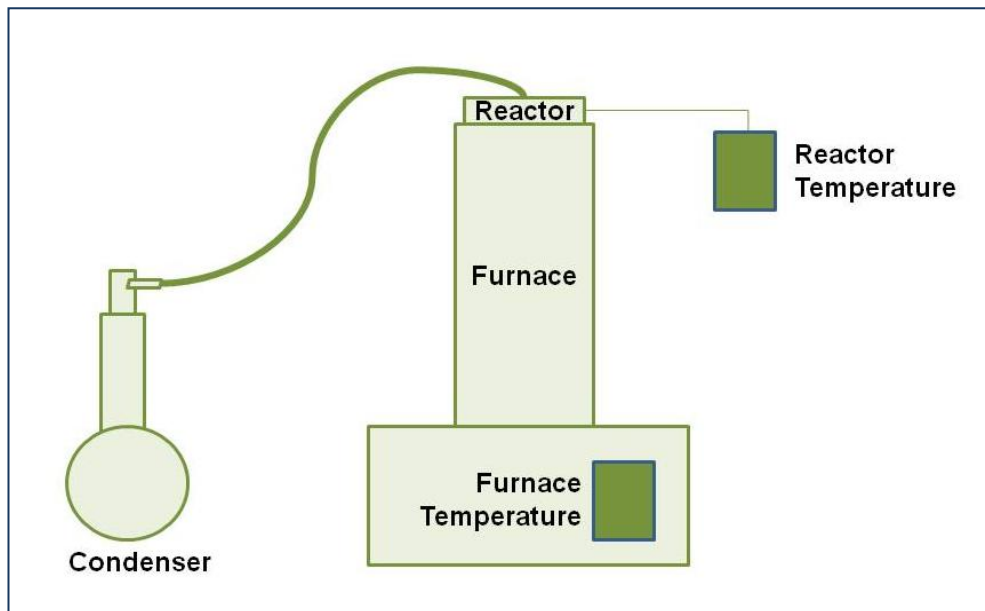


Figure 8

### 2.3 PRETREATMENT

Currently, many methods of pre treatment have been discovered in order to overcome biomass recalcitrance. Kamm B, Kamm M (2007) says that “Lignocellulose, which is the most abundant organic biomass in the natural world, has been thought as one of the most important feed stocks in future bio refinery to produce bio energy, bio based chemicals and biodegradable materials.” While in other articles by Claasen PAM, Van Lier JB, Contreas AML, Van Niel EWJ, Stijmsma L, Stams AJM et al, (1999) says that, “It has been estimated that worldwide

production of 10-50 billion tons of dry lignocellulosic biomass, accounting for about half of the global biomass yield.”

Even though it is quiet big demands, the biomass recalcitrance which is an outcome of the chemical composition and physical structure makes lignocellulosic biomass to be access to be broken down to its constituents. Thus, pre treatment is playing important role in order to achieve worldwide demands. Sticklen MB (2008) stated that “Plant cell walls are matrix of crosslinked polysaccharides networks, glycosylated proteins and lignin, in which cellulose mirofibrils are linked together with hemcelluloses and embedded with lignin, being like steel rods embedded in concrete.”

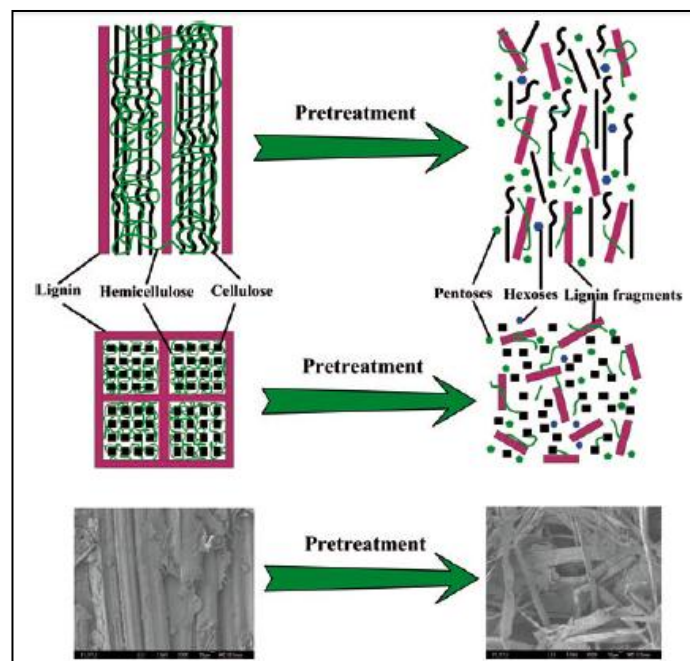


Figure 9

(Source: Lignins Grip on Biofuel production, 2007)

According to Xuebing Zhao, Lihua Zhang and Dehua Liu (2012)

The goals of pre treatments generally can be summarized as follows:

1. Remove and separate hemicelluloses from cellulose.
2. Disrupt and remove lignin sheath and/or redistribute he lignin components in cell wall.
3. Remove acetyl group for eliminating its interference with enzyme recognition to cellulose.

4. Decrease crystallinity and degree of polymerization of cellulose.
5. Increase the SSA of substrates by reducing particle size or increasing the porosity of the substrates.
6. Increase the porosity of substrates for facilitating the penetration of hydrolysis agent.

Basically, the ultimate aims of pre treatment is to make the cell wall of lignocellulosic biomass more accessible to the chemical or catalyst so that it can be broken down to its constituents thus can be convert to the bio fuel. Currently, the cost of converting lignocellulosic biomass is higher due to the first step of it which is to access the cell wall even though the cost of the feed stocks is the cheapest.

There are two main methods of pre treatment which are chemical pre treatment and biological pre treatment. For chemical pre treatment, various chemicals are used to pre treat the lignocelluloses while biological pre treatment is using microorganisms to increase enzymatic digestibility.

#### **2.4 BIOLOGICAL PRE TREATMENT**

Pre treatment is the first step of converting the lignocellulosic biomass as it is significantly improve the process of conversion and cut down the cost if the process can be done effectively. Xuebing Zhao, Lihua Zhang and Dehua Liu say that “Biological pre treatment refers to using some of microorganisms to pre treat biomass to increase enzymatic digestibility of remaining solids.”

Bio pre treatment also consider as cheap pre treatment as it does not have to use any additional equipment and chemicals. It also environmentally friendly green method.

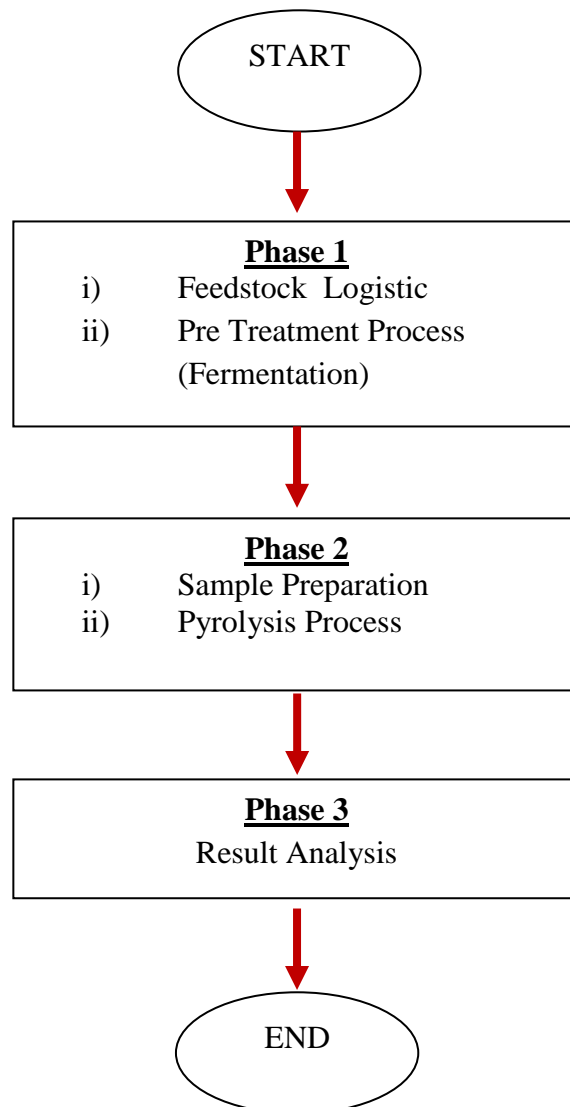
Building Blocks	Yeast or Fungal	Bacterial	Yeast or Fungal	Bacterial	Chemical & Catalytic Processes	Biotransformation
	AEROBIC FERMENTATIONS		ANAEROBIC FERMENTATIONS		CHEM-Enzyme TRANSFORMATIONS	
3 Carbon	Commercial Product - C	Commercial Product - C	Commercial Product - C	Commercial Product - C	Commercial Product - C	Commercial Product - C
3-Hydroxy propionic acid	X	X				
Glycerol	X	X	X	X	C	
Lactic acid	X		X	C		
Malonic acid	X				X	
Propionic acid				X		
Serine	X	C				C
4 Carbon						
3-Hydroxy butyrolactone					X	
Acetoin	X	X		X		
Aspartic Acid	X	X				X
Fumaric Acid	X	X			X	
Malic acid	X	X				
Succinic acid	X	X		X		X
Threonine	X	C				
5 Carbon						
Arabitol	X		X		C	X
Furfural					C	
Glutamic	X	C				
Itaconic Acid	C					
Levulinic acid					X	
Xylitol	X		X	X		C
8 Carbon						
2,5 Furan dicarboxylic acid					X	
Aconitic acid	X					
Citric acid	C					
Glucaric acid	X	X			X	
Gluconic acid	C	X				X
Levoglucozan					X	
Lysine	X	C				
Sorbitol	X	X			C	X
Number In each Pathway category*	21	14	4	6	11	7
Commercial processes	3	4	0	1	4	2

Table 3: Biological Pre treatment  
(Source: Top Value added Chemicals from biomass, 2004)

**CHAPTER 3**

**METHODOLOGY**

**3.1 FLOWCHART**



**3.2 METHODOLOGY OF THE EXPERIMENT**

**Phase 1**

**1. Feed stocks Logistic**

- Feed stock which is lignocellulosic biomass (oil palm residue) is selected and collected.
- The feed stocks then are dried for few days before going for fermentation process.



Figure 10: Oil Palm residue



Figure 11: Dried feedstock

**2. Pre Treatment**

- After that, the feed stocks then fermented. EM 1 microbes one of microbes available in market.
- Five samples have been collected from the fermentation process.

Sample	Duration
Sample 1	0 day
Sample 2	7 days
Sample 3	14 days
Sample 4	21 days
Sample 5	28 days

Table 4: Five samples

**Phase 2**

**1. Sample Preparation**

- All five samples have been dried in oven at temperature 103°C for a day to minimise the moisture in the samples.
- Then, all the samples have been grinded to small parts so that fasten the process of the pyrolysis.



Figure 12: Dried Samples



Figure 13: Grinded Samples

**2. Pyrolysis Process**

- Fixed Variable : Temperature (500°C)  
Sample Weight ( 20g)
- Manipulated Variable : Duration of fermentation ( five samples)
- Responding Variable : Duration for complete pyrolysis process

**Phase 3**

**1. Result Analysis**

The duration of individual pyrolysis without biological pre treatment and with biological pre treatments will be recorded and compared.

**3.3 GANTT CHART**

	ACTIVITY	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Feed Stock Logistic														
2	Fermentation Process														
3	Pyrolysis Process														
4	Sample Analysis														
5	Progress Report														
6	Pre SEDEX														
7	Final Report Draf														
8	Final Report Submission														



### 3.4 TOOLS

NO	EQUIPMENT/ HARDWARE	REMARKS
1	Feed stock Logistic	Forestry waste ( dried and cut into smaller pieces
2	Microbes	Yeast
3	Fermenter	Custom
4	Pyrolysis system	

**CHAPTER 4**

**RESULT AND DISCUSSION**

**4.1 RESULT**

**4.1.1 Characterization of Raw Material**

Based on APHA standard Calculation

Weight of Dried Samples, g	Weight of wet samples, g	Weight of samples, g	Total solid, %	Volatile Solid, %
50	100	20	50	60

Table 7

Where:

$$\text{Total Solid, \%} = \frac{\text{Weight of Dried Samples, g}}{\text{Weight of Wet Samples, g}} \times 100\%$$

$$\text{Volatile Solid, \%} = \frac{\text{Weight of Dried Samples, g} - \text{Weight of Samples, g}}{\text{Weight of Samples, g}} \times 100\%$$

**4.1.2 Pyrolysis Result**

Sample	Samples Amount, g	Temperature, °C	Duration, min	Yield amount, g	Yield Percentage, %
1	20	500	60	10	50
2	20	500	43	15	75
3	20	500	59	2.5	12.5
4	20	500	40	16	80
5	20	500	38	18	90

Table 8

Where, Yield Percentage, % =  $\frac{\text{Yield Amount, g}}{\text{Samples Amount, g}} \times 100\%$

Impact to Duration for complete process

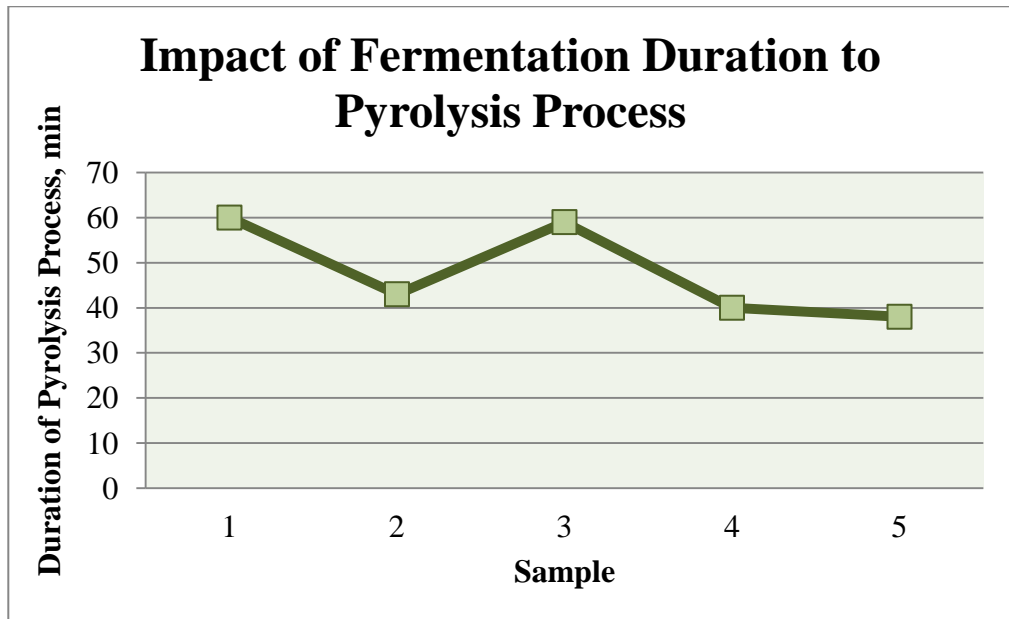


Figure 14

Impact to Yield Percentage

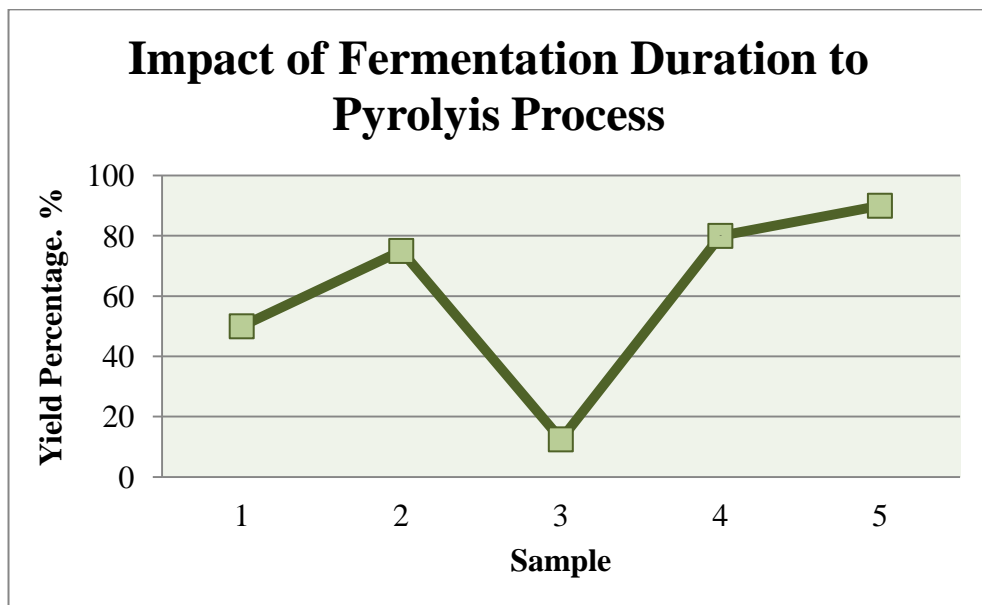


Figure 15

**4.2 DISCUSSION**

**4.2.1 Effect of Fermentation Duration**

Fixed Variable : Temperature (500°C)

Sample Weight (20g)

Manipulated Variable: Duration of fermentation (five samples)

Sample	Duration
Sample 1	0 day
Sample 2	7 days
Sample 3	14 days
Sample 4	21 days
Sample 5	28 days

Table 9

Responding Variable : Duration for complete pyrolysis process

Yield Percentage

Hypothesis:

- 1) The more the samples being fermented the faster the duration for pyrolysis process took place
- 2) The more the samples being fermented the higher the production yield percentage

Expected Result

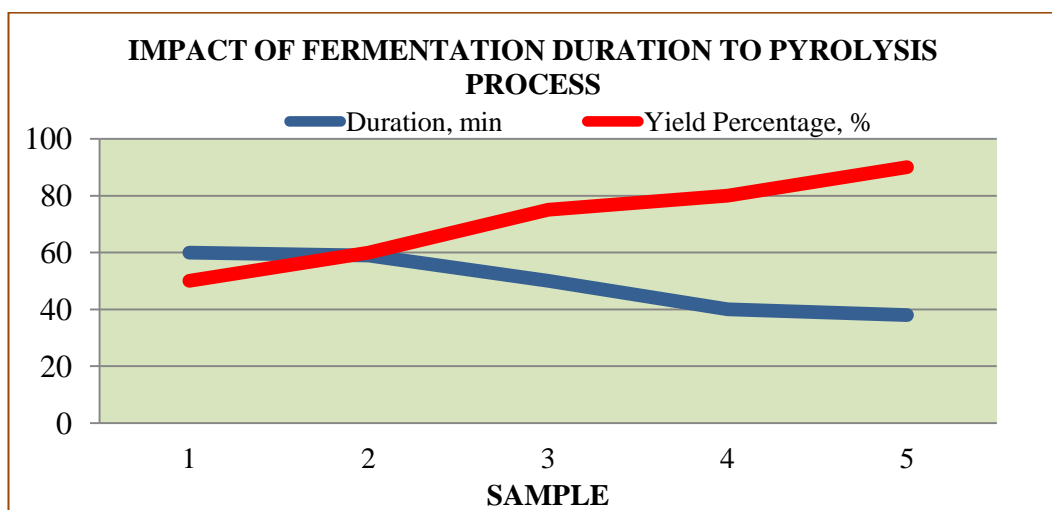


Figure 16

Based on the results obtained, the duration for completing pyrolysis process is decreasing as the fermentation duration is increasing except for Sample 3.

Fermentation is considered as biological pre treatment as it is using microorganism and no additional equipments and chemical is used. It will increase the enzymatic digestibility of the lignocellulosic biomass structure hence it is easier to be accessed.

According to Xuebing Zhao, Lihua Zhang and Dehua Lu (2012),

The employed micro organisms are usually capable of degrading lignin and carbohydrate polymer.

Basically, fermentation employs micro organism to break the cell wall which is consequently made the lignocellulosic biomass easy to be accessed for further conversion to bio fuel. These statements support the hypothesis that has been made which is fermentation is affected the duration of pyrolysis process. The more fermentation duration, duration of pyrolysis process is reduced.

However, due to some error while conducting the experiment the result turns to be slightly different from the hypothesis statement. Sample 3 takes longer time from expectation result. It might be due to the error while sample preparation. For better result in the future, the experiment needs to be repetitive so that the result acquire more precise.

In terms of production yield, the more fermentation duration the higher the production yield except for sample 3. As been mentioned above the fermentation process will increase the enzymatic digestibility of the structure. Therefore, the structure is easier to be accessed. Consequently, more yields is produced once the duration of fermentation is increasing.

**4.2.3 Characterization of Feed Stock**

Pyrolysis process is a thermal decomposition with absence of oxygen or air in order to produce bio fuel.

Below are the elemental analysis for the samples that has been made by Mohamad Azri Sukiran, Loh Soh Kheang, Nasrin Abu Bakar and Choo Yuen May (2011)

Characteristics	
Proximate analysis (wt%)	
Volatiles	81.9
Fixed Carbon	12.6
Ash	3.1
Moisture	2.4
Ultimate analysis (wt%)	

Table 10

(Source: Production and Characterization of Bio char from Pyrolysis Process, 2011)

**4.2.4 Default Feeds Stock of Yield Production**

In pyrolysis process, bio fuel is produced by controlling oxygen feed. Basically, conversion of lignocellulosic biomass to bio fuel is still pilot plan and need further investigation for most impactful product.

Below is the extract of possible yield produced by lignocellulosic biomass to bio fuel.

ELEMENT	EFB (%)	TRUNK	FRONDS
C	53.78	40.64	52.28
H	4.37	5.09	
O	41.5	53.12	
N	0.35	2.15	0.75
S	-	-	
CV(MJ/KG)	17.08	17.27	

Table 11

(Source: Biomass in Asia, 2008)

Below are the summarize of the elements of the products.

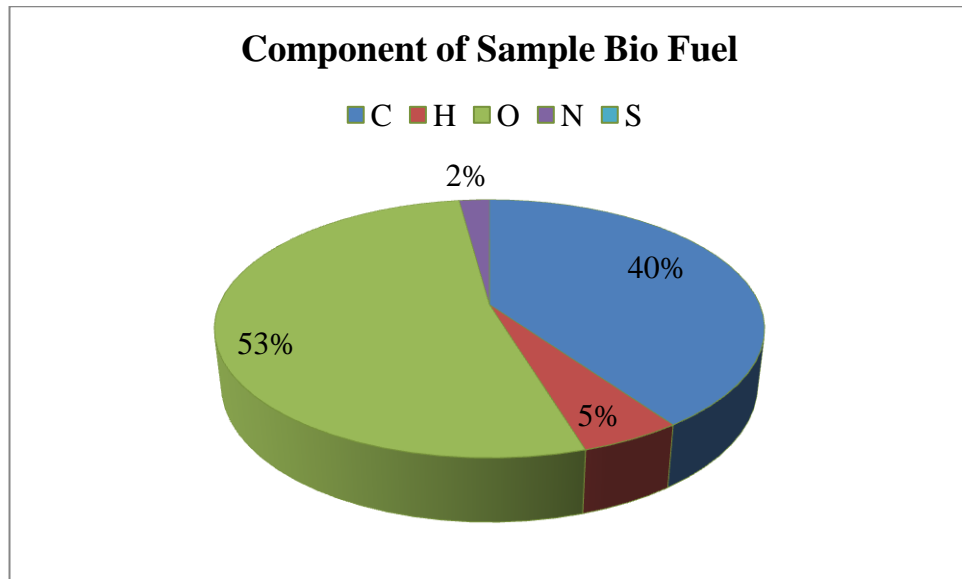


Figure 17

## CHAPTER 5

### CONCLUSION AND RECOMMENDATION

#### 5.1 CONCLUSION

Pre treatment is very important process in converting lignocellulosic biomass. Biological pre treatment is one of the methods of the pre treatment. It is very cheap compared to other pre treatment as it does not need any additional chemical and equipments. Besides it is very environmentally friendly green methods. If previously fermentation took place after the chemical pre treatment, in this study the fermentation will come first as pre treatment before converting to bio fuel.

#### 5.2 RECOMMENDATION

Lignocellulosic biomass is very significant feed stock in order to replace the diminishing of petroleum as currently being the primary sources for the fuel production. Therefore:

- i. Further study need to be done on the possible pre treatment for the lignocellulosic biomass so that the cost for converting it to bio fuel is more efficient.
- ii. Fermentation is one of the pre treatments that need to be seriously taken into consideration as the methods are very easy and cheaper as no additional chemical and equipment are needed. Besides, it is very friendly and green technology. Thus, careful studies on these methods need to be done.
- iii. Due to time constraint and technical error, the analysis of the products produced cannot be done. Hence, for more efficient investigation further study on the products need to be done and if possible further converted to fuel transportation



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## **APPENDICES**

APPENDIX I

**Table 1. Effects of different pre-treatments on chemical compositions and physical structures of lignocellulosic biomass.**

Physical	Chemical compositions				Physical structures			Main references
	Cellulose	Hemicelluloses	Lignin	Acetyl group	Accessible surface area (ASA)	Cellulose crystallinity	Cellulose DP	
Ball milling	No degradation	No degradation	No removal	No removal	Size reduction, Significant increase of ASA <sup>1</sup>	Significant decrease <sup>2</sup>	Significant decrease <sup>2</sup>	21, 22, 26
High energy radiolysis	Some degradation <sup>1</sup>	Significant degradation <sup>1</sup>	Partial removal and structure change <sup>1</sup>	Partial removal	Small cart increase of ASA <sup>1</sup>	Some decrease <sup>1</sup>	Some decrease <sup>1</sup>	7, 65
Steam explosion	Little degradation	Significant degradation <sup>1</sup>	Partial removal, significant structure change and radiolysis <sup>1</sup>	Significant removal	Significant increase of ASA <sup>1</sup>	NA	Some decrease <sup>1</sup>	21, 24, 26
AFEX	Little degradation	Partial degradation <sup>1</sup>	Partial removal, significant structure change <sup>1</sup>	Significant removal	Significant increase of ASA <sup>1</sup>	Some decrease <sup>1</sup>	Little decrease <sup>1</sup>	16, 43, 46
Liquid hot water	Some degradation <sup>1</sup>	Significant degradation <sup>1</sup>	Partial removal, significant structure change <sup>1</sup>	Significant removal	Significant increase of ASA <sup>1</sup>	Some decrease <sup>1</sup>	Some decrease <sup>1</sup>	8, 14, 47
Chemical	Some degradation <sup>1</sup>	Nearly complete degradation <sup>1</sup>	Partial removal, significant structure change and radiolysis <sup>1</sup>	Complete removal	Significant increase of ASA <sup>1</sup>	Some decrease <sup>1</sup>	Some decrease <sup>1</sup>	9, 49, 60
H <sub>2</sub> O <sub>2</sub>	Some degradation <sup>1</sup>	Partial degradation <sup>1</sup>	Significant removal and structure change <sup>1</sup>	Complete removal	Significant increase of ASA <sup>1</sup>	None decrease <sup>1</sup>	Some decrease <sup>1</sup>	6, 26
ARP	Some degradation <sup>1</sup>	Partial degradation <sup>1</sup>	Significant removal and structure change <sup>1</sup>	Complete removal	Significant increase of ASA <sup>1</sup>	None decrease <sup>1</sup>	Some decrease <sup>1</sup>	9, 10, 100
Lime	Little degradation <sup>1</sup>	Partial degradation <sup>1</sup>	Significant removal and structure change <sup>1</sup>	Complete removal	Significant increase of ASA <sup>1</sup>	None decrease <sup>1</sup>	Some decrease <sup>1</sup>	8, 25, 106
Oxidative degradation	Little degradation	Partial degradation <sup>1</sup>	Significant removal and structure change <sup>1</sup>	Partial removal	Significant increase of ASA <sup>1</sup>	None decrease <sup>1</sup>	Some decrease <sup>1</sup>	10, 27, 103
Organic solvents (non-organic acids)	Partial degradation <sup>1</sup>	Nearly complete degradation <sup>1</sup>	Significant removal and structure change <sup>1</sup>	Complete removal	Significant increase of ASA <sup>1</sup>	None decrease <sup>1</sup>	Some decrease <sup>1</sup>	14, 26
Organic solvents (organic acids)	Partial degradation <sup>1</sup>	Nearly complete degradation <sup>1</sup>	Significant removal and structure change <sup>1</sup>	Increase <sup>1</sup>	Significant increase of ASA <sup>1</sup>	Increase of decrease <sup>1</sup>	Some decrease <sup>1</sup>	14
Ionic liquids	Little degradation	Partial degradation <sup>1</sup>	Partial removal <sup>1</sup>	NA	Small cart increase of ASA <sup>1</sup>	Converted to amorphous <sup>2</sup>	Little decrease <sup>1</sup>	7, 127, 160
Concentrated phosphoric acid	Little degradation	Significant degradation <sup>1</sup>	Significant removal and structure change <sup>1</sup>	Complete removal	Significant increase of ASA <sup>1</sup>	Converted to amorphous <sup>2</sup>	Some decrease <sup>1</sup>	140, 150
SPCBL	Little degradation	Significant degradation <sup>1</sup>	Partial removal, significant structure change <sup>1</sup>	Significant removal	Significant increase of ASA <sup>1</sup>	NA	Some decrease <sup>1</sup>	110, 127, 167
Biological	Some degradation	Partial degradation <sup>1</sup>	Partial removal <sup>1</sup>	NA	Increase of ASA <sup>1</sup>	NA	Some decrease <sup>1</sup>	26, 104, 107, 164

Notes: NA: not affected; 1: increasing the enzymatic digestibility of cellulose; 2: decreasing the enzymatic digestibility of cellulose; 3: depending on pre-treatment conditions; NA data is not available.

**APPENDIX II**



**APPENDIX III**



**APPENDIX IV**



**APPENDIX V**



**APPENDIX VI**



APPENDIX VII



UNIVERSITI  
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PETRONAS

**IMPACT OF FERMENTATION DURATION TO PYROLYSIS PROCESS**

By:

**NURULAIN BINTI RASMAN ID NO.: 12159**  
**SUPERVISOR : PUAN ASNA BINTI MOHD ZAIN**

**PROBLEM STATEMENT**

Lignocellulosic biomass is a worldwide demand due to its possibility to replace the diminishing petroleum. However, one of the challenges is the pre-treatment process as it has a very complex structure.

**OBJECTIVE**

The objective of this study is:  
• To study the impact of duration fermentation (pre treatment) to the pyrolysis process.

**METHODOLOGY**

**PHASE 1**

- 1. Feed Stock Logistic**
  - Feed stock are collected then dried for few days.
- 2. Fermentation Process**
  - Dried feed stock then being fermented with microbes.

Sample	Duration
Sample 1	0 day
Sample 2	7 days
Sample 3	14 days
Sample 4	21 days
Sample 5	28 days



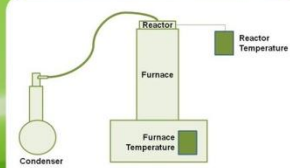
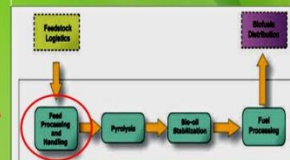
**PHASE 2**

- 1. Sample Preparation**
  - All five samples are dried in oven for a day.
  - Next, the samples are grinded.
- 2. Pyrolysis Process**
  - Fixed parameter
    - i) Temperature: 500°C
    - ii) Weight : 20 g
  - Manipulated parameter: Samples
  - Responding parameter: Duration for complete process



**PHASE 3**

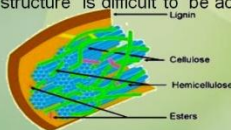
**1. Result Analysis**



Simple flowchart of pyrolysis process

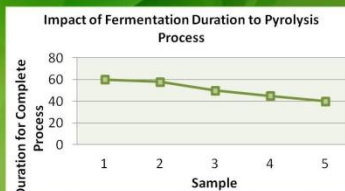
**LITERATURE REVIEW**

- Lignocellulosic biomass is a potential feed stocks to replace the diminishes of petroleum due to its abundance represent low cost production for bio based chemicals.
- The main concern of converting linocellulosic biomass is the structure is difficult to be accessed.



- Fermentation as one of the biological pre treatment which is using microbes to break the structure to the constituents

**RESULTS & DISCUSSION**



- The more the samples being fermented the faster pyrolysis process.
- It is because microbes help to ingest the wall of the residue before the pyrolysis took place. Thus, the process becomes less complicated and easier to produce the bio fuel.

**CONCLUSION**

- Pre treatment is very important process in converting lignocellulosic biomass. compared to other pre treatment as it does not need any additional chemical and equipments Biological pre treatment is one of the methods of the pre treatment. It is very cheap. Besides it is very environmentally friendly green methods
- The duration of fermentation do affected the pyrolysis process.



**APPENDIX VIII**

# Impact of Fermentation Duration to Pyrolysis Process

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**Abstract-** Diminishing of fossil fuels cause the price of it becomes highly expensive and due to that it becomes less attractive to be the source of fuels especially in transportation sector. Thus most study is focus on searching the alternatives as the demands of it is also increasing day by day. The second generation of fuels production is biomass. However, the cost of raw biomass is also expensive that make the products also expensive. Now most researcher moving to third generation of fuels production that is lignocellulosic biomass. It is non value materials which is the residue of forestry has possibility to be one of the alternatives to the diminishing of fossil fuels. In this study, it will be discussed the lignocellulosic biomass, the conversion process of lignocellulosic biomass, pre treatment process and the modification on the pre treatment in order to produce low cost pre treatment and less time consuming.

Most industrial processes have considered lignocellulosic biomass as a promising alternative due to its advantageous and by concerning current demands. Compared to other renewable sources, lignocellulosic biomass feed stocks cost will be less due to its abundance represents. Besides, it will not affect food supplies as lignocellulosic biomass often a waste material of food and forestry. Utilizing it is considered as clean technology as burning it with coal power plant will not add carbon to the environment that brings major environmental impact. Lucia (2008) says that "It is not feasible to use a non-lignocellulosic, corn, to make a significant dent in our national or global demands."

## I. Introduction

### A. Literature review

#### Lignocellulosic Biomass

Lignocellulosic biomass is attractive as an inexpensive and most abundant feed stocks compared to two other biomass classification. Generally, it consists of 40-50% of cellulose, 25-35% of hemicelluloses and 15-20% of lignin. Sun JX, Sun XF, Zhao H, Sun RC (2004) stated that "In lignocellulosic materials, cellulose is ordered into fibrils which are surrounded by a matrix and hemicelluloses."

According to Jean Paul Lange (2007)

Lignocelluloses are much more difficult to convert than sugars, starches and oils. It is the fibrous material that forms the cell walls of the plants *architecture* that is consisted of three major components.

The components of lignocellulosic biomass can be summarized as follow:

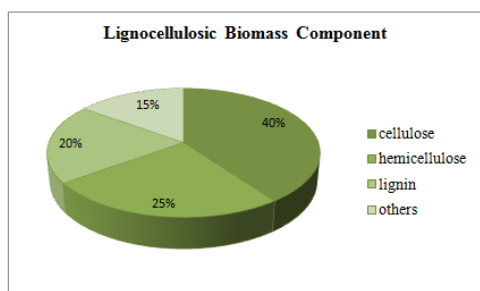


Figure 1

According to Jean Paul Lange (2007) clearly state that

The world demand for energy is expected to double between years 2000 to 2050. Ways to overcome this is sought in the utilization of lignocellulosic materials such as residues from agriculture and forestry or energy crops.

However, lignocellulose is quiet difficult to be converted due to its fibrous materials that form the cell walls of the plants. Darryn W. Rackemann and William OS Doherty (2011) says that "As a result of the architectural of lignocellulosics, one of the challenges of our time is to develop cost effective fractionation procedures for the release of sugars from lignocellulosics."

	Cellulose (wt%)	Hemicellulose (wt%)	Lignin (wt%)
Agricultural residues	35-55	25-35	15-30
Energy crops	30-50	20-40	10-20
Forestry residues	40-50	25-35	20-30

(Source: The Conversion of Lignocellulosic to Levulinic Acid, 2011)

#### Biomass Conversion Process

According to David Martin Alonso, Jesse Q. Bond and James A. Dumesic (2010),

The overall goal of converting lignocellulosic biomass to hydrocarbon fuels is the removal of oxygen, combined with the formation of C-C bonds to control the molecular weight of the final hydrocarbons, and requiring the least amount of hydrogen from an external source such as steam reforming of petroleum.

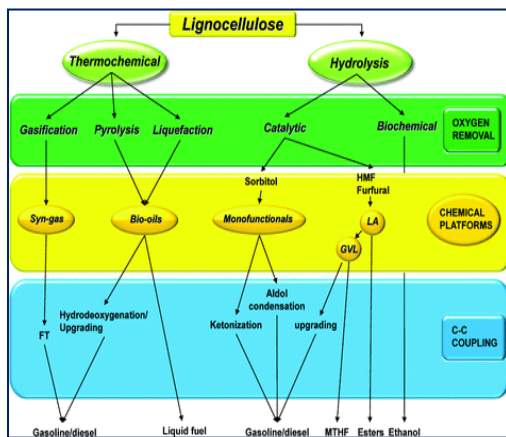
Klass DL (1998) says that “A variety of chemistries and process can be applied to convert lignocellulosic materials to valuable fuels and chemicals.”

There are two main strategies for conversion of lignocellulosic biomass to liquid biofuels. They are thermochemical platform which consists of gasification and pyrolysis and secondly is biochemical platform or also known as hydrolysis. Thermochemical are exploited in the pyrolysis to produce charcoal, oil and gases. Then, its gasification is to synthesis gas and hydrogen. Besides that, lignocelluloses can also be hydrolysed to liberate the lignin and depolymerised the polysaccharides to sugar. Basically all these conversion involve complex reactions of carbohydrate, the lignin and between carbohydrates and lignin.

According to Jean Paul Lange (2007)

Lignocellulose can be converted via three major routes:

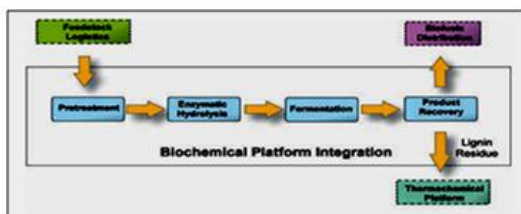
- Pyrolysis to a complex and unstable oil with some gas and char
- Gasification and subsequent conversion to electricity or to liquid products such as alkanes or methanol
- Hydrolysis to sugars with subsequent transformation to fuels and chemicals via chemical conversion or fermentation.



(Source: Conversion of Biomass to Bio fuel,2010)

### Biochemical (Hydrolysis) Platform

Thomas D. Foust, Andy Aden and Abhijit Dutta (2008) says that “Biochemical conversion routes rely on biocatalyst such as enzymes and microbial cells, in addition to heat and chemicals to convert biomass first to an intermediate mixed sugars streams and then to ethanol or other fermentation produced biofuel.”



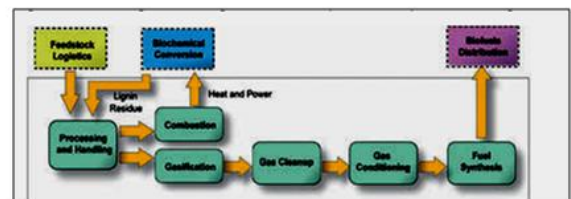
(Source: Biochemical Conversion Process, 2008)

### Thermochemical Platform

In the other hand, thermochemical platform depends on heat or catalyst to be converted to an intermediate gas or liquid. Then followed by conversion step of that intermediate to biofuel. Both gasification and pyrolysis require higher temperature in order to convert lignocelluloses to biofuel. However, compared to gasification pyrolysis might needed lower temperature.

According to Thermochemical Conversion (2012)

It is effectively applied to any biomass feed stocks and complement biochemical work by converting lignin rich non-fermentable material left over from high starch feed stocks conversion. It also will enhance fuel yields in integrated bio refineries by combining conversion types with heat and power efficiencies to produce fuel and products.



(Source: Thermochemical Conversion Process, 2011)



(Source: Thermochemical Conversion Process, 2011)

After comparing, all the methods, pyrolysis process is the best choice for this study as it needs lower temperature instead of other methods. Besides it is much easier and simpler compared to other three methods.

For this study, the pyrolysis process is took place in Universiti Teknologi Mara (UiTM) Shah Alam and the pyrolysis reactor is customized by the university. Basically, the furnace will heat the temperature with the optimum temperature of pyrolysis process which is 773 K. Then, the gas will form and transferred to condenser to be condensed. The bio fuel is collected as the final product of the process Following us the process flowchart for pyrolysis.

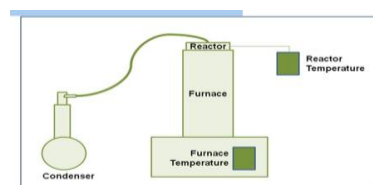
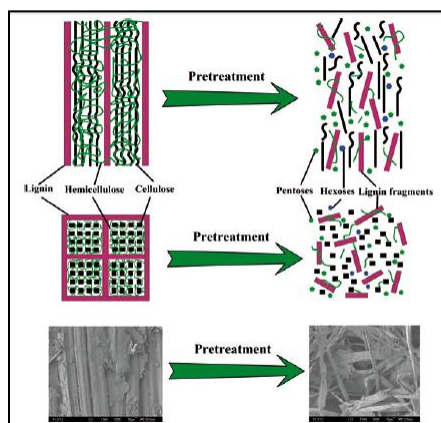


Figure 5

## Pretreatment

Currently, many methods of pre treatment have been discovered in order to overcome biomass recalcitrance. Kamm B, Kamm M (2007) says that “Lignocellulose, which is the most abundant organic biomass in the natural world, has been thought as one of the most important feed stocks in future bio refinery to produce bio energy, bio based chemicals and biodegradable materials.” While in other articles by Claasen PAM, Van Lier JB, Contreas AML, Van Niel EWJ, Stijnsma L, Stams AJM et al, (1999) says that, “It has been estimated that worldwide production of 10-50 billion tons of dry lignocellulosic biomass, accounting for about half of the global biomass yield.”

Even though it is quiet big demands, the biomass recalcitrance which is an outcome of the chemical composition and physical structure makes lignocellulosic biomass to be access to be broken down to its constituents. Thus, pre treatment is playing important role in order to achieve worldwide demands. Sticklen MB (2008) stated that “Plant cell walls are matrix of crosslinked polysaccharides networks, glycosylated proteins and lignin, in which cellulose microfibrils are linked together with hemicelluloses and embedded with lignin, being like steel rods embedded in concrete.”



(Source: Lignin Grip of Biofuel Production, 2007)

According to Xuebing Zhao, Lihua Zhang and Dehua Liu (2012)

The goals of pre treatments generally can be summarized as follows:

1. Remove and separate hemicelluloses from cellulose.
2. Disrupt and remove lignin sheath and/or redistribute the lignin components in cell wall.
3. Remove acetyl group for eliminating its interference with enzyme recognition to cellulose.
4. Decrease crystallinity and degree of polymerization of cellulose.
5. Increase the SSA of substrates by reducing particle size or increasing the porosity of the substrates.
6. Increase the porosity of substrates for facilitating the penetration of hydrolysis agent.

Basically, the ultimate aims of pre treatment is to make the cell wall of lignocellulosic biomass more accessible to the chemical or catalyst so that it can be broken down to its constituents thus can be convert to the bio fuel. Currently, the cost of converting lignocellulosic biomass is higher due to the first step of it which is to access the cell wall even though the cost of the feed stocks is the cheapest.

There are two main methods of pre treatment which are chemical pre treatment and biological pre treatment. For chemical pre treatment, various chemicals are used to pre treat the lignocelluloses while biological pre treatment is using microorganisms to increase enzymatic digestibility.

## Biological Pre Treatment

Pre treatment is the first step of converting the lignocellulosic biomass as it is significantly improve the process of conversion and cut down the cost if the process can be done effectively. Xuebing Zhao, Lihua Zhang and Dehua Liu say that “Biological pre treatment refers to using some of microorganisms to pre treat biomass to increase enzymatic digestibility of remaining solids.”

Bio pre treatment also consider as cheap pre treatment as it does not have to use any additional equipment and chemicals. It also environmentally friendly green method.

## B. Project Background

Nowadays most research in chemistry, engineering, agriculture, and environmental mainly focus on clean technologies that operate on a sustainably produced feed stocks to the largest extent possible. Jean Paul Lange (2007) says that “governments across the world are stimulating the utilization of renewable energies and resources such as solar, wind, hydroelectricity and biomass.”

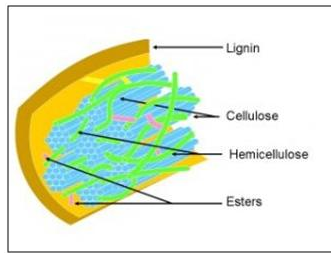
This study basically significant to transportation fuel sector which is strongly dependent on petroleum, the non renewable source of carbon. Furthermore, as worldwide supply of fossil fuels is diminishes the price is very expensive hence is becomes less attractive as carbon source. Biomass is one of the alternatives of sustainable feed stocks that can replace the diminishing fossil fuels for the production energy.

Based on the journal of Catalytic Conversion of Biomass to Bio-fuels by David Martin Alonso, Jesse Q. Bond and James A. Dumesic(2010)

The overall strategy in the production of hydrocarbon fuels from biomass is (i) to reduce the substantial oxygen content of the parent feed stock to improve energy density and (ii) to create C-C bonds between biomass derived intermediates to increase the molecular weight of the final hydrocarbon product.

There are three general classes of feed stocks derived from biomass which are starchy feed stocks (sugars), triglyceride feed stocks and lignocellulosic feed stocks. Dr. Eid MA

MAGEED and Dr.Hisham FM ALY (2008) say that “out of these lignocellulosic materials are composed from carbohydrate polymers (cellulose and hemicelluloses) and the most abundant in the globe.”



(Source: What it is Lignocellulose ,2011)

Lignocellulosic biomass can be classified into four main categories which are agricultural residue, dedicated energy crops, forestry residues and municipal organic. However, from this four groups agricultural and forestry residue have major advantages as they have infrastructure in place for collection; transport and processing together with their abundance represent promising low cost feed stocks for the production of bio based chemicals

### C. Problem statement

The uses of biomass as a renewable source of carbon for production of transportation fuels are promising alternative. There are three classes of biomass which are starchy feed stocks; triglyceride feed stocks and lignocellulosic feed stocks. Lignocellulosic biomass has potential to be one of the alternatives to replace the diminishing fuels due to its abundant represents and low cost feed stocks. Main challenges in converting the lignocellulosic biomass is the pre treatment process as it has very complex structure that makes it difficult to be access.

### D. Objectives of project

Objectives of this study are as follows:

1. To study the impact of fermentation process which is biological pre treatment towards converting lignocellulosic biomass to biofuel?
2. To investigate the impact of fermentation duration to pyrolysis process.

## II. EXPERIMENTAL METHODOLOGY

### Phase 1

#### Feed stocks Logistic

- 1) Feed stock which is lignocellulosic biomass (oil palm residue) is selected and collected.
- 2) The feed stocks then are dried for few days before going for fermentation process.

#### Pre Treatment

- 1) After that, the feed stocks then fermented.

- 2) Five samples have been collected from the fermentation process.

### Phase 2

#### Sample Preparation

- 1) All five samples have been dried in oven at temperature 103°C for a day to minimise the moisture in the samples.

Sample	Duration
Sample 1	0 day
Sample 2	7 days
Sample 3	14 days
Sample 4	21 days
Sample 5	28 days

Table 2

- 2) Then, all the samples have been grinded to small parts so that fasten the process of the pyrolysis.

#### Pyrolysis Process

Fixed Variable : Temperature (500°C)  
Weight ( 20g)

Manipulated Variable: Duration of fermentation (five samples)

Responding Variable: Duration for complete pyrolysis process

## III. RESULTS AND DISCUSSIONS

### A. Result

#### Characterization of Raw Material

Weight of Dried Samples, g	Weight of wet samples, g	Weight of samples, g	Total solid, %	Volatile Solid, %
50	100	20	50	60

Where:

$$\text{Total Solid, \%} = \frac{\text{Weight of Dried Samples,g}}{\text{Weight of Wet Samples,g}} \times 100\%$$

Volatile Solid, %=

$$\frac{\text{Weight of Dried Samples,g}-\text{Weight if Samples,g}}{\text{Weight of Samples,g}} \times 100\%$$

#### Pyrolysis Result

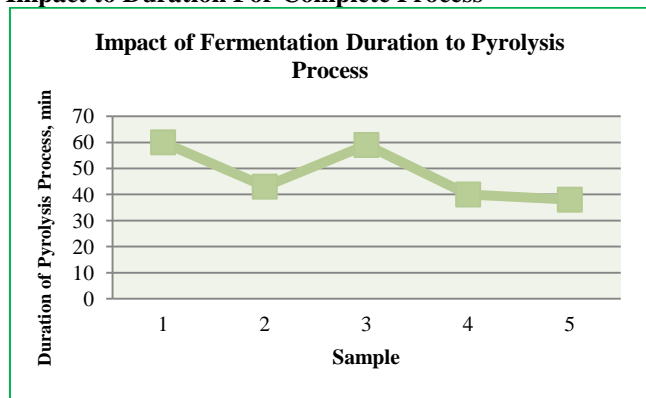
Sample	Amount before process, g	Temperature, °C	Duration, min	Amount After Process, g	Yield Percent age, %
1	20	500	60	10	50
2	20	500	43	15	75
3	20	500	59	2.5	12.5

4	20	500	40	16	80
5	20	500	38	18	90

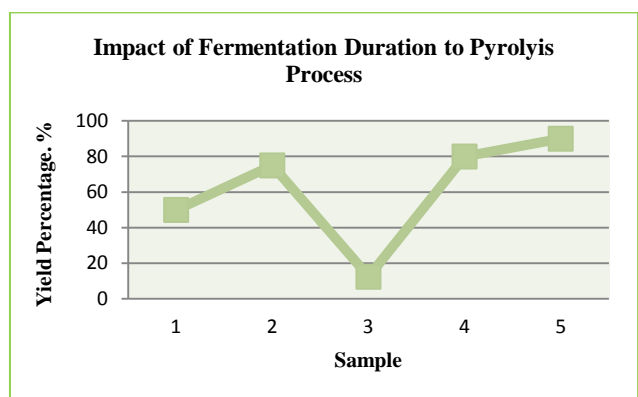
Table 7

Where, Yield Percentage, % =  $\frac{\text{Yield Amount, g}}{\text{Samples Amount, g}} \times 100\%$

### Impact to Duration For Complete Process



### Impact to Yield Percentage



## B. Discussion

### Effect of Fermentation Duration

Fixed Variable : Temperature (500°C)

Sample Weight (20g)

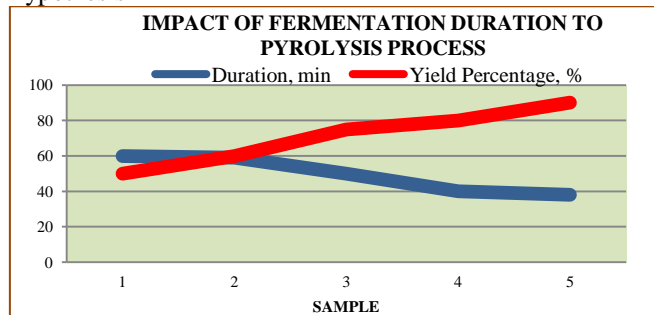
Manipulated Variable: Duration of fermentation

(five samples)

Sample	Duration
Sample 1	0 day
Sample 2	7 days
Sample 3	14 days
Sample 4	21 days
Sample 5	28 days

Table 9

## Hypothesis



Based on the results obtained, the duration for completing pyrolysis process is decreasing as the fermentation duration is increasing except for Sample 3.

Fermentation is considered as biological pre treatment as it is using microorganism and no additional equipments and chemical is used. It will increase the enzymatic digestibility of the lignocellulosic biomass structure hence it is easier to be accessed.

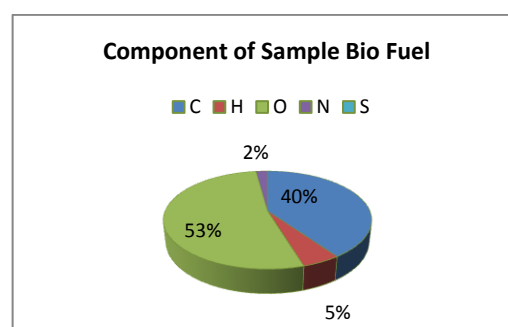
According to Xuebing Zhao, Lihua Zhang and Dehua Lu (2012),

The employed micro organisms are usually capable of degrading lignin and carbohydrate polymer.

Basically, fermentation employs micro organism to break the cell wall which is consequently made the lignocellulosic biomass easy to be accessed for further conversion to bio fuel. These statements support the hypothesis that has been made which is fermentation is affected the duration of pyrolysis process. The more fermentation duration, duration of pyrolysis process is reduced.

## Characterization of Samples

ELEMENT	EFB (%)	TRUNK	FRONDS
C	53.78	40.64	52.28
H	4.37	5.09	
O	41.5	53.12	
N	0.35	2.15	0.75
S	-	-	
CV(MJ/KG)	17.08	17.27	



## IV CONCLUSION AND RECOMMENDATION

### A. Conclusion

Pre treatment is very important process in converting lignocellulosic biomass. Biological pre treatment is one of the methods of the pre treatment. It is very cheap compared to other pre treatment as it does not need any additional chemical and equipments. Besides it is very environmentally friendly green methods. If previously fermentation took place after the chemical pre treatment, in this study the fermentation will come first as pre treatment before converting to bio fuel.

### B. Recommendation

Lignocellulosic biomass is very significant feed stock in order to replace the diminishing of petroleum as currently being the primary sources for the fuel production. Therefore:

- i. Further study need to be done on the possible pre treatment for the lignocellulosic biomass so that the cost for converting it to bio fuel is more efficient.
- ii. Fermentation is one of the pre treatments that need to be seriously taken into consideration as the methods are very easy and cheaper as no additional chemical and equipment are needed. Besides, it is very friendly and green technology. Thus, careful studies on these methods need to be done.
- iii. Due to time constraint and technical error, the analysis of the products produced cannot be done. Hence, for more efficient investigation further study on the products need to be done and if possible further converted to fuel transportation

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