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# Microwave Assisted Hydrolysis of Oil Palm Empty Fruit Bunch: Effect of Wave Distance Sources, Power, Time and Acid Concentration

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**Abstract.** Hydrolysis of EFB's fiber was carried out using sulfuric acid in two series reactors each operates under microwave irradiation. First reactor operates at various microwave power level (medium, medium high and high), irradiation time (5, 7.5 and 10 minutes) and acid concentration (0.5, 1 and 1.5%) while the second reactor operates at constant time using similar combination of power and acid concentration. This study placed reactor A next to the waveguide, while the reactor B on the opposite position. Microwave energy emitted from magnetron into cavity through the waveguide. This study evaluated effect of wave distance source on temperature and glucose yield in both reactors. Effect of microwave power, irradiation time and acid concentration and their relationship with glucose and temperature were investigated. Overall, increment of time and power increased yield of glucose at first hydrolysis using sulfuric acid of 0.5%. Glucose yielded from reactor A usually higher as compared to reactor B. Increment acid concentration reduced the yield except the one with high power irradiation. This study observed that second stage hydrolysis had no significant contribution to add the yield.

## 1. Introduction

Bioethanol is an alternative fuel that reduce greenhouse gas emissions by up to 75%. The most crucial issue for bioethanol production is selection of raw materials with respect to economic and environmental consideration. Biomass from agricultural and industrial waste can be used for bioethanol production because the source is abundant, cheap, and does not interfere with the availability of food [1, 2]. One of potential industry as a source of biomass is palm oil mill.

Production of palm fruit into palm oil contributes to several pollution problems namely palm oil mill effluent (POME), gas emission and biomass from steam batch sterilization, boiler operation, threshing, bunch digestion and pressing respectively. Biomass of palm oil mill comprises of empty fruit bunch (EFB), fiber and frond in abundant amount. They all categorized as lignocellulosic materials containing 50% cellulose, 25% hemicellulose and 25% lignin in their cell wall [3, 4]. Even though palm oil mill is implementing best practice waste management and some of those typical wastes can be reused and recycled such as utilization of POME for land application in oil palm plantation and EFB for composting. EFB is lignocellulosic material comprises of 37.1% glucose, 20.7% hemicellulose, 22.2% lignin, 1.3% ash, and 3.5% extractive substances [5]. Problem arising from composting of EFB in palm oil mill is composting performs very slow. Digestion rate requires 3 to 4 weeks to complete the fermentation while

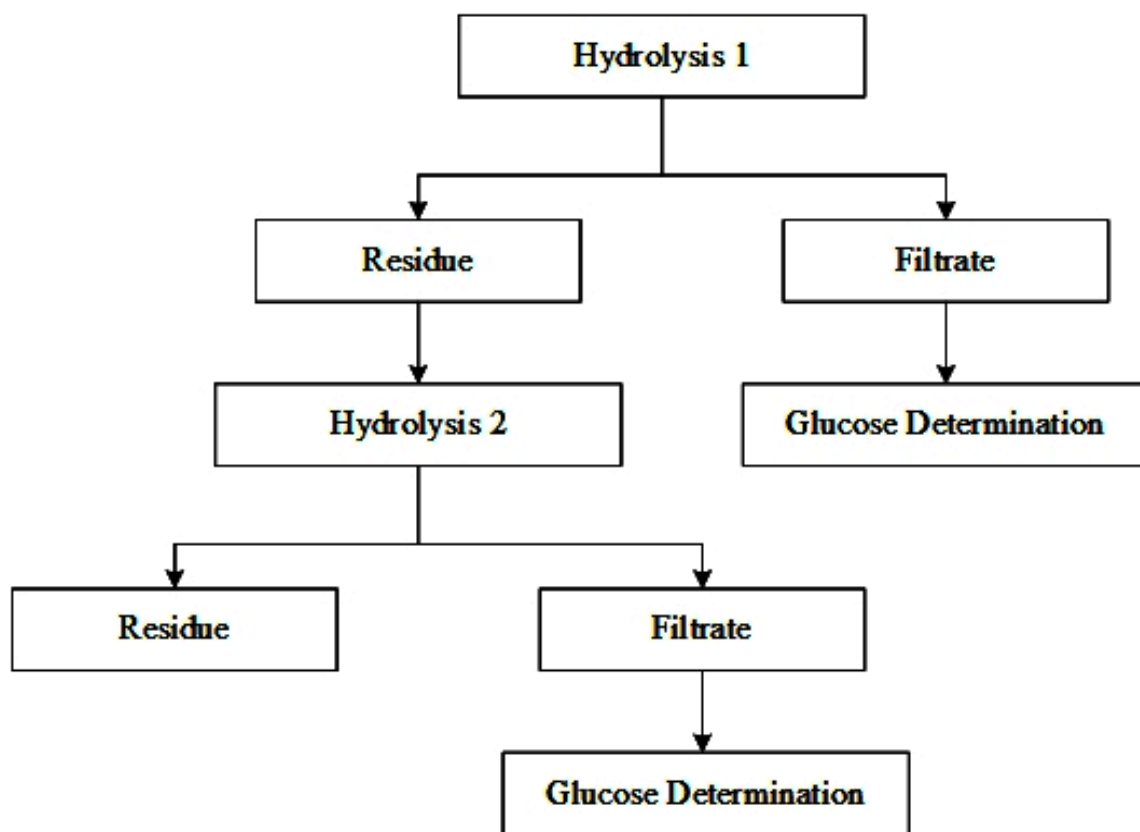


palm oil production rate is very high. This situation results abundant pile of EFB presence around the mill. With respect to those huge quantity of EFB and effort to improve economic value of EFB conversion, investigation to evaluate EFB potency to produce bioethanol is conducted. This conversion carried out through hydrolysis of EFB and glucose fermentation for bioethanol production.

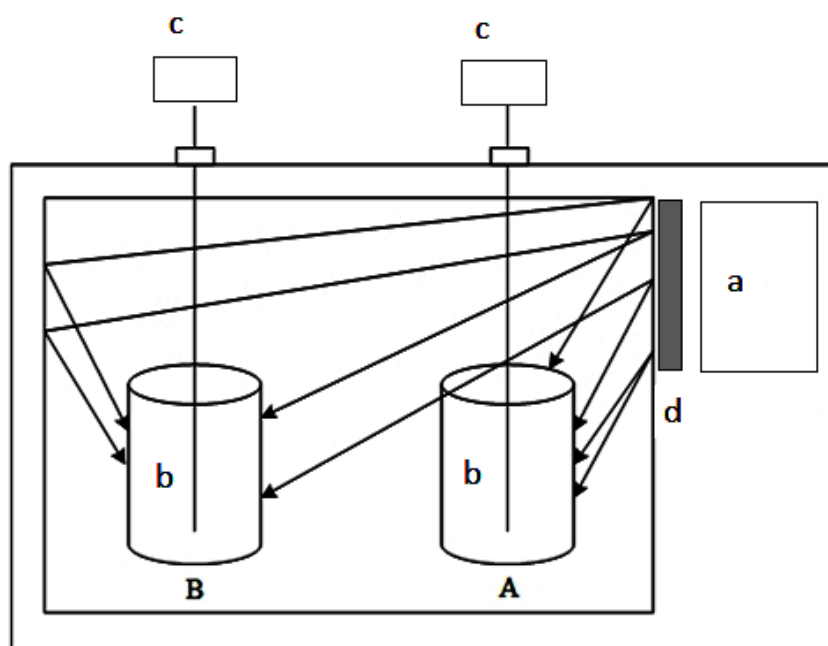
Unlike hydrolysis with dilute acid to treat lignocellulosic materials that previously reported elsewhere [6], this study investigates microwave assisted hydrolysis of EFB to produce glucose. Glucose will be fermented further to produce bioethanol. Several studies reported utilization of microwave irradiation as an effective heating method either alone or in combination with other chemicals due to high heating efficiency, uniformity, and short processing times [7, 8, 9, 10, 11]. Microwaves enhances lignin removal and facilitates cellulosic and hemicellulose ultrastructure conversion thereby increasing cellulosic accessibility [12, 13]. When the treatment provides a more effective hydrolysis process in converting cellulose to glucose for further ethanol production, less cellulose is available to produce nanocellulose or other components [14]. During hydrolysis process, microwave irradiation facilitates the loosen of cellulose bonds and release lignin from lignocellulosic biomass. Microwaves increase the temperature and structure of cellulose through the expansion of water molecules, decrease cellulose crystallinity and increase the efficiency of hydrolysis [15]. The principle of heating occurs because microwaves induce substances in lignocellulose biomass which cause resonance and vibration in high frequencies. Very fast friction causes the temperature to increase rapidly in just minutes [16]. Sarah et al. (2017) reported hydrolysis of EFB using sulfuric acid assisted by microwave irradiation. Increment of microwave power from medium to high level during exposure and prolong heating from 5 to 10 min elevated final temperature of hydrolysis process approximately 97 to 105°C [17]. In this study, investigation on the effect of microwave irradiation on hydrolysis of EFB using sulfuric acid was carried out to determine glucose concentration as raw material for bioethanol production.

## 2. Materials and Methods

The materials in this study were oil palm empty fruit bunch (EFB), sulfuric acid, and DNS (3,5-Dinitrosalicylic acid). Prior hydrolysis process, EFB was washed, dried under the sun, cut into fibers, and stored in a dry container. Hydrolysis of EFB with sulfuric acid performed in two stages each assisted by microwave irradiation to obtain temperature for hydrolysis as shown in Figure 1. Temperature during hydrolysis measured by using thermocouple K (Krupp and Closs size diameter of 3 x 300 mm (Mineral Insulated) C/w Cable 2 m) connected with thermo controller (Shimaden). First stage hydrolysis carried out at various acid concentration of 0.5 to 1.5%, various level of microwave power (medium, medium high, and high) and irradiation times from 5 to 10 min. Liquid from first stage hydrolysis process was separated from their residue and was taken for glucose determination using DNS method and UV-VIS Spectrophotometer at a wavelength of 550 nm, while residue fed into second stage hydrolysis process. The second reactor operates at constant acid concentration using similar combination of power and time with experimental design of first hydrolysis. Similar glucose procedure determination was repeated for liquid obtained from second hydrolysis. In this study, reactor was located near (A) and far (B) from the waveguide as source of microwave and evaluated effect of wave distance source on temperature and glucose yield. Experimental rigs and reactors position in the microwave cavity during irradiation are shown in Figure 2.



**Figure 1.** Scheme of experimental procedure



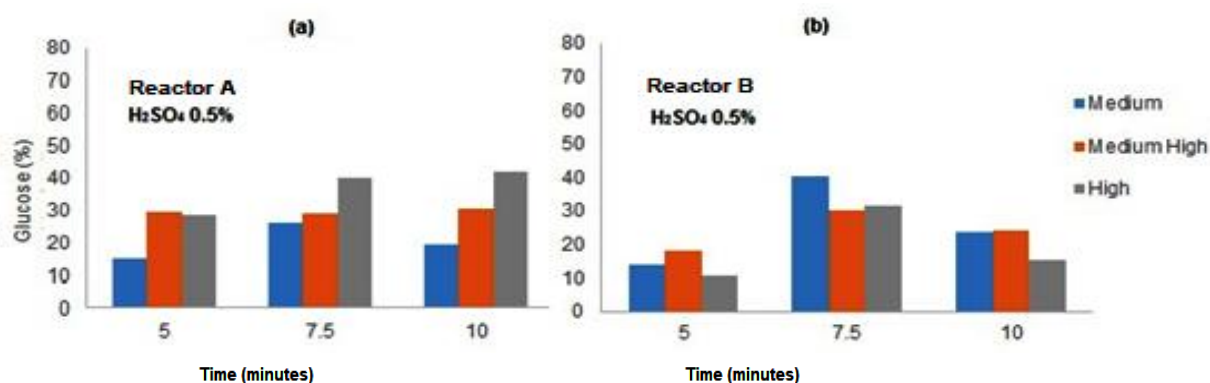
**Figure 2.** Experimental rigs consists of (a) magnetron (b) reactor with thermocouple (c) thermo controller (d) waveguide

### 3. Results and Discussions

#### 3.1 Effect of wave distance source, power, time and acid concentration during 1<sup>st</sup> hydrolysis on glucose concentration

Effect of time, microwave power and acid concentration on glucose concentration inside reactor located near and far from the waveguide for first hydrolysis are shown in Figure 3. Overall, increment of time and power increased the glucose concentration, excluded glucose concentration from treatment at reactor B after exposure for 7.5 min. This study observed glucose obtained from first hydrolysis at medium power as depicts in Figure 3(a) increased from 15.36% and 26.22% as time extended from 5 to 7.5 min respectively. On the contrary, prolong hydrolysis period from 7.5 to 10 min at constant microwave power decreased glucose concentration fall to 19.55%. Similar phenomena observed also for time extension reactor B treatment as depict in Figure 3(b). Overall, increment of power from medium to medium high and high increased concentration of glucose along hydrolysis period exclude glucose obtained from treatment combination of irradiation using medium power for 7.5 min. The resulting glucose in reactor A increased from 29.82% to 30.56% and from 28.35% to 42.19% respectively as time for hydrolysis extended from 5 to 10 min. It was observed that glucose concentration obtained from hydrolysis at reactor A higher as compared to reactor B. It indicates wave distance source affected glucose concentration due many waves penetrated directly into the reactor A in very short time while wave in reactor B required more time to penetrate into EFB because some of the waves had to be reflected from cavity wall across the waveguide.

The effect of wave distance source to glucose concentration for irradiation using sulfuric acid concentration of 1 and 1.5% are shown in Figure 4. Glucose concentration as depicted in Figure 4(a) and 4(b) was influenced by wave distance source. EFB hydrolysis in reactor A yielded glucose higher than reactor B if hydrolysis performed for maximum 7.5 min at power of medium and medium high level. Similar phenomena observed from Figure 4(c) and 4(d) with respect to wave distance source aspect. Increment of acid concentration from 0.5% as depicted in Figure 3 to acid concentration of 1 and 1.5% as shown in Figure 4 reduced the concentration of glucose except the one with power of medium treatment. Figure 4(c) and 4(d) shows glucose only obtained from treatment with power of high level. This indicated concentration of sulfuric acid more than 0.5% should performed with combination of high power and high concentration of acid to attain temperature of hydrolysis. Lower power failed to facilitate hydrolysis of EFB with higher acid concentration.

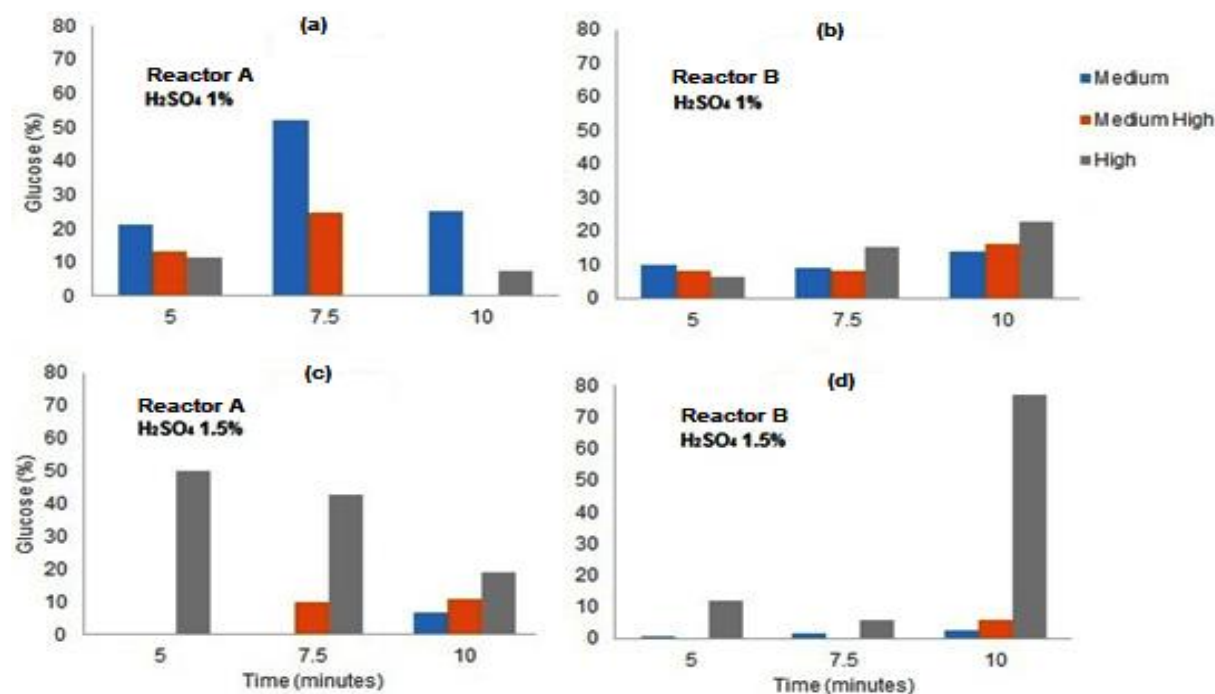


**Figure 3.** Resulting glucose obtained from 1<sup>st</sup> hydrolysis using 0.5% sulfuric acid in reactor A and B

### 3.2 Effect of wave distance source, time and power during 2<sup>nd</sup> hydrolysis on glucose concentration

This study carried out second hydrolysis to investigate remaining glucose in the residue from first hydrolysis. The 2<sup>nd</sup> hydrolysis aimed to evaluate the effects of wave distance sources, time and microwave power to glucose concentration. Figure 5 shows glucose concentration obtained after hydrolyzed residue from 1<sup>st</sup> hydrolysis in reactor A and B respectively at constant acid concentration (0.5%). The Figure 5 illustrates possible indication to conduct two stage hydrolysis of EFB with the aims to increase the yield total glucose but difficult to make any conclusion on this observation data due to inconsistency trend of resulting glucose obtained in the 2<sup>nd</sup> hydrolysis.

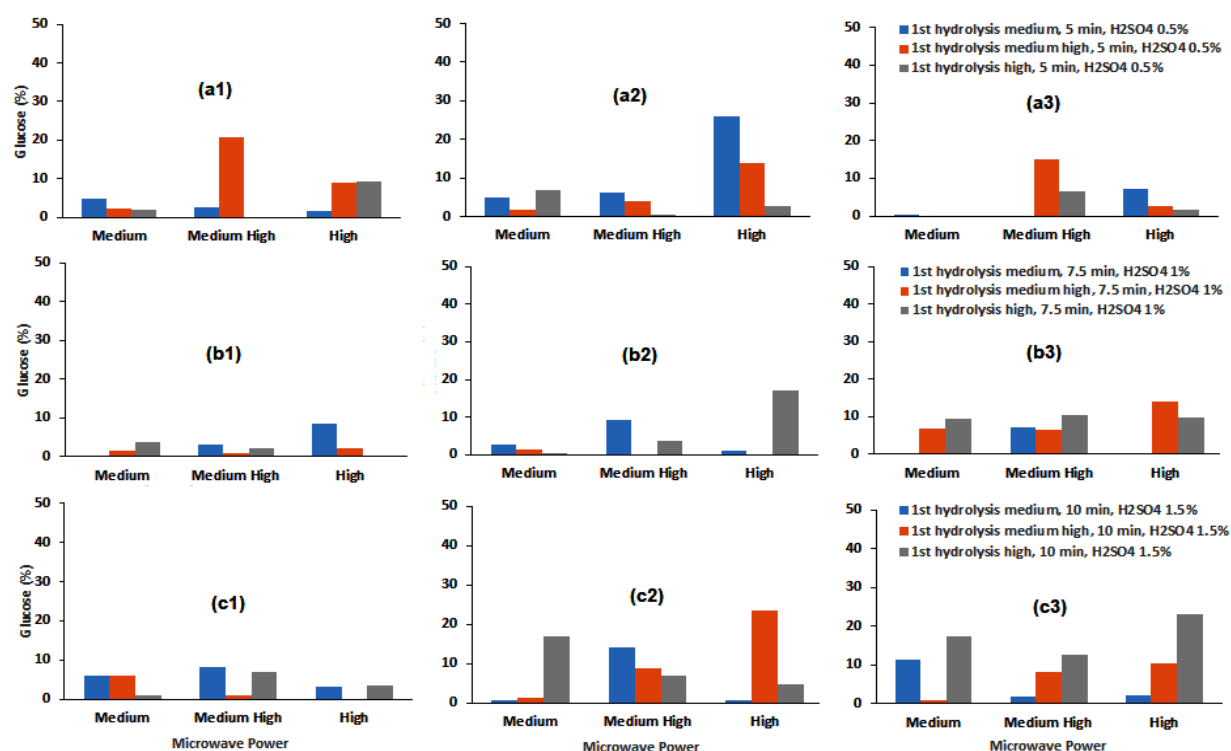
Figure 5(a1), Figure 5(a2) and Figure 5(a3) show performance of samples originated from residue obtained after hydrolysis for 5 min in the previous stages. Increment of time from 5 to 7.5 and 10 min as depict in Figure 5(a1), Figure 5(a2) and Figure 5(a3) have not significant effect to resulting glucose at elevated microwave power. This study observed similar phenomena also occurred during hydrolysis of samples originally came out from hydrolysis for 7.5 min in the 1<sup>st</sup> stages as depict in Figure 5(b1), Figure 5(b2) and Figure 5(b3). Excluded hydrolysis for 7.5 min, this study observed every treatment combination resulted glucose. Some of those treatment resulted glucose concentration higher than their previous hydrolysis. This explain residue from 1<sup>st</sup> stage hydrolysis still contained residual cellulosic and hemicellulose that converted further into glucose contents in 2<sup>nd</sup> stage hydrolysis. Figure 5(c1) to Figure 5(c3) show effect of time to glucose concentration for some treatments such as treatment using microwave power of medium high. Time increment for this particularly from 5 to 10 min increased glucose concentration from 10 to 33%. This study observed increment of acid concentration influenced hydrolysis of 2<sup>nd</sup> stage. Overall concentration increment from 0.5 to 1% increased the concentration of glucose and decreased at concentration of 1.5%.



**Figure 4.** Resulting glucose obtained from 1<sup>st</sup> hydrolysis using sulfuric acid of 1 and 1.5% in reactor A (a and c) and reactor B (b and d)

Comparing glucose obtained in reactor A and reactor B as depict in Figure 5, hydrolysis performed in reactor A resulted higher glucose concentration as compared to reactor B's results. This study

proposed intensity of microwave energy headed for reactor B lower than to reactor A during their penetration into material in reactor B and A respectively. Reactor located near wave sources received more microwave energy and dielectric materials (residues and acid solutions) inside the reactor absorbed microwave energy and converted it into thermal energy. Overall final glucose concentration influenced by wave distance source. Reactor on the position near the waveguide (reactor A) will obtained higher glucose than reactor B that located quite far from the waveguide.



**Figure 5.** Resulting glucose obtained from 2<sup>nd</sup> hydrolysis using sulfuric acid of 0.5% in reactor A for 5 min (a1, b1, c1); 7.5 min (a2, b2, c2) and 10 min (a3, b3, c3)

#### 4. Conclusion

Microwave energy can be used to assist 2 stages hydrolysis of palm oil EFB. Overall yield of glucose obtained from first hydrolysis increase at elevated time and power after irradiated by microwave energy in sulfuric acid of 0.5% solution. Reactor A usually resulted higher glucose as compared to reactor B, but treatment at elevated acid concentration reduced the yield except the one with high power irradiation. No significant contribution of second stage hydrolysis to overall glucose in this study. The maximum resulting glucose from first hydrolysis was 77% obtained from hydrolysis performed at high microwave power using sulfuric acid of 1.5% for about 10 minutes. Irradiation proceeded as microwave emitted from waveguide into the reactor that placed far from the waveguide. On the contrary, second hydrolysis yielded glucose of 43% after irradiated fiber residue for 5 minutes using sulfuric acid of 0.5% and microwave power of medium high.

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