

Study on Hydrolysis of Oil Palm's Empty Fruit Bunch using Microwave Irradiation

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Abstract: This study investigates microwave application to assist hydrolysis of Oil Palm's Empty Fruit Bunch (OPEFB) for the production of bioethanol. Hydrolysis was carried out by two steps microwave irradiation to maximize yield of glucose. On the 1st hydrolysis, microwave energy irradiates OPEFB fibre at various concentrations of H_2SO_4 (0.5 and 1%) and level of microwave power (medium, medium high and high). Residue from 1st hydrolysis was irradiated on 2nd hydrolysis process. Temperature profile and moisture loss during hydrolysis process were observed to study heat generation inside the reactor. Glucose resulted from each process was determined by DNS method using UV-VIS spectrophotometric. Overall hydrolysis of OPEFB yielded glucose ranged between 2-47%.

Key words: Microwave, hydrolysis, oil palm's empty fruit bunch, lignocellulose, glucose, DNS method, Medan

INTRODUCTION

Palm oil mill disposes Oil Palm's Empty Fruit Bunch (POEFB) as solid waste. The POEFB consist of cellulose (45.95%), hemicellulose (22.84%), lignins (16.49%), ash (1.23%), nitrogen (0.53%) and oil (2.41%) (Haryati and Elisabeth, 2003). Cellulose is composed of β -D-glucose units which are linked together with 1, 4-glycosidic bonds to form long linear chains. These chains further aggregated via hydrogen bonds to form a rigid crystalline structure that is resistant to hydrolysis. On the contrary, hemicellulose is composed of short and highly branched copolymers of glucose, mannose, galactose, xylose and arabinose and its branched structure makes it relatively easy to be hydrolysed to its monomeric components (Jonsson and Martin, 2016). This background of material characteristic makes hydrolysis of POEFB to extract glucose from cellulose is difficult to conduct. Basically hydrolysis may occur through heating process to release glucose from cellulose matrix at acidic environment (pH ranged between 2-4). Thermal hydrolysis of lignocellulose material by weak or strong acid may extract glucose at temperature range from 185-210°C. This process is energy intensive involving thermal processing for approximately 30 min. To improve hydrolysis of lignocellulose material, microwave may be used to generate heat that is needed to release glucose from cellulose matrix. Similar method reported by Klein *et al.* (2016) when hydrolyse *Ficus religiosa* leaves to produce glucose for bioethanol production.

Microwave irradiation had been proven to be effective to heat materials (Lau and Tang, 2002;

Schneider *et al.*, 2005; Sun *et al.*, 2007; Coronel *et al.*, 2008). Terminology of microwaves is defined as the electromagnetic waves with the wavelengths between 1 mm and 1 m or frequencies ranging from 0.3-300 GHz (Iwaguch *et al.*, 2002; Khraisheh *et al.*, 1997). Microwave irradiation may be used to hydrolyse POEFB. Transformation the electromagnetic energy into thermal energy during hydrolysis process occurs in material (POEFB fibre on acid solution) inside the reactor. Hydrolysis by microwave irradiation has advantages in reducing time and lowering temperature for hydrolysis. Fundamentally, heat generation occurs at time microwave penetrates into dielectric material. Parameters that indicates ability of dielectric material to absorb microwave energy is dielectric constant and dielectric loss factor indicates conversion of microwave energy into thermal energy. In this study, heat generation comes from medium consist of POEFB and sulphuric acid solution. No study, reported dielectric properties of fibre POEFB so far but (Sukaribin and Khalid, 2009) reported dielectric properties of abscission zone of Palm Oil Fresh Fruit Bunch (POFFB) greater than mesocarp of oil palm fruit. Dielectric constant and dielectric loss factor of POFFB at abscission zone were reported, respectively ranged between 20-50 and 10-25. Ratio of dielectric loss factor to dielectric constant is approximately 0.5. This ratio show 50% of energy absorbed by POEFB sample is converted into thermal energy. This fact shows there is potency of microwave energy to assist hydrolysis of POEFB. The aim of this research is to study the hydrolysis of POEFB using microwave irradiation. In this study, heat generation was indicated from moisture loss during hydrolysis process.

MATERIALS AND METHODS

Preparation of POEFB: POEFB that taken from palm oil mill was washed, dried, cut into fibres. The fibres were kept in dry container for further process.

Hydrolysis of POEFB: POEFB was hydrolysed using H₂SO₄ at various concentrations (0.5-1.5%) in a reactor assisted by microwave oven (R-249 IN (S))/(W). Magnetron inside microwave oven generates electromagnetic wave to irradiate POEFB and H₂SO₄ inside the reactor. Hydrolysis was carried out into 2 steps. The 1st hydrolysis occurred for about 5-10 min using microwave power of medium, medium high and high. Residue from 1st hydrolysis was reused for 2nd hydrolysis. Temperature during hydrolysis was measured using thermocouple type K (Krupp and Closs size diameter of 3×300 mm (Mineral Insulated) C/w Cable 2 m) connected with thermo controller (Shimaden). The liquid separated from residue of each process were collected and measured to determine moisture loss.

Determination of moisture loss: Moisture loss is determined by the gravimetric method (wet basis). The percentage of moisture loss during hydrolysis process was determined as ratio of moisture loss toward initial moisture.

Determination of reducing sugar: After hydrolysis step, sample was filtered to separate the filtrate and residue. Residue from 1st hydrolysis was reused for 2nd hydrolysis while filtrate was collected to determine reducing sugar. Reducing sugar concentration was determined using UV-VIS spectrophotometer. The measurement was taken at 550 nm of the absorbance of a homogenized and diluted sample. Prior the sugar determination, sample preparation was carried out using DNS method.

RESULTS AND DISCUSSION

Effect of microwave power and acid concentration on heat generation during 1st hydrolysis: Sample is a dielectric material which contains water of approximately 85%. During hydrolysis process, generation of heat occurs after re-orientation of water molecules inside the sample when microwave energy penetrates into the sample. Number of microwave energy converted into thermal energy can be estimated from moisture loss during irradiation period. Heat transfer process was indicated by increment of temperature from room temperature to material's boiling point. In this study, microwave powers arranged into level power of medium, medium high and high, to study effect of power on hydrolysis process. Furthermore, H₂SO₄ concentrations (0.5 and 1%) were

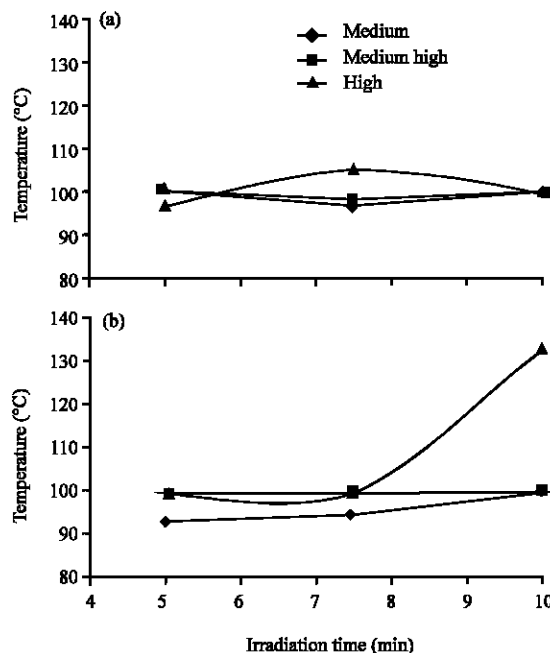


Fig.1: Effect of microwave power on 1st hydrolysis final temperature at various acid concentrations: a) H₂SO₄ 0.5% and b) H₂SO₄ 1%

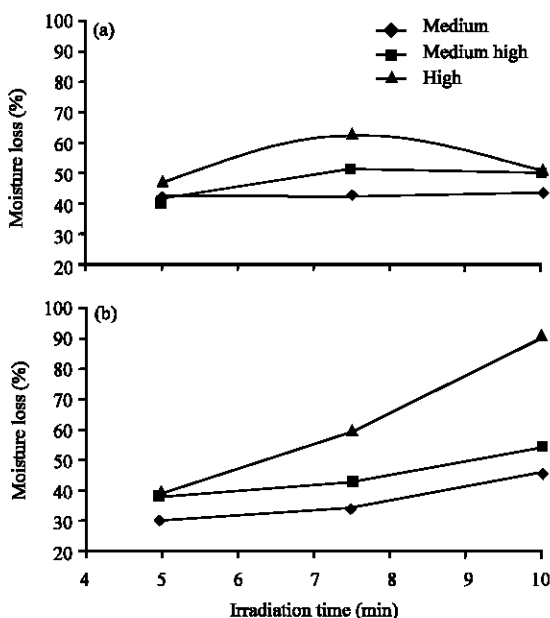


Fig. 2: Effect of microwave power and acid concentration on moisture loss during 1st hydrolysis at various acid concentrations: a) H₂SO₄ 0.5% and b) H₂SO₄ 1%

examined to study the effect of acid concentration as well while irradiation time varied from 5-10 min. Figure 1 and 2 show the effect of microwave power and acid

concentration during irradiation of POEFB in acid solution. Overall, power increment from medium to high level and time extension for microwave irradiation elevate final temperature of hydrolysis process, not include hydrolysis using 1% H₂SO₄ at medium high power level. The final temperatures of hydrolysis at various irradiation time (5, 7.5 and 10 min) observed similar at approximately 100°C. Observation on hydrolysis using H₂SO₄ at concentration of 0.5% with high power level showed that prolonged heating from 5-7.5 min elevate final temperature approximately 97-105°C. Furthermore, extending irradiation time from 7.5-10 min reduce final temperature from 105-99°C. On the contrary, hydrolysis using H₂SO₄ at concentration of 1% with high power level, elevate final temperature from 99-133°C. The rise of final temperature for high level of power as depicted in Fig. 1b shows phenomenon of thermal runaway. Thermal runaway is uncontrolled temperature's rise that affected quality of product or destruct the composition of material (Clemens and Saltiel, 1996).

Temperature is an indicator of occurrence of generation of heat inside the hydrolysis reactor which indicated by increment of moisture loss during hydrolysis process as shown in Fig. 2. Moisture loss represents the number of water molecules which vaporized after electromagnetic wave absorption that followed by re-orientation of water molecules. Friction force between each molecule during re-orientation process may generate heat. The fact increment of microwave power elevate final temperature of hydrolysis as depicted in Fig. 1 was supported by increment of moisture loss during hydrolysis process that observed increasing as well. Loss of moisture may determine rate of vaporization during hydrolysis. Similar conclusion was deduced from hydrolysis at various acid concentrations. The temperature rises was promoted by water vaporization. Increment of acid concentration and microwave power level elevate rate of vaporization. This rate of vaporization had close relationship with rate of dis-orientation of polar water molecule inside the reactor which increases temperature of POEFB in acid solution.

Reducing sugar concentration resulted from 1st hydrolysis observed increase if irradiation time extend from 5-10 min. Similar situation was observed if level of microwave power increases. Hydrolysis at high power level yielded higher sugar concentration as compared to lower level of power. On the contrary, increment of acid concentration decreased the sugar concentration. Overall combination of H₂SO₄ 0.5%, level of power high and irradiation time of 10 min yielded sugar concentration of 47%.

Effect of microwave power on heat generation during 2nd hydrolysis:

In order to evaluate effect of microwave power on heat generation process during 2nd hydrolysis, residue of 1st hydrolysis was irradiated by microwave energy for 5 min in acidic environment using H₂SO₄ of 0.5%. The microwave power was adjusted at various levels from medium, medium high and high level. Final temperature and moisture loss observed at each combination of 1st hydrolysis and 2nd hydrolysis as shown in Fig. 3 and 4. Figure 3 shows effect of microwave power on 2nd hydrolysis final temperature. Samples of 2nd hydrolysis were residues of 1st hydrolysis. Overall, temperatures of 2nd hydrolysis observed ranged between 80-100°C, excluding temperature that is shown in Fig. 3a (medium), Fig. 3d (medium high and high) and Fig. 3e (medium high). Figure 3ab shows level of the microwave power during 2nd hydrolysis has no significant effect on 2nd hydrolysis final temperature. It can be deduced that heating residue (that had been irradiated by microwave energy for 5 and 7.5 min) at various level of microwave energy affected almost water molecules in acid solution.

The residue contains lower moisture because water content in the residue almost vanish during 1st hydrolysis (Fig. 2). Only small part of water molecules inside the residue being affected by microwave energy. Increment of acid concentration from 0.5-1% as depicted in Fig. 3d-f reduced final temperatures of 2nd hydrolysis, excluding hydrolysis of residues from samples irradiated by high power level. Overall only re-irradiation of residues from hydrolysis at high power level as depicted in Fig. 3 may achieve temperature of 100°C on 2nd hydrolysis. Prolong heating from 5-10 min in the 1st hydrolysis level had no significant effect to final temperature of 2nd hydrolysis but it affects the moisture loss. The moisture losses during 2nd hydrolysis as depicted in Fig. 4 were observed range between 15-32%. The level increment of microwave powers from medium level to high during 2nd hydrolysis showed fluctuation of moisture loss of residue, depending on combination of level of microwave power and irradiation time from process combination on 1st hydrolysis. Moisture loss only affected by increment of acid concentration as depicted in Fig. 4.

Increment of acid concentration reduces moisture loss of residue from average 20% into 1-5% (minimum value). This explained, irradiation of residues from sample that hydrolysed with 1% acid solution using medium and medium high power level for 5-10 min obtain final temperature below 100°C (Fig. 3d-f), compare with (Fig. 4d-f). Lower moisture loss indicates low heat generation during 2nd irradiation or 2nd hydrolysis.

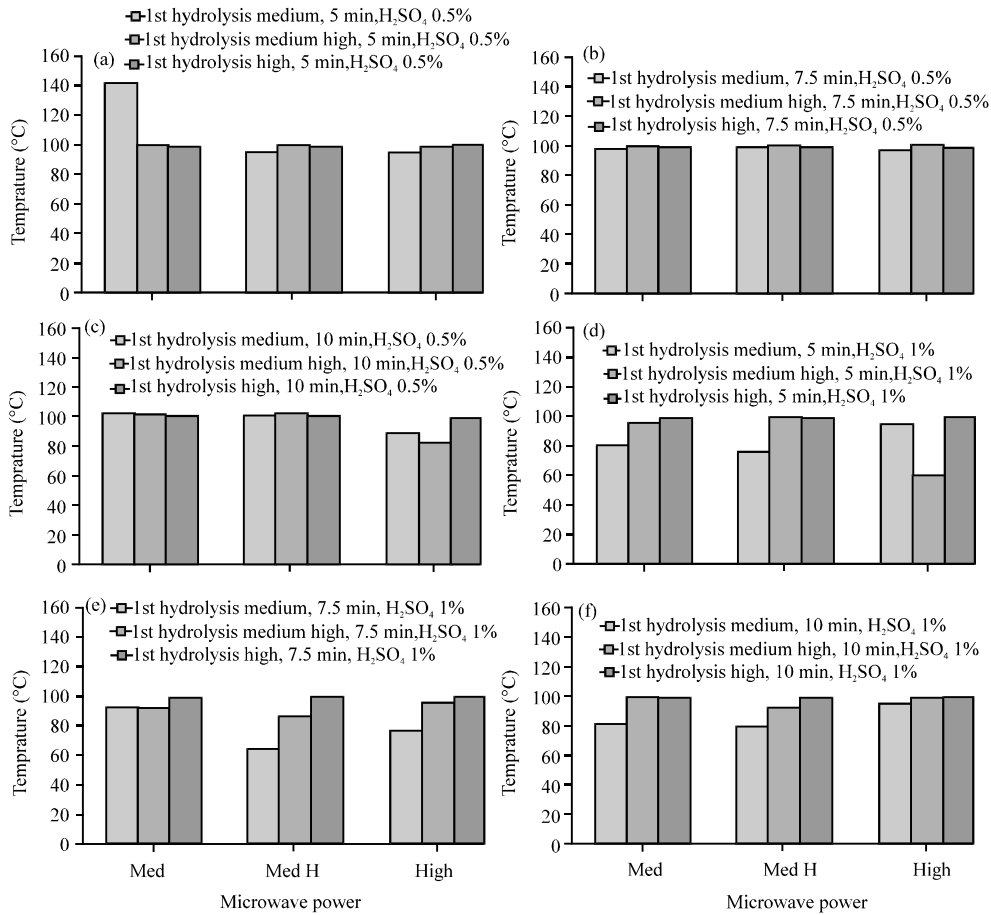


Fig. 3: Effect of microwave power on 2nd hydrolysis final temperature using residue from various combination of 1st hydrolysis: a) 5 min and H₂SO₄ 0.5%; b) 7.5 min and H₂SO₄ 0.5%; c) 10 min and H₂SO₄ 0.5%; d) 5 min and H₂SO₄ 1%; e) 7.5 min and H₂SO₄ 1% and f) 10 min and H₂SO₄ 1%

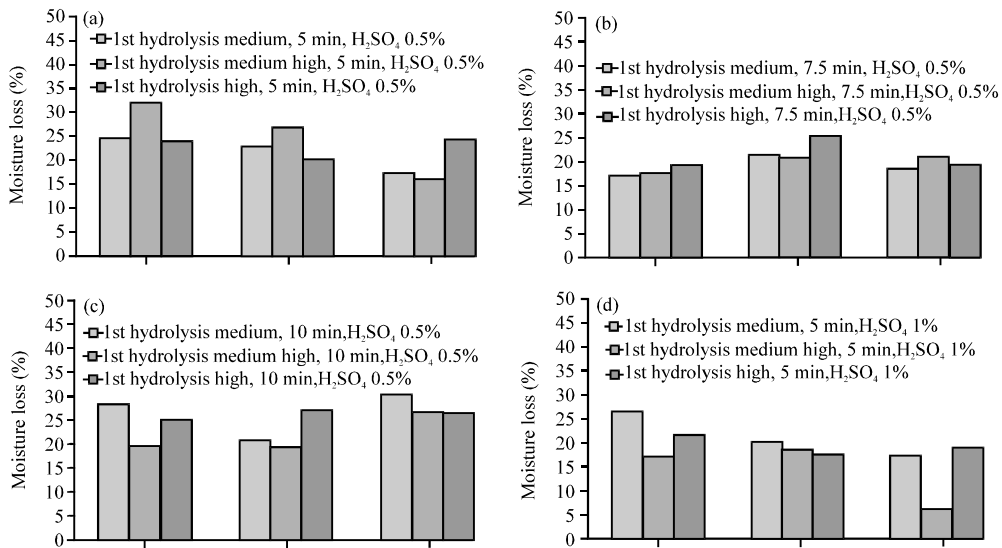


Fig. 4: Continue

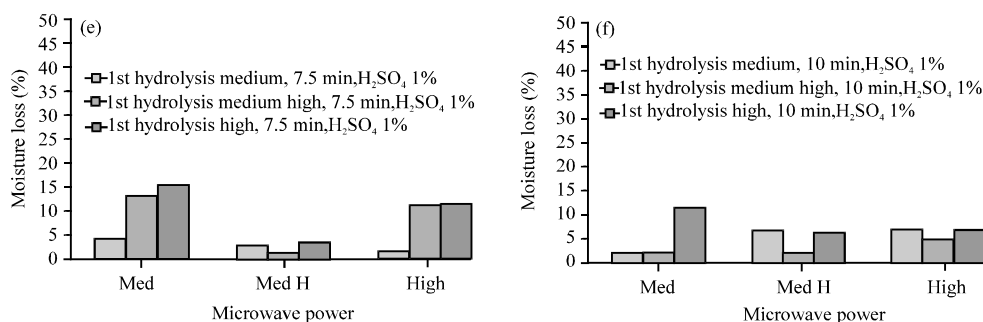


Fig. 4: Effect of microwave power on moisture loss of 2nd hydrolysis using residue from various combination of process on 1st hydrolysis: a) 5 min and H₂SO₄ 0.5%; b) 7.5 min and H₂SO₄ 0.5%; c) 10 min and H₂SO₄ 0.5%; d) 5 min and H₂SO₄ 1%; e) 7.5 min and H₂SO₄ 1% and f) 10 min and H₂SO₄ 1%

The reducing sugar resulted from 2nd hydrolysis observed higher if level of microwave power increases from medium level to high level. The reducing sugar approximately 50% lower as compared to reducing sugar yielded from 1st hydrolysis. Overall combination of H₂SO₄ 0.5%, level of power high and irradiation time of 10 min at 1st hydrolysis yielded sugar concentration of 23% at 2nd hydrolysis.

CONCLUSION

Microwave irradiation may be used to assist hydrolysis of POEFB into glucose. Hydrolysis process was influenced by acid concentration, microwave power and irradiation time. Combination of H₂SO₄ concentration of 0.5%, level of microwave power high and irradiation time of 10 min on 1st hydrolysis yielded sugar concentration of 47% while 2nd hydrolysis yielded glucose concentration of 23% as the reducing sugar.

ACKNOWLEDGEMENT

The researchers thank to University of Sumatera Utara for financial support on this research project under Scheme Project BBPTN FY of 2016.

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