Hydrolysis of oil palm empty fruit bunch using rotating microwave reactor

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Abstract. This study using microwave application as a heat source to facilitate the conversion of oil palm empty fruit bunch (OPEFB) into glucose by hydrolysis process. The hydrolysis process using sulfuric acid (H2SO4) as a catalyst at a various concentration (0 and 0.25 N) and level of microwave power (100 and 300 W). The glucose concentration is measured by the DNS method using UV-VIS spectrophotometric. The best glucose concentration is 23.038 ppm at 60 minutes, 0.25 N H2SO4, and the level of microwave power is 300 W.

1. Introduction
The oil palm empty fruit bunch (OPEFB) is the largest lignocellulose biomass from palm oil mills. Lignocellulose from oil palm empty fruit bunch is a potential raw material for glucose production because it consists of cellulose (44.2%), hemicellulose (33.5%) and lignin (20.4%). Glucose is a raw material of bioethanol production and it consists of two basic step that are pretreatment and hydrolysis [1]. The lignocellulose hydrolysis is the process of converting cellulose into glucose using acids or enzymes as catalysts. Hydrolysis that using sulfuric acid (H2SO4) has proven to be a very efficient method in increasing saccharification so it can produce more glucose [2] [3].

In theory, cellulose is composed of β-D-glucose units which are connected with 1,4-glycosidic bonds to form long linear chains. This chain is regulated with hydrogen bonds to form a crystal structure that is resistant to hydrolysis. On the contrary, hemicellulose is composed of short and highly branched polymer chains it caused hemicellulose is easy to hydrolyze into its monomeric components. Because the background of this material characteristic makes the hydrolysis of oil palm empty fruit bunch become more difficult [4]. Therefore heat energy is needed to facilitate the hydrolysis process in separating glucose from oil palm empty fruit bunch fibers. The source of heat energy that can be used in the process of hydrolysis is a microwave. Microwaves are electromagnetic waves that have frequencies from 0.3 to 300 GHz. Microwave irradiation has been widely used in chemical reactions because it has a higher efficiency of heating, increases the reaction rate and reduces the reaction time [5]. Some studies also prove that microwaves can change the structure of cellulose, reduce levels of lignin and hemicellulose, and increase the enzymatic susceptibility in lignocellulose, so it can increase
the efficiency of hydrolysis, reduce lost glucose levels and increase conversion of materials into glucose [6].

2. Method
In this study, the materials used are oil palm empty fruit bunch from PTPN II, Pagar Merbau, Lubuk Pakam, Deli Serdang, North of Sumatera, Aquadest, DNS (Sigma, India), H₂SO₄ (95-97%, Merck, Germany), Potassium Sodium Tartaric (Merck, Germany), NaOH (Merck, Germany), and NaOCl 2%. The main equipment used is a rotating microwave reactor (ME731K, 230 V, 20 L) with a capacity of 2 L and a thermocouple type K (Krupp and Closs size of diameter 3 x 300 mm (Mineral Insulated) C/w Cable 2 m) connected with thermo controller (Shimaden) and monitored using PLX-DAQ Arduino Software.

![Experimental equipment](image)

**Figure 1.** Experimental equipment.

2.1. Delignification of oil palm empty fruit bunch
Oil palm empty fruit bunch is cleaned and direct sunlight used to dry oil palm empty fruit bunch, the dried oil palm empty fruit bunches are cut to a size about 1 cm. 100 gram of cut size oil palm empty fruit bunch put into a beaker glass and 1000 ml of 17.5% NaOH is added and then heated at 80ºC for 2 hours. After 2 hours, the pulp is filtered and washed until the filtrate is neutral, then purification with 250 ml of 2% NaOCl solution at 60ºC for 1 hour. After purification, the pulp is filtered and washed again until the filtrate is neutral then the pulp is dried until the weight is constant.

2.2. Hydrolysis of oil palm empty fruit bunch
Oil palm empty fruit bunch was hydrolyzed using sulfuric acid (H₂SO₄) (0 and 0.25 N) with a ratio of 1:20 (g / ml) [7] in a microwave rotating reactor with a power variation of 100 and 300 watts, magnetron in the microwave produces electromagnetic waves to irrigate the oil palm empty fruit bunch and H₂SO₄. The hydrolysis was carried out into 1 step with a reaction time of 60 minutes. The temperature during the hydrolysis process was measured using type K thermocouples. After the hydrolysis process sample was filtered to separate the filtrate and residue using Whatman filter paper. The filtrate was collected to determine glucose concentration using UV-VIS spectrophotometric.

2.3. Determination of glucose concentration
Glucose concentration is determined by the DNS method with the ratio of sample and DNS reagent is 1:1, the mixture put into the test tube and heated for 5 minutes in boiling water until its color changed to more concentrated. The mixture was cooled to ambient temperature and measured its absorbance using a UV-VIS spectrophotometric with a wavelength at 540 nm.
3. Results and discussions

3.1. Effect of reaction time of hydrolysis process on glucose production

Figure 2 shows the effect of reaction time on glucose concentrations formed from the hydrolysis process.

![Figure 2. Effect of reaction time of hydrolysis process on glucose production.](image)

From Figure 2 it can be seen that the graph has increased where the longer reaction time, the more glucose was formed. This is following the theory of Muhaimin [8] which says that the longer the hydrolysis time, the glucose content in hydrolysis products will increase because the contact between reactants will be more frequent and can increase the conversion of reactants into products. But on the graph 0 N, 100 W fluctuations occurred at 25, 30 and 35 minutes, this can be caused by a less homogeneous sample with DNS reagents so that the absorbance measured in the spectrophotometer is less precise.

3.2. Effect of sulphuric acid concentration on glucose production

Figure 3 shows the effect of sulphuric acid concentration on glucose concentration formed from the hydrolysis process.

![Figure 3. Effect of sulphuric acid concentration on glucose production.](image)

From Figure 3 it can be seen that the use of 0.25 N sulphuric acid produces higher glucose. The reaction between water and starch runs so slowly so the reaction needs a catalyst which is sulphuric acid to increase the rate of hydrolysis [9]. Hydrolysis using acids is divided into two groups that are
concentrated-acid hydrolysis and dilute-acid hydrolysis. This study using dilute acid hydrolysis because there are several advantages such as low acid consumption and no need for acid recovery [10].

3.3. Effect of microwave power on glucose production

Figure 4 shows the effect of microwave power on glucose concentrations formed from the hydrolysis process.

![Graph](image)

**Figure 4.** Effect of microwave power on glucose production.

From Figure 4, it can be seen that the use of microwave power of 300 W produces glucose higher than the use of microwave power 100 W. Microwave energy is used as an alternative to heating because microwave energy has several advantages such as improving product quality and has high energy efficiency [11]. Microwave power is proportional to temperature, where the higher the power used, the operating temperature will also be higher. The effect of the temperature follows the Arrhenius equation where high temperatures can increase the reaction rate, this is caused by the increasing number of sugar monomers which are released from the bonds as the temperature increases, the yield of glucose yields is even higher [12].

4. Conclusions

The glucose concentration obtained from the hydrolysis process can be influenced by reaction time, sulfuric acid concentration, and microwave power. Where the concentration of glucose will increase with increasing reaction time, the concentration of sulfuric acid and microwave power. The best concentration obtained is 23.038 ppm at an acid concentration of 0.25 N microwave power 300 W and 60 minutes.

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