



## The Effect of H<sub>2</sub>SO<sub>4</sub> Concentration and Micro Wave Power in Microwave Assisted Hydrolysis of Furfural Production from Empty Palm Fruit Bunches

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### Abstract

Empty fruit bunches (EFB), which are wastes from fresh palm fruit bunches, are one of organic wastes from palm oil processing industries as much as 23% of the total production. The utilization of this waste, which has a high economic value, is still not optimum although it has been used as a boiler fuel. In fact, EFB has a high economic value if processed further. The EFB as a waste can be processed using hydrolysis method to obtain its pentosan content and changed into furfural with the addition of acid as a catalyst. The objectives of this research are to find out the optimum yield on the sulfuric acid concentration and the effects of the power of microwaves used on hydrolysis process of EFB. On this hydrolysis process, 10 grams of EFB was added with 250ml of sulfuric acid with the variations of 3%, 6%, 9%, 12%, 15%, and 18%, and microwave was used with the power variations of 400W, 600W, and 800W for 75 minutes. The analysis method used in this study to determine the furfural content is Gas Chromatography (GC). The results showed the highest furfural content was obtained at H<sub>2</sub>SO<sub>4</sub> concentration of 9% with 800W power on the microwave and the concentration of 0.39 mg/mL and furfural yield of 0.9620%.

### INTRODUCTION

Empty palm fruit bunches are one of the organic wastes from palm oil processing plants that have a high level of availability throughout the year. In March 2017, there were 75 palm oil processing plants with an average consumption of Fresh Fruit Bunches (FFB) of 80 tons / month or 953 tons / year per plant (Disbun Kaltim, 2017). Empty bunches produced from 1 ton of oil palm were 23% (Sampepana & Saputra, 2011).

In general, the utilization of empty palm fruit bunches (EPFB) itself is still not optimal, even though it has been used as boiler fuel (Rahim & Nadir, 2015). In addition, the EPFB have only been used as compost and activated carbon, whereas the quality of the waste can be increased to become an important chemical raw material (Kurniasih et al., 2012).

The EPFB contained 22.60%,  $\alpha$ -cellulose 45.80%, pentosan 25.90%, ash 1.60%, and water 4.1% (Purwito & Firmanti, 2005). With the high content of pentosan, the potential EPFB as a raw material for making furfural is processed through the hydrolysis process. Furfural functions as a solvent in the lubricating oil refining industry, purification of vegetable and animal oils, resins and waxes, and the production of hexamethylene diamine for the manufacture of nylon (Parasta, 2014).

The study of the effect of temperature, sulfuric acid concentration (as a catalyst) and hydrolysis time on furfural production from EPFB was studied by Kurniasih et al. (2012), Parasta (2014), and Rahim & Nadir (2015). The research conducted by Kurniasih et al. (2012) obtained the optimum results at concentrations of 9% H<sub>2</sub>SO<sub>4</sub> with a heating time of 3 hours. The results of the

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study obtained the highest yield of 30.017%. However, the density obtained is 1.3409 g / mL. This is possible in furfural still containing impurities. Parasta (2014) conducted a study of the effect of temperature, H<sub>2</sub>SO<sub>4</sub> concentration and hydrolysis time on making furfural from conventional EPFB. In this study the best results were obtained at 90°C, 15% H<sub>2</sub>SO<sub>4</sub> concentration, and 120 minutes hydrolysis time with a one-stage hydrolysis process. Furfural concentration obtained is still low at 0.368 mg / mL with a percentage of 30.357%. In the research conducted by Rahim and Nadir (2015), by varying the hydrolysis time using microwave aid with a temperature of 90°C and a concentration of 15%, the optimum hydrolysis time was obtained 75 minutes, which obtained the highest furfural concentration of 1.34mg / mL.

In this study there were several things that made it possible to improve, including a large hydrolysis time (3 hours) with a relatively low acid concentration (9%) and a relatively high temperature, i.e. 100°C (Kusniasih et al. 2012), furfural concentration which relatively low with the hydrolysis process at low pressure and medium temperature (90°C) even though acid concentration (15%) and hydrolysis time (2 hours) are used relatively high (Parasta, 2012). In the research of Rahim and Nadir (2015), producing furfural with a higher concentration only used relatively high acid concentration (15%) with temperature (90°C) and low hydrolysis time (75 minutes).

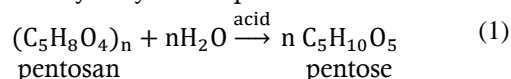
The three studies can be refined by adding microwave aids to hydrolysis as done by (Rahim & Nadir, 2015) with variations in the concentration of sulfuric acid and microwave power. Variations of acid concentration were used to increase furfural concentration, this is due to the increasing concentration of acid used resulting in an increase in the number of reactants that are activated, so that the reaction speed constants become large and the reaction speed increases. But with the help of microwaves, the acid concentration used does not need to be too high because, at high concentrations furfural decomposition can be a further reaction. Microwaves can be used to speed up the hydrolysis process and help to increase the furfural products. The basic mechanism of microwave heating is due to the agitation of polar molecules or moving ions because of magnetic field movements. The presence of magnetic and electrical movements causes these particles to be limited by a limiting force. This causes particle movements to be held back and

generate random movements that produce heat (Taylor & Atri, 2005).

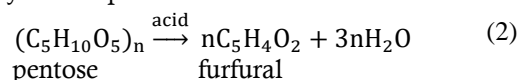
The purpose of this study is to obtain furfural concentrations at optimum yield conditions from the hydrolysis process of empty palm fruit bunches into furfural at various H<sub>2</sub>SO<sub>4</sub> concentrations using microwave assisted power. From this research, it is expected to increase the utilization of empty palm fruit bunches so that they are not wasted, and increase the economic value of empty palm fruit bunches as a raw material for making furfural. Empty palm fruit bunches have various uses for both food and non-food industries (oleochemicals) and by-products / waste. Oil palm waste includes leaf midribs, intisawit cake, sludge, palm fruit empty bunches, shells and fiber (Dirattanhun, 2008).

Empty palm fruit bunches contain a lot of pentosan (25.90%) which can be hydrolyzed to pentose and then dehydrated to furfural with the help of an acid catalyst (H<sub>2</sub>SO<sub>4</sub>). Acid hydrolysis in Furfural production is the most important stage in furfural formation of lignocellulose found in TKF. The formation of furfural is through two stages of reaction, the first reaction is hydrolysis of pentosan or hemicellulose to be pentose and followed by the second reaction namely pentose dehydration to form furfural.

Pentosan hydrolysis into pentose



Dehydration pentose forms furfural



In the hydrolysis process of cellulose, pentosan decomposes into pentose. Then pentose is dehydrated to furfural. Hydrolysis of cellulose can be carried out enzymatically or with the use of catalysts (Parasta, 2014). However, the common process in furfural production is hydrolysis with acid. Acid catalysts that are often used are HCl and H<sub>2</sub>SO<sub>4</sub> (Hambali et al., 2016). The use of microwaves on heating is expected to provide a more even heating effect because the heating process is not transferring heat from the outside but generating heat from inside the material. This results in faster energy transfers, and has the potential to improve product quality.

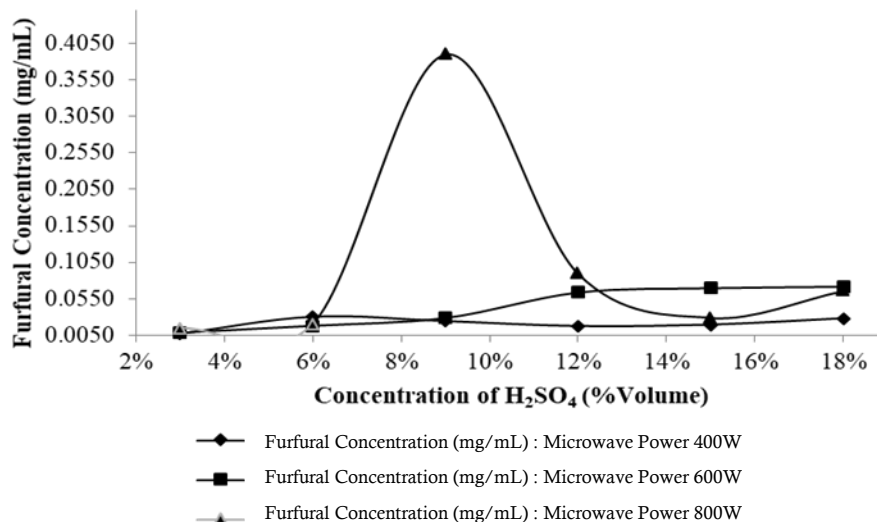


Figure 1. Effect of H<sub>2</sub>SO<sub>4</sub> Concentration on the Furfural Concentration at 400W, 600W, and 800W microwave power.

## RESEARCH METHODOLOGY

This research will be carried out through the following stages:

### Preparation Phase

EPFB material taken from the CPO industry of PT. TELEN Teladan Prima Group Pengada Village, Karangany District, East Kutai Regency, East Kalimantan Province Indonesia. The material is reduced to 12mesh and then dried at 60°C for 24 hours.

### Hydrolysis Phase

A total of 10 g of dried EPFB was put in a 1000 mL flask and mixed with 250 mL to vary the concentration of sulfuric acid (E. Merck) 3%, 6%, 9%, 12%, 15% and 18% v/v. The mixture is put into a microwave oven that has been equipped with thermocouple and temperature control (temperature control). The hydrolysis process is carried out at a temperature of 90°C, aided by a variation of 400W, 600W and 800W microwave power for 75 minutes.

### Furfural Separation Stage

After the hydrolysis process, the material that has fallen in temperature according to the room temperature is then separated using filter paper so that the liquid (hydrolysate) was separated from the dregs of solid EPFB. Hydrolysate was then extracted by adding 50 mL of chloroform (E Merck) and shaken strongly so that furfural can be

transferred to chloroform. Furthermore, the hydrolysate will become two layers, the top layer is the remaining hydrolysate containing water and sulfuric acid, while the lower layer is chloroform which has dissolved furfural. The upper and lower layers were separated by using a separating funnel.

### Analysis Phase

Furthermore, furfural concentration in mg/mL units was analyzed by using the GC-FID method. The accuracy of the GC-FID analysis used as a standard comparison of furfural is a pro analysis of E-Merck. GC-FID analysis was carried out at the Samarinda State Polytechnic Chemical Engineering Instrument Laboratory. The chromatogram comparison method measured by the area of furfural sample area with standard furfural was carried out based on the similarity of peak retention times of standard furfural chromatograms with peak retention time of chromatogram furfural samples. The area of the chromatogram produced at the same retention time using the standard curve method was used to obtain the sample furfural concentration.

## RESULTS AND DISCUSSION

The results of the study influence the concentration of H<sub>2</sub>SO<sub>4</sub> on the concentration of furfural produced in the hydrolysis process of EPFB fibers at 400, 600 and 800W as shown in Figure 1.

Figure 1 shows that the higher of microwave power used resulted the higher furfural

concentration (mg/mL). This is because the higher power causes increased microwave and material interactions. Materials contain polar molecules when exposed to magnetic fields that are isolated at certain frequencies, polar molecules try to follow the orientation of the field and position themselves in the direction of the field (Taylor & Atri, 2005). This random movement of molecules and random interactions will generate heat. The higher the power, the greater the resonance of the wave, the greater the vibration produced thus it accelerates the movement and random interaction of molecular particles which formed the heat quickly.

In this study, the concentration rose significantly and reached the optimum value at 800W with the concentration of 0.39 mg/mL. From this study, the resulting furfural concentration was higher than previous researchers conducted by Parasta (2014), which was 0.368 mg/mL. This indicates that the use of microwave power is able to increase furfural concentration, compared to conventional processes. Whereas the power variations of 400W and 600W increase in furfural concentration tends to decrease and stagnate at concentrations ranging from 0.001 to 0.05 mg/mL.

The effect of concentration on microwave power on furfural yield can be seen in Figure 1. The higher H<sub>2</sub>SO<sub>4</sub> concentration and power in microwaves, the greater the concentration obtained. This is due to the increase in the number of reactants activated so that the reaction speed constants become large and the reaction speed increases.

Acid hydrolysis is the most important stage in furfural formation of lignocellulose found in EPFB. The formation of furfural is through two stages of reaction, the first reaction is hydrolysis of pentosan or hemicellulose to be pentose and followed by the second reaction namely pentose dehydration to form furfural.

The process of pentosan hydrolysis using sulfuric acid can be carried out enzymatically because sulfuric acid serves as a catalyst that helps work water in the hydrolysis process (Zeitsch, 1990 in Parasta, 2014). From the results of this study, it can be seen that in H<sub>2</sub>SO<sub>4</sub> variations concentrations of 3%, 6%, 9%, 12%, 15%, and 18% the highest furfural concentration was obtained at a H<sub>2</sub>SO<sub>4</sub> concentration of 9% by using 800W power. The furfural concentration obtained is 0.5340 mg/mL. This is caused by the increase in the number of reactors that are activated so that the

reaction speed constants become large and the reaction speed increases too, but after reaching the optimum acid concentration the furfural results will decrease. This is due to the decomposition of furfural into furoic acid as a result of the breakdown of the aldehyde group formed by a type of black resin (Dunlop, 1948). This condition is seen in variations in H<sub>2</sub>SO<sub>4</sub> concentrations of 12%, 15%, and 18%, where a decrease in furfural concentration compared with variations in the concentration of 9% H<sub>2</sub>SO<sub>4</sub>, once after reaching an optimum acid concentration of 9%, furfural results will decrease.

## CONCLUSION

The results of the research conducted can be summarized as follows; first, the value of furfural obtained is 0.39 mg/mL with a concentration of 9% H<sub>2</sub>SO<sub>4</sub> and uses microwave power of 800W. Second, in the variation of 9% H<sub>2</sub>SO<sub>4</sub> concentration and 800W microwave power obtained furfural yield of 0.962%.

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