FURFURAL SEPARATION FROM PALM EMPTY FRUIT BUNCH (PEFB) USING SOLID WASTE CATALYST (SWC)

NURUL ARPAH BINTI ABD MALEK

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UNIVERSITI MALAYSIA PAHANG

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ABSTRACT

Oil-derived chemicals is been used to produce nylon, spandex, and other products. However, it seems that in the near future time, we would exchange the used of oil-derived chemicals into the furfural as before. This is due to oil-derived chemical is a non-renewable sources while we can found furfural in many renewable sources. Furfural is a colourless oily liquid with the odour of almonds chemical that produced from hemicelluloses. It is an organic heterocyclic aldehyde compound that stable at room temperature. Hemicelluloses can be found from agricultural waste such as palm empty fruit bunch (PEFB), corncobs and etc. PEFB is an abundant by product of palm oil industry that contains lignocelluloses biomass which composed of hemicelluloses, cellulose, and lignin. Furfural is produced by the hydrolysis and dehydration process of pentose that contained in lignocelluloses. First of all, PEFB is characterized and the raw materials in dried, grinded and sieved before hydrolysis and dehydration process take places. In this project, we synthesized furfural from PEFB and can get the improvement of yield by using solid waste catalyst (SWC). As the result, the optimum condition to produce the highest yield (%) of furfural is at 90 min, with the liquid/solid ratio of 16.67 ml/g and 30 g of catalyst.

ABSTRAK

Hasil kimia berdasarkan minyak telah digunakan dalam pemprosesan nilon, spandex dan berbagai produk yang lain. Walaubagaimanapun, ianya dilihat bahawa dalam masa terdekat ini, kita bakal mengubah daripada penggunaan hasil kimia berdasarkan minyak ini kepada furfural seperti sebelum ini. Hal ini disebabkan oleh hasil kimia daripada minyak merupakan sumber yang tidak boleh diperbaharui manakala furfural boleh didapati daripada pelbagai hasil bahan yang boleh diperbaharui. Furfural adalah sejenis cecair minyak yang mempunyai haruman seperti kacang badam yang diperolehi daripada hemisellulose. Ia adalah sejenis organik sebatian aldehid heterosiklik yang stabil pada suhu bilik. Hemicellulose boleh ditemui daripada hasil buangan agrikultur seperti tandan kosong kelapa sawit, jagung dan lain – lain. Tandan kosong kelapa sawit adalah sejenis produk sampingan yang banyak dihasilkan daripada penanaman kelapa sawit yang mengandungi lignocelluloses biojisim yang merangkumi hemicelluloses, cellulose dan lignin. Furfural dihasilkan melalui proses hidrolisis and dehidrasi oleh pentose. Sebelum melalui dua proses tersebut, tandan kosong kelapa sawit akan di uji cirri – cirinya menggunakan beberapa jenis alatan makmal dan ia akan dikeringkan, dikisarkan dan diasingkan. Melalui projek ini, furfural dapat dihasilkan daripada tandan kelapa sawit dan penambahbaikan kadar hasil produk dapat ditingkatkan melalui implementasi hasil buangan pepejal sebagai pemangkin tindak balas. Hasil kajian menunjukkan bahawa keadaan terbaik bagi menghasilkan kadar furfural yang tertinggi adalah pada masa 90 min, nisbah air/pepejal sebanyak 16.67 ml/g dan jumlah pemangkin sebanyak 30 g.

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LIST OF ABBREVIATIONS

EFB	Empty Fruit Bunch
HPLC	High Performance Liquid Chromatography
MC	Moisture Content
PEFB	Palm Empty Fruit Bunch
SWC	Solid Waste Catalyst

1. INTRODUCTION

1.1 Motivation and problem statement

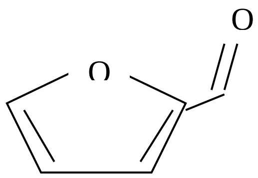
Due to high dependency of non-renewable resources in order to obtain energy and chemicals, many researchers have shifted their view to sustainable sources. Among these, palm cultivation had produced lots of products that can be used for sustainable production. Palm oil is originated from West Africa and widely grown in many tropical regions of Southeast Asia especially in Malaysia and Indonesia. In Malaysia, palm oil production has growth over the years. Thus producing the great number of empty fruit bunch (EFB) as solid residue (Chang, 2014). There were approximately 55.73 million tons of lignocelluloses agricultural waste and by-products per years had been generated through palm oil industries in these two countries including PEFB (Kim et al., 2012). In the past few years, EFB has been used as fuel to generate steam by incineration at the mills and the ash produced was used as fertilizer or soil conditioner. However, it is soon discouraged due to the emission of large amount of white smoke that affects the surroundings (Chang, 2014). Recently, there are great numbers of abundant EFB that acts as a waste. The abundant empty fruit bunch consists of 41.3% - 46.5% cellulose, 25.3% - 33.8% hemicelluloses and 27.6% – 32.5% lignin (Kim et al., 2012). Cellulose is a homopolysaccharide composed of ß-D-pyranose units while lignin is the most complex natural polymer consisting of predominant building block of phenylpropane. Whereas, hemicelluloses is a mixture of polysaccharides, that includes of pentoses, hexoses and uronic acids (Verardi et al.,2012). This project is proposed to turn the waste product to become an important product. EFB is expected can produce the furfural as it contains lignocelluloses components which composed of all those three compounds as stated (Sánchez et al., 2013).

1.2 Background of study

Furfural is an important chemical to gradually increase the production of chemicals and fuels from renewable sources (Agirrezabal-Telleria et al., 2014). It is also a versatile chemical that acts as a starting material for a large family of other chemicals and polymer products (Lamminpää et al., 2014). Currently, furfural plays crucial roles in oil refining as well as food industry (Ong & Sashikala, 2007). Many researchers found that there is no synthetic route furfural as in is industrially produced from lignocelluloses materials that contains pentosans (Zhang et al., 2013) (Win, 2005), which is the main components in the furfural synthesis.

Shown in Table 1, are the general properties of furfural. Furfural also known as 2-furaldehyde or furfuraldehyde has a molecular formula of $C_5H_4O_2$ has an aromatic odor reminiscent of almonds (Yan et al.,2014).

Table 1-1: General physical properties of furfural

Molecular weight	96.08
Molecular structure	
Boiling point(°C)	161.7
Freezing point (°C)	-36.5
Density at 25 °C	1.16
Relative index,n_o	1.5261
Critical pressure,P_c (MPa)	5.502
Critical temperature, T_c (°C)	397
Solubility in water, wt% (25 °C)	8.3
Organic solvent (e.g diethyl ether)	∞
Dielectric constant at 20 °C	41.9
Heat of vaporization (liquid), (kJ/mol)	42.8
Viscosity, mPa.s, 25 °C	1.49
Heat of combustion at 25 °C (kJ/mol)	234.4
Enthalpy of formation (kJ/mol)	-151
Heat of vaporization (kJ/mol)	42.8
Surface tension at 29.9 °C, (mN/m)	40.7
Explosion limit (in air) (vol%)	2.1-19.3

The largest producer of furfural is mainly dominated by China using the corncobs that composed of lignocellulosic biomass (Zhang et al., 2013). The market price of furfural on 2002 is about $1700/ton (Yan et al.,2014) and it is increase to $2000/ton on 2012 (Yan et al.,2014) (Mao et al.,2012). Figure 1 shows the trend for demand of furfural since 1988 until 2026. It shows the past market demand, present and also the expected or projection for the future demand. As shown in figure, the demand is expected to keep increasing over the years. As the China is dominated the growth at the moment, it is targeted that in future the China still be the main producer from this market (Dalin Yebo Trading,2014).

Figure 1-1: Furfural Market

1.3 Objectives

- To separate the furfural from palm empty fruit bunch (PEFB) using solid waste catalyst (SWC).
- To search for yield improvement of furfural using different parameters which are liquid to solid ratio, catalyst loading and reaction time.

1.4 Scope of research

The following are the scope of this research:

i. Characterization of Palm Empty Fruit Bunch (PEFB)
ii. Drying and grinding of the raw materials
iii. Hydrolysis and dehydration of pentoses

iv. Separation of furfural from palm empty fruit bunch (PEFB) by using solid waste catalyst
v. Furfural analysis using HPLC
vi. Yield determination of furfural

1.5 Main contribution of this work

Throughout this research work, furfural synthesis is achieved by using waste product such as PEFB. In the meantime, the number of abundant waste can be reduced to some significant value with the commercial value returns. The production of furfural also turn the dependency of non-renewable resources to renewable resources as it can be used as a based product to produce spandex and nylon.

Although commercial furfural synthesis has been developed by the used of Sulphuric Acid as a catalyst with a high steam for stripping, it encountered some problems regarding this commercialize method whereby, corrosion of reactors, environmental problems, and lack of biomass comprehensive utilization (Ma et al., 2014).

Due to the several limitation of homogeneous catalyst, many researchers have shifted their focussing to heterogeneous catalyst as it is more environmental friendly and have a higher reusability (Boey et al., 2011). Besides that, heterogeneous catalyst is very ease of handling whereby, it is easy to separate from the solvent. Therefore, throughout this research project, the acid catalyst is replaced by base catalyst and solid waste catalyst which is more environmental friendly.

In addition, the used of solid waste catalyst also contribute to the recycle of waste product into the beneficial products. With the introduction of heterogeneous catalyst instead of homogeneous in the research work, it can solve the generation of substantial amount of process wastewater and incapable of catalyst reused that had been faced this while (Boey et al., 2011).

1.6 Organisation of this thesis

The structure of the remainder of the thesis is outlined as follow:

Chapter 2 provides a description of palm empty fruit bunch (PEFB), furfural, its application, and general synthesis routes. Besides, it consists of the reviews on literature related to the synthesis of furfural in past and presence. The reviews on steps involving in the synthesis of furfural also has been comprised in this chapter. The comparison between different types of technologies used is also described and the types of catalyst used in commercial production and its problems were discovered in this chapter. Lastly, the fundamental of HPLC analysis is present in this chapter. Throughout this research, the new types of catalyst have been suggested and been discussed in this project work.

Chapter 3 describes about the materials and methods used to conduct the hydrolysis and dehydration process in order to formed furfural. Materials descriptions and experimental procedures are covered for both the catalyst preparation and synthesis of furfural. A brief explanation of the methods used is also provided in this chapter. The equation involved is also stated.

Chapter 4 presents the results and discussion. The standard curve from the analysis of HPLC is included. The results obtained from the hydrolysis and dehydration process will be present in this chapter.

2 LITERATURE REVIEW

2.1 Overview

This chapter review on introduction of raw material which is the Palm Empty Fruit Bunch (PEFB), the composition of PEFB, and current situation of PEFB in our country. Besides that, throughout this chapter, the synthesis of furfural also had been described with different technologies used in the past. Different types of catalyst used also had been discussed and there is new catalyst that had been proposed in this chapter. Lastly, the fundamental of HPLC analysis is illustrated.

2.2 Raw Material (PEFB)

Nowadays, the worldwide has focusing to utilize biomass for renewable and sustainable fuel and energy development especially on vegetable oil. This due to the beneficial in order to minimize the greenhouse gas emission (Kouzu et al.,2008). However, this scenario leads to the greater number of biomass waste. In the palm oil industry, there are such a bigger number of waste produced which known as lignocellulosic biomass.

Lignocellulosic biomass consists of some major chemical constituents such as cellulose, hemicelluloses, lignin and extractive components (Ye et al.,2008) (Ma et al., 2014). It can be found in many agricultural waste, forest products (hardwood and softwood) and their residues are renewable resources of energy (Iranmahbood et al.,2002). It has the potential to be one of the lower cost materials and as an alternative renewable bio-resource besides using food source such as corncobs. One of the abundant biomass in Malaysia can be found under palm oil industry which is palm empty fruit bunch (PEFB).

A vast number of waste palm empty fruit bunch is produced is West Africa and Southeast Asia (Pimenidou & Dupont, 2012) after the extraction oil from the fresh fruit bunches. As Malaysia being one of the larger exporters of palm oil exporters in the world, it is accounted that about 15.8 Million ton per year of PEFB produced from this industry. This indicates that EFB available in Malaysia is capable providing a sustainable resource for the production of value-added fiber-based products (Ying et al., 2014).

In 2010, Malaysia has becoming the dominant agriculture under palm cultivation with the number around 3.38 Million hectares of land for this cultivation (Lahijani & Zainal, 2011). For these number, it is recorded that 368 palm mills produced lignocellulosic biomass including palm empty fruit bunches (53%), palm mesocarp fibre (32%), and palm kernel shell (15%) (Lahijani & Zainal, 2011). The numbers has significantly growth over the years.

Empty fruit bunch (EFB) is a bulky and brown in color that left over from palm oil mills after the extraction of oil from palm fruit (Chang, 2014). It contains high percentage of hemicelluloses and lignin per gram as compared to other feedstock such as wheat straw and switch grass (Kim et al., 2012). It is irregular in shape. It is a lignocelluloses biomass waste from palm industry. Table 2 below shows the chemical composition in the EFB by weight percentage on dry basis (Chang, 2014) (Abdullah & Gerhauser, 2008) (Kim et al., 2012).

Table 2-1:Properties of EFB Fibers

Components	Chemical composition (%)
Cellulose	•23.70 – 65.00 •41.30 – 46.50
Hemicellulose	•20.58 – 33.52 •25.30 – 35.80
Lignin	•14.10 – 30.45 •27.60 – 32.50
Extractive	•3.21 – 3.70

2.3 Furfural

2.3.1 Furfural Synthesis

Recently, the only way for furfural synthesis is through biomass hydrolysis and pentose dehydration since there is no synthetic route for the production yet (Agirrezabal-Telleria et al., 2014). Figure 2, shows that the synthesis of furfural is from the composition of hemicelluloses in lignocellulosic biomass by hydrolysis process. As shown in figure 2, different components would produces different types of chemicals (Huang & Fu, 2013)**.** Basically, furfural can be produced from xylose (pentose) that contain in hemicelluloses in lignocellulosic biomass. The primary hydrolysis of pentosans will gives the respective pentose and by dehydration process will result on formation of furfural (Antunes et al., 2012)

Hemicellulose and cellulose are the polysaccharides that formed by the linkage of monosaccharides via glycosidic bonds. As our research are more concentrated on hemicelluloses, it is a heteropolysaccharides that generally comprised of five different sugar monomers which are D-xylose, L-arabinose, D-galactose, D-mannose and D-glucose with the vast number of xylose (Zhou et al., 2013).

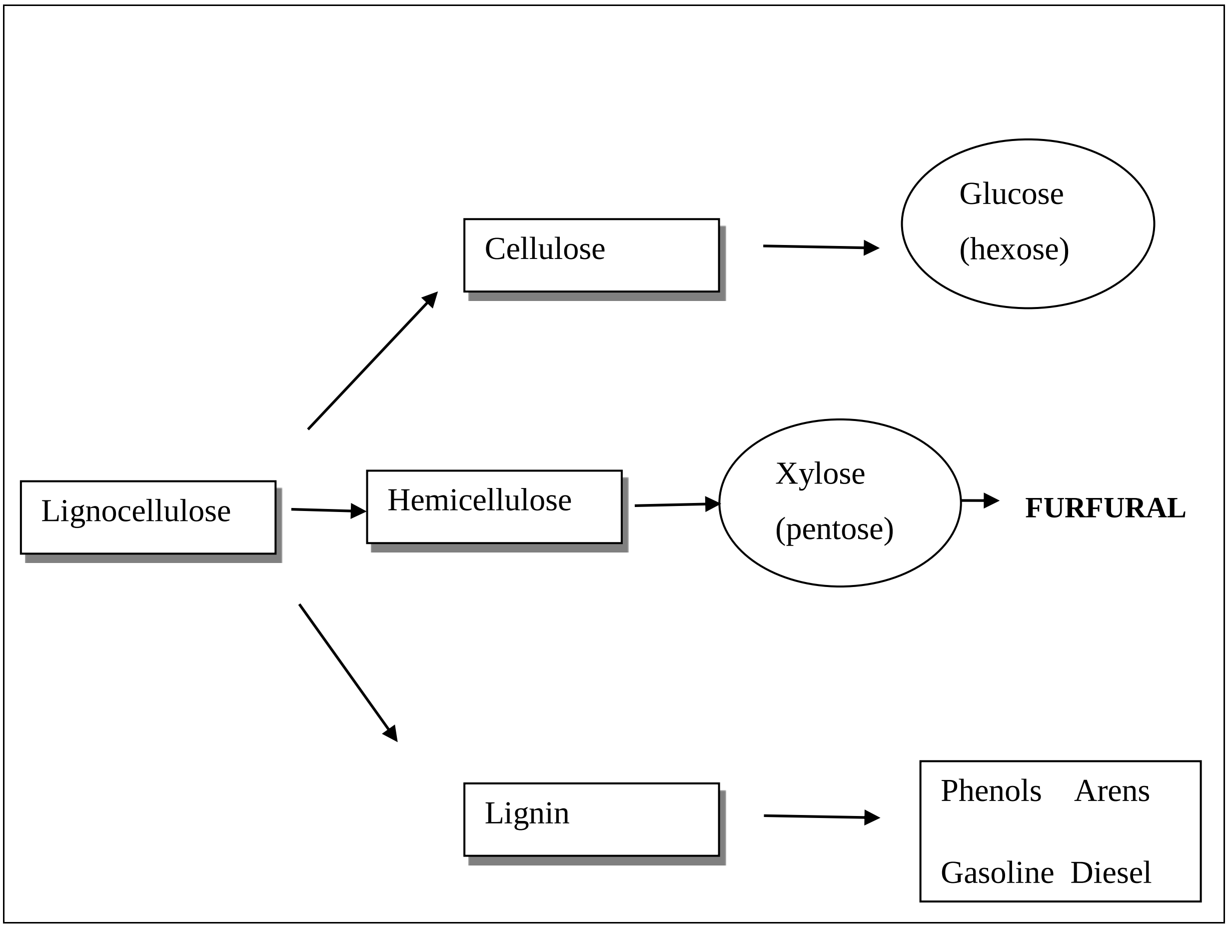


Figure 2-1: Utilization of lignocellulose to produce furfural

In figure 3, it shows the pathway of furfural production by using acid catalyst. Xylan from plant biomass is converted to D-xylose by hydrolysis process. Then, it will further to dehydration step, whereby the furfural is formed (Antunes et al., 2012).

In the dehydration process, many literature stated that lignin might disturbed the furfural yield (Lamminpää et al., 2014). It shows that lignin will accelerate glucose decomposition in acidic conditions and it will prevent the hydrolysis of lignocelluloses to sugars (Lamminpää et al., 2014). However, it is said that the effect of lignin on furfural yields are not clear whether it is affected by the types of catalyst used or the reaction conditions or it might cause of both of the reasons (Lamminpää et al., 2014).

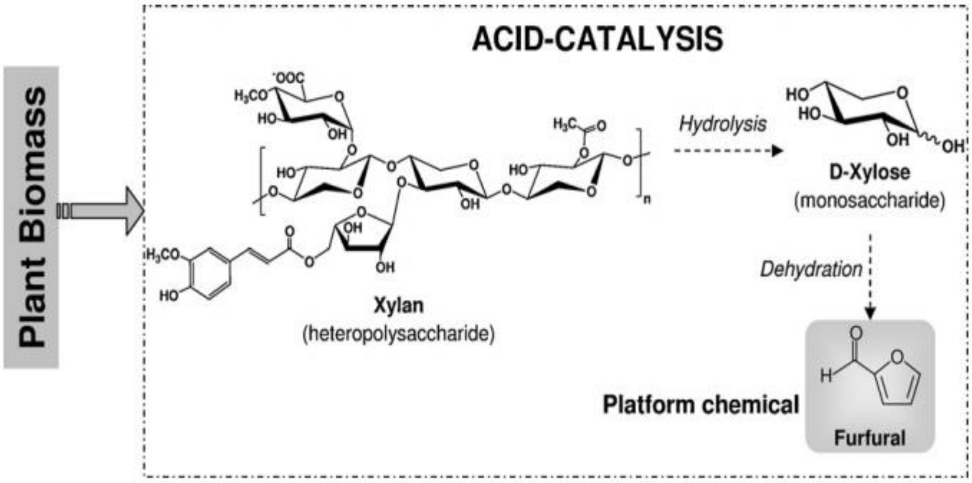


Figure 2-2 : Pathway in furfural synthesis

The potential yield of furfural might lower because of non-selective process that occurs during the xylose cyclodehydration (Molina et la., 2012). These sides reactions includes the condensation of furfural, resinification reactions of furfural and fragmentation or decomposition reactions of xylose (Molina et la., 2012). Attributed to these side reactions, pentose molecules are not selectively converted to furfural and consequently produced small amount of desired product (Molina et la., 2012).

Figure 4, shows the pathway of side reactions occurs during the dehydration of xylose to furfural (Agirrezabal-Telleria et al., 2014).

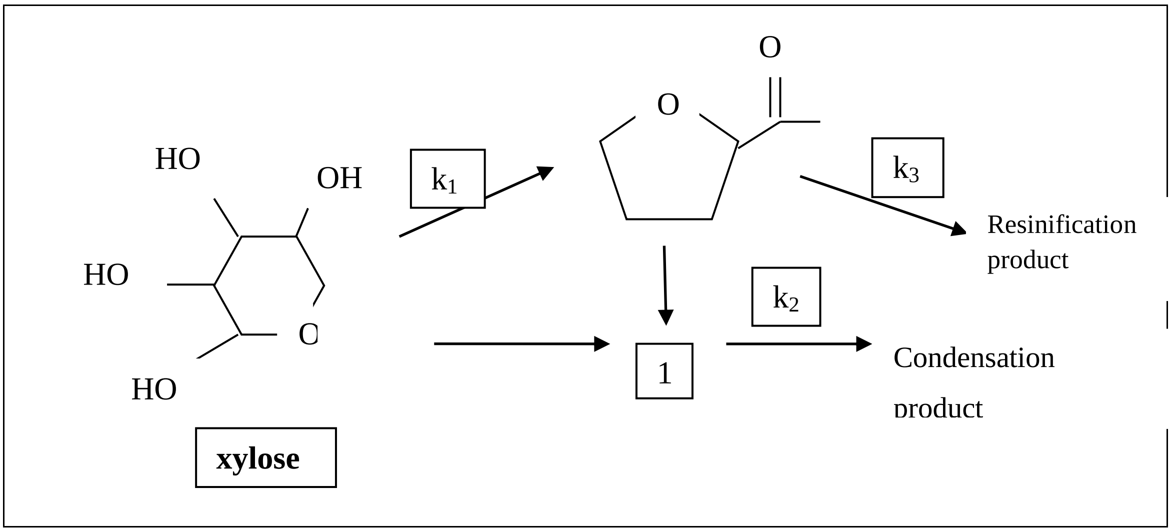


Figure 2-3 : Dehydration and side reactions involved during the production of furfural from xylose

One of the most common and important chemical transportation of carbohydrates is the hydrolysis and dehydration process of polysaccharides into the furan platform products. (*Catalytic Process Development for Renewable Materials*, 2013). Furfural is one of the furan platform products as well as methyl-furyl and 5-hydroxymethylfurfural.

2.3.2 Importance of furfural

Furfural has many beneficial functions. From the study conducted before, furfural is often uses as the feedstock for the sustainable production of value-added chemicals and bio-fuels (Yan & Chen, 2014). It is important as oil refineries, plastics, food, pharmaceutical and agricultural industries (Riansa-Ngawong & Prasertsan, 2011). For oil refineries, it is acts as selective solvent. By the extraction, aromatics, polar components, mercaptans are removed from petroleum. Besides that, it is also used as decolorizing agent to refine crude wood resin. Furfural and its derivatives have been used to make a jet and diesel fuel, to serve as gasoline blend stock and to develop new generation of bio-fuels and bio-plastics (Xing et al., 2011). Plus, it is also important as a chemical stock for production of furfuryalcohol, methylfuran, acetylfuran and etc (Bamufleh et al., 2013).

2.3.3 Raw materials of furfural (Last research)

It was detected that the presence of furfural can be found in various processed foods and beverages for examples, cocoa, coffee, tea and etc. The food products have been exposed to thermal treatment and led to the breakdown of carbohydrates to furfural (Loi et al., 2011). Before this, the research was done on the hazel nuts shell, sunflower, walnut and almond (Demirbas, 2006) and also rice husk (Ong & Sashikala, 2007).

2.3.4 Process involved in producing furfural (Last research)

There are two types of processes involves in obtaining the furfural which are one-stage process and two-stage process (Mansilla et al., 1998). Depolymerization of pentosans in xylose and dehydration to furfural occurs simultaneously in one-stage technology or in other words the process of hydrolysis and dehydration occurs in the same reactor (Riansa-Ngawong & Prasertsan, 2011) , while in two-stage technology, a dissolution and depolymerisation of pentosans occurs under mild conditions, followed by dehydration of pentose into furfural (Ma et al, 2014) in two different reactors (Riansa-Ngawong & Prasertsan, 2011).

The advantages of two-stage process is the residual lignocelluloses are less degraded and can be utilized to synthesize others products such as ethanol, glucose, pulp and paper through fermentation process (López et al., 2014) (Sánchez et al., 2013) and produced higher yield (Riansa-Ngawong & Prasertsan, 2011).

From the past researchers, the technical process involved was by using Westpro Modified Huaxia Technology (Win, 2005). Through this process, it includes the step of pre-treatment, hydrolysis and refining. Pre-treatment is the preparation of raw materials whereby it usually crushed before blended with sulphuric acid. Then, the process is continues by hydrolysis step which is done in steel reactors because of the used of sulphuric acid. All the furfural formed will be removed by steam by steam distillation. In order to recover the by-product which is acetic acid, two recovery towers are added. Secondary steam generator may be used for distillation in line to save cooling water and energy. The last step is take place by using azeotropic distillation. At the top of refining tower, furfural layer is fed continuously.

In the synthesis of furfural, it will turn out the waste residue that often used for solid fuel after drying (Mao et al., 2012). The residue usually contained large portion of cellulose that could be transformed into glucose by fermentation techniques (Mao et .,2012). $FeCl_3$ is more effective in cellulose degradation and acetic acid is needed for lignin removal (Mao et al.,2012). However, the problems occurs in degrade all the residue where it is cost-effective to release sugars from recalcitrant lignocelluloses (Mao et al.,2012).

Figure 5 shows that the experiment set up for furfural in lab scale. The acid- hydrolysis process is take places in the acid-impregnated biomass whereby the acid catalyst is mixed with the biomass and it is heated. The furfural form is stripped away and the liquid form of furfural is obtained (Marcotullio., 2013).

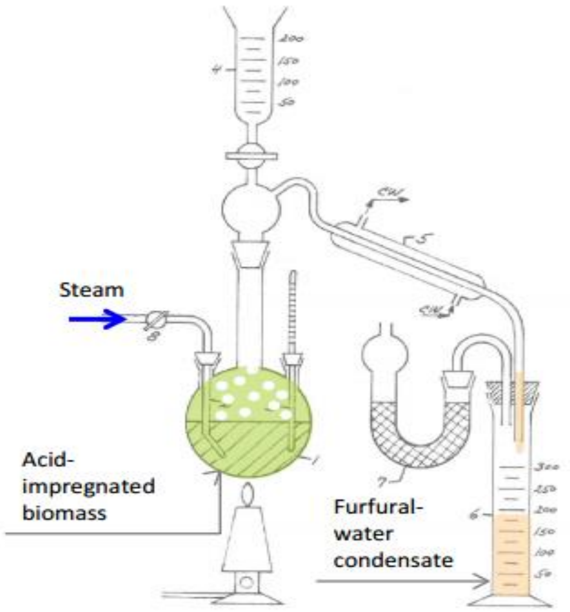


Figure 2-4 : Lab experiment set up

In the case of enzymatic hydrolysis process were run, it is needed to go through the pre-treatment process whereby to remove the lignin and also enhance the porosity of the lignocelulosic materials (Verardi et al.,2005)

In the dehydration process problems, there are many technological approaches that have been proposed to solve these problems. Two of them are SUPRATHERM and SUPRAYIELD processes (Molina et la., 2012). Not only that, other approach is by applying N_2 to be strip under semi-batch conditions in the lab scale experiments (Molina et la., 2012).

Recent researchers of dehydration process uses solvent to extract the generated furfural in batch biphasic reactors. This is because, the reactor is easy to operate, however, it leads to unsatisfactory extraction efficiency and time consuming (Ma et al., 2014). In order to promote extraction efficiency, mechanical agitation is suggested (Ma et al., 2014). Still, the agitation is should not be too excessive as it could lead to the serious axial back-mixing followed by a worse extraction performance (Ma et al., 2014).

2.4 Catalyst used in furfural production

Commercially, the synthesis of furfural had been introduced with the presence of homogeneous catalyst. The common homogeneous acid used is H_2SO_4 and HCl. HCl is more advisable over H_2SO_4 because it permeates wood easily and it is volatile compound that assist in the acid recovery steps (Demirbas, 2006). However, both of them faced the same problems where by large quantities of corrosive wastewater is produced (Zhang et al.,2014), corrosion of pipe, expensive recovery problems (Demirbas, 2006), and end products inhibition (Ormsby et al., 2012).

Alternatively, due to those reasons, enzymatic hydrolysis was suggested (Ormsby et al., 2012). There are three major groups of proteins in enzymes or cellulases. There are endoglucanase, exoglucanases and ß-glucosidase that plays different roles in enzymatic reactions. Endoglucanase works to attacks low crystalinity regions in the cellulose fibers by endoaction. Exoglucanases hydrolyze the 1,4-glycocidyl linkage to form cellobiose and ß-glucosidase converts cello-oligosaccharides into glucose residues (Verardi et al.,2005). In addition, there are others enzymes that also functions to attack hemicelluloses and celluloses such as glucomonide, acetylesterase, xylanase, ß-xylosidase, galactomannase and glucomannase (Verardi et al.,2005).However, it has disadvantages whereby it production is too expensive and practical processes for enzymes recovery and reuse currently do not exist thus, it is discouraged due to those reasons (Tan et al., 2013) (Ormsby et al., 2012) (Verardi et al.,2005). At the moment, there are no commercialize plant that use the enzymatic hydrolysis. It has only the plant for demo scale (Verardi et al.,2005).

Recently, the researchers has focus on the use of heterogeneous acid-catalyst as it shows a biggest potential and easy post-separation through simple filtration step (Agirrezabal-Telleria et al., 2014). Besides that, it also contain a variety of acid sites with different strength of Bronsted or Lewis acidity, compared to the homogenous acid catalyst (Helwani et al.,2009). The most common catalyst that have been studied is zeolite as it can be strong acidic catalyst and shows thermal and chemical stability (Agirrezabal-Telleria et al., 2014). In the line of choosing the acid solid catalyst, there are some important requirements that need to be considered which are water tolerance, hydrothermal stability, and good thermal stability if the regeneration process of the catalyst includes the thermal decomposition of the accumulated carbonaceous deposit or chemical stability if carbonaceous deposits are removed by harsh chemical treatment (Antunes et al., 2012). However, acid catalyst can cause severe problems that same to the used of homogeneous acid catalysts. For example, the corrosion of pipe and environmental effect (Kouzo et al.,2008).

Solid base catalyst is a noble process that has a bright technology that featuring to the fast reaction rate (Kouzu et al.,2008). There are few types of solid base catalyst that have been introduced. Some of them are magnesium oxide, strontium oxide, calcium hydroxide and carbonate (Viriya-empikul et al.,2010).

For this experiment, calcium oxide will be used for the types of catalyst. CaO has been reported as an active heterogeneous base catalyst due to its high basicity, low solubility and easy handling (Ho et al.,2014). The selection of this acid is because of the reusability characteristics where CaO was found to be still active after the twentieth used (Boey et al., 2011) (Chouhan & Sarma, 2011) and it is environmental-friendly (Viriya-empikul et al.,2010). Shown in table 3 is the properties of calcium oxide (Boey et al.,2011).

Table 2-2 : Properties of calcium oxide

Items	Description
Chemical name	Calcium oxide
Chemical formula	CaO
Common name	Lime, calx, quicklime,burnt lime, unslaked lime, fluxing lime, caustic lime
Density(g/cm^3)	3.40
Melting point (°C)	2572
Boiling point (°C)	2850
Heat of formation (kcal/mol)	151.9
Heat of hydration (kcal/mol)	15.1
Solubility of $Ca(OH)_2$ (g/100g H_2O)	0.219
Decomposition temperature (°C)	547

Applications	In furnace lining, metal smelting, glass making, fertelizer, drying agent, mortar, paper and pulp production, drilling fluid, pollution control, water purification, sugar and cellulose industries, medical (destroy warts , moles)

Modification proposed in this research work is the used of solid waste catalyst in order to represent the calcium oxide. By introducing the solid waste as a catalyst, it could eliminate the number of waste and at the same time produced the catalyst with high cost effectiveness (Viriya-empikul et al.,2010).

There are various types of natural calcium sources from waste, such as egg shell, mollusc shell and bone (Viriya-empikul et al.,2010). Solid waste that used in this research work as a catalyst to replaced CaO is egg shells. The advantage is it has low level of toxic substance (Oliveira et al.,2013). Egg shell catalyst can be obtained by calcinations-hydration-dehydration treatment of the waste (Niju et al.,2014). It is found that these catalyst have a cycle 6 times. Solid catalyst was separated from reaction mixture by filtration for each cycle. It has to be wash with methanol to remove the adsorbed strains and recalcined at 600 °C for further usage (Niju et al.,2014).

2.5 HPLC analysis

HPLC or High –Performance Liquid Chromatography is a technique in analytical chemistry used to separate the components in a mixture, to identify each component, and to quantify each component. It involves the separation techniques and operates by the placement (injection) of a small volume of liquid sample into a tube packed with porous particles (stationary phase), where individual components of the sample are transported along the packed tube (column) by a liquid moved by gravity (Hplc, Agilent Technologies.)

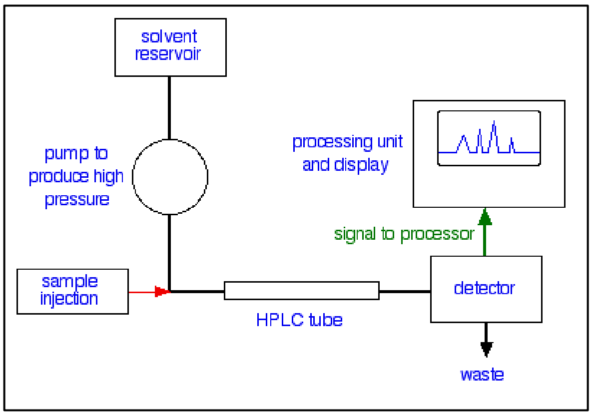


Figure 2-5 : A flow scheme for HPLC

Figure 2-5 shows the flow scheme for HPLC analysis. The sample is placed in the HPLC tube by the injection. Then, the detector used is a column. The type of column used is different with different types of samples used. Lastly, the response or chromatograph will be appears at the processing unit.

2.6 Summary

Generally, this section describes about the raw material used, the description about the expected product and also the types of catalyst used. The types of analysis used and it fundamental is also illustrated. The drawbacks of the past researchers are also included in this section to compare with the best method to be used.