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Research Article

Hydrolysis of Abundant Biomass in Thailand by a Combination of Microwave Irradiation and a Homogeneous Acid Catalyst HCl

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Abstract: Thailand produces many important agricultural products. The productions generate large amounts of cellulose wastes, especially rice straw, palm leaf, and bagasse, which are Thailand's most abundant agricultural wastes. Cellulose is the most common polysaccharide structure abundantly found in almost any plant cell wall. Cellulose is a polymer of glucose units. Upon breaking down by hydrolysis, cellulosic wastes may serve as low-cost starting materials to produce soluble carbohydrates, which are high-value-added edible fibers or glucose. The feasibility of the hydrolysis facilitated by microwave irradiation and chemical catalyst to hydrolyze biomass into soluble carbohydrates is evaluated in this study compared to the typical edible cassava starch. The key strategy relies on microwave irradiation as a heating source to shorten the reaction time. The amounts of solid loss, reducing sugar, and total carbohydrates obtained from the hydrolysis of each biomass are evaluated. Bagasse gave the highest yields of soluble carbohydrates. The reaction temperature and HCl concentration have significant effects on the hydrolysis. The higher reaction temperature and HCl concentration led to a higher solid loss. However, the resulting soluble carbohydrates tend to be partially decomposed. Thus, the medium HCl concentrations at 0.6 M or 0.8 M and the lower reaction temperature at 100 °C gave the highest reducing sugar. The results reveal a potentially efficient method to valorize abundant agricultural wastes in Thailand.

Keywords: Palm leaf, Bagasse, Cellulose, Hydrolysis, Microwave irradiation

1. Introduction

Thailand is one of the most important agricultural countries in the world. Agricultural goods are among the major exports of Thailand. Consequently, Thailand has plenty of different agricultural waste biomass [1]. Plant biomass is organic materials that are renewable and sustainable sources [1-11]. Biomass may potentially be used as raw materials to be converted into chemical commodities which are much more valuable. Cellulose is abundantly found in nature as the major structure in plant cell walls. It is the main component of wood, cotton, hemp, and other plant-based materials.

It can be found in many different sources, such as seed fibers, bast fibers, grasses, algae, fungi, some bacteria, and wood in the cell walls and fiber parts in plants, which is the most important source of cellulose [12]. Cellulose is made of linear polymers of β-(1, 4)-glycosidic linkages glucose repeating units. Cellulose differs from other polysaccharides because it is water-insoluble and has a rigid structure. It is a renewable resource, biodegradable and non-toxic material. Cellulose is mainly found together with lignin and hemicelluloses. Sugarcane is the principal industrial crop in many counties. Sugarcane bagasse is typically composed of cellulose (25 ± 2.7%), hemicellulose (48 ± 2.3%), lignin (31 ± 1.2%), and ash (0.83 ± 0.03%) [13]. Oil palm (Elaeis guineensis), a persistent monocot in the Palmae family and Cocoineae tribe, is a vital vegetable oil feedstock. Rice is one of Thailand's important crops, so rice straw is synchronously produced. Rice straw is generally composed of cellulose (40%), hemicellulose (35%), lignin (10%), and silica (5%) [14].

The hydrolysis process [15] is the reaction that breaks down the chemical bonding of material by adding water and acid solution. Chemical hydrolysis [5,8,16-22] and enzymatic hydrolysis [23-30] are the most common techniques. Acid and base catalysts such as sulfuric and hydrochloric acid have been used in hydrolysis since 1960. The catalysts give more hydrolyzed products by increasing the rate of hydrolysis, which increases the efficiency of cleaving the glycosidic linkages. An increase in temperature and an increase in catalyst concentration can increase the rate of hydrolysis. In addition to sulfuric acid and hydrochloric acid, diluted phosphoric acid was also employed to catalyze the hydrolysis reaction [31-32]. Fontana et al. (2008) can produce glucose as a primary product from the hydrolysis of tapioca starch using dilute phosphoric acid at about 0.1% at 160 °C. This method may be an alternative to the more expensive enzymatic method. Dilute phosphoric acid can lower the concentration of the by-product, for example, hydroxymethyl furfural (HMF), compared with hydrochloric acid [32]. Amirkhani et al. (2015) used diluted sulfuric acid to hydrolyze hemicellulose to xylose and other sugars. The reaction was carried out at a temperature range from 120 – 220 °C in short reaction times. At a high temperature and longer reaction time, more by-products, such as furan compounds, weak carboxylic acids, and phenolic compounds, are produced. The amount of glucose production from oil palm was investigated by hydrolysis using dilute sulfuric acid at various temperature ranges of 100 – 400 °C, various sulfuric acid concentrations at 2 – 6%, and various reaction times (30 – 240 minutes). The results showed that, at 100 °C and 6% of sulfuric acid, the amount of glucose produced is 1.43 mg/mL, 4% at 1.48 mg/mL, and 2% at 1.26 mg/mL [33]. A higher glucose level from the extraction at 7.61 mg/mL was reported using the reaction conditions at 130 °C, 6% sulfuric acid concentration, and a reaction time of 90 minutes [34].

Microwave irradiation is a heating technique widely used in organic chemistry. Microwave irradiation provides a clean, fast, and convenient energy source [35-36]. Many chemical reactions in recent years have relied on this technology because it gives a high yield and requires a short reaction time. Microwave irradiation directly transfers microwave energy to the molecules of solvents, catalysts, and other reagent molecules in the reaction. Thus, microwave heating provides efficient internal heat in the process. Heating by microwave irradiation can improve cellulose accessibility and reactivity. Microwave heat removes lignin and hemicellulose. Bui et al. (2016) have developed a chemical method to produce fructooligosaccharides, fructose, and glucose from *Jerusalem artichoke* tuber powder by microwave irradiation heating technology with hydrochloric as a catalyst. The chemical reaction facilitated by microwave irradiation heating instead of the conventional enzymatic method can shorten the whole process and reduce the cost. The operating conditions were at a low temperature of 130 °C and the concentration of HCl was 0.05 M. The irradiation time is 15 minutes, and the reaction volume ratio to *Jerusalem artichoke* tuber powder mass is 10:1 (mL/g). The results showed that 44.7% of fructose production was obtained [37].

This research explored the feasibility of using a simple, fast, and less expensive process relying on homogeneous catalytic hydrolysis to convert abundant agricultural biomass waste in Thailand into soluble carbohydrates. The agricultural waste biomasses, including palm leaf, rice straw, and bagasse, were evaluated as cellulosic starting materials compared to typical edible tapioca starch. In this study, we applied hydrochloric acid as a catalyst because chemical hydrolysis is faster, easier to operate, and cheaper than enzymatic hydrolysis. We aim to convert cellulose into soluble carbohydrates, which are glucose and edible fibers. The soluble carbohydrates are economically valuable and important intermediates for other chemicals. The soluble carbohydrate product may be used in the food industry.

2. Methodology

2.1 Reagents

Coomassie brilliant blue G-250, 5% phenol, and 95 – 97% sulfuric acid were purchased from Merck, Germany. 3,5-dinitrosalicylic, 95% ethanol, sodium hydroxide, and hydrochloric acid were purchased from Sigma-Aldrich, USA. Potassium sodium tartrate was purchased from Ajax FineChem Pty Ltd., Australia.

2.2 Methods

Fresh biomass, including rice straw, palm leaf, and bagasse, was collected from nearby farms in Pathum Thani province, Thailand. First, each biomass was cleaned with tap water to remove all dirt until the water was clear. To remove most of the moisture, the biomass was cut into small pieces and dried in a hot air oven at 70 °C until obtaining dried weight was constant. Dried biomass chips were ground by a household blender to reduce the size of dried biomass. The ground biomass was sieved (250 µm) to obtain fine biomass (Figure 1(a)).

Microwave irradiation and a homogeneous catalyst were applied for physiochemical hydrolysis. The reaction was carried out in a batch-type, closed-system microwave reactor (CEM, Discover SP 909155, USA). Before being used to remove residual moisture, fine biomass was dried in an oven at 60 °C overnight (Figure 1(b)). Then, 0.1 g of fine biomass was stirred with 1 mL HCl catalyst solution at a specified concentration. The effect of catalyst concentration on biomass hydrolysis was studied. The HCl concentrations in the hydrolysis range from 0.2 to 1.0 M with a 0.2 M increment. The reaction temperature was fixed at 100 °C, 15 minutes reaction time, and the ratio of catalyst per biomass was 10:1 (mL:g). These conditions were obtained from preliminary experiments. The effect of temperature on hydrolysis was also investigated. The reaction temperatures of 100 °C, 150 °C, and 200 °C were used in this study. The HCl catalyst concentration was fixed at 0.2 M, while the reaction time and the ratio of catalyst per biomass were the same. The reaction was done with 290 psi maximum pressure, 150 watts maximum power, and 5 minutes of ramping time. All experiments were done at least 3 times. After the reaction, the sample was removed from the reactor and left to cool down at room temperature. Deionized water (DI) 8 mL was added to the hydrolyzed sample and stirred for 1 hour to separate the soluble and insoluble parts. The solid part was collected by filtering the sample through a Whatman filtering paper No.1 on a Buchner funnel with a vacuum pump. The residual solid was dried overnight at 60 °C to determine solid loss. The hydrolyzed sample was adjusted by using NaOH to pH 7 before being kept in a refrigerator at 4 °C for further analysis.

2.3 Sample analysis

Proximate constituent analysis of biomass

The proximate constituents of rice straw, palm leaf, and bagasse were analyzed following a standard method of the Association of Analytical Communities (AOAC). Moisture, total ash, crude protein, and crude fat were measured. Moisture was measured by the hot air oven method at 100 °C for 16 – 18 h. Total ash was measured by the drying ash method at 550 °C for 4 – 6 h. Kjeldahl method with a 6.25 conversion coefficient was used to measure crude protein, and Soxhlet extraction was used to measure crude fat.

The solid loss determination (SL) was determined by Equation 1.

$$SL = \frac{IS - RS}{IS} \times 100\% \qquad (1)$$

where SL: Solid loss (% based on the dried weight of biomass),
IS: Initial dried solid (g),
RS: Residual dried solid (g)

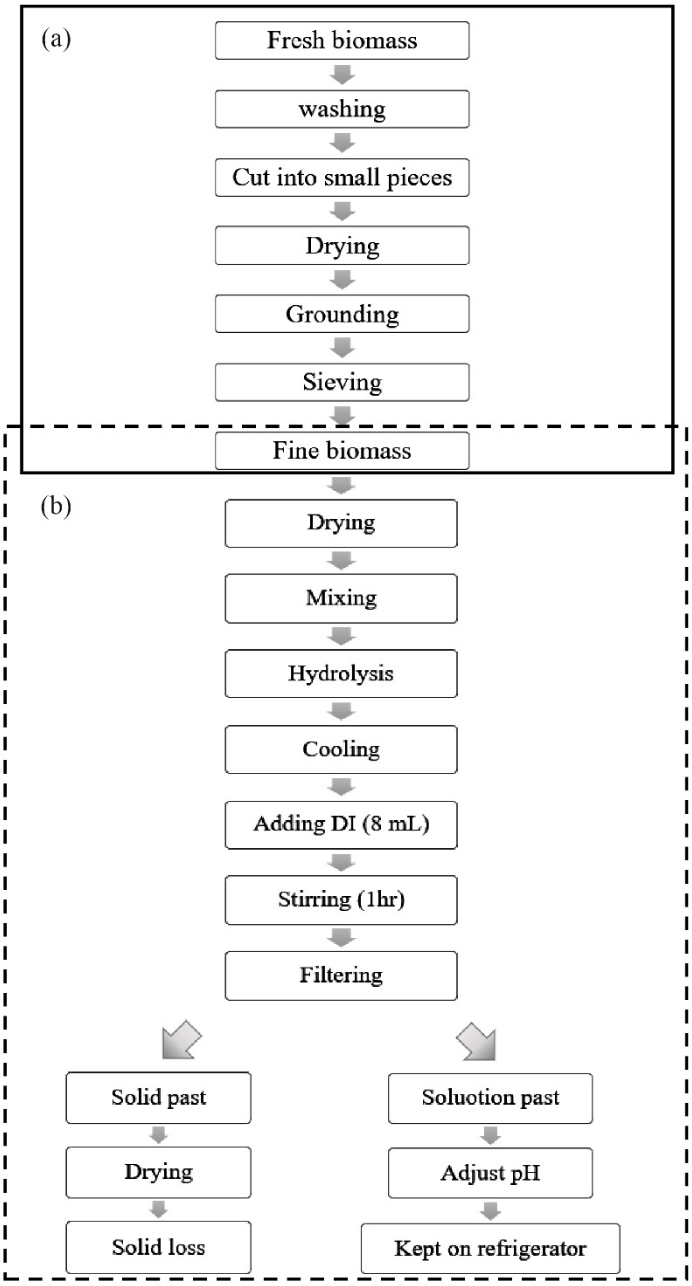

Figure 1 Flow diagrams of (a) the biomass preparation process and (b) the flow diagram of the physiochemical hydrolysis process.

Reducing sugar determination (RS)

Reducing sugar in the hydrolyzed products was measured by the dinitrosalicylic assay. The hydrolyzed product (0.2 mL) was mixed with 2 mL dinitrosalicylic acid solution in a closed test tube and immersed in boiling water for 10 min before being rapidly cooled to room temperature by ice water. The UV-VIS spectrophotometer (Thermo Fisher Scientific, G10S UV-VIS, USA) was used to record the UV absorbance of the mixture at 570 nm. A mixture of RO water and dinitrosalicylic acid solution (0.2:2, v/v) was used as blank. The result of reducing sugar was reported in % (g of reducing sugar in 100 g of dried weight of biomass).

3. Results and discussion

3.1 Analysis of proximate constituents of biomass

Biomass, including bagasse, palm leaf, rice straw, and tapioca starch, are rich in carbohydrates. The result of proximate constituents of biomass was analyzed by the Food Technology Laboratory, Department of Food Technology, Faculty of Chemical Engineering, University of Science and Technology, The University of Danang, Viet Nam.

Table 1 Percentage of proximate constituents of biomass

Proximate constituent (%)	Rice straw	Palm Leaf	Bagasse	Cassava Starch
Moisture	4.84 ± 0.21	3.27 ± 0.12	6.73 ± 0.19	12.74 ± 0.48
Total ash	10.84 ± 0.00	14.56 ± 0.00	1.84 ± 0.00	0.14 ± 0.00
Crude protein	5.85 ± 0.05	13.98 ± 0.22	0.00 ± 0.00	0.00 ± 0.00
Crude fat	2.11 ± 0.09	2.82 ± 0.03	0.49 ± 0.11	0.26 ± 0.00
Total carbohydrates	76.37 ± 0.35	65.37 ± 0.37	90.94 ± 0.30	86.86 ± 0.48

Table 1 shows the amounts of moisture, total ash, crude protein, crude fat, and total carbohydrate. The total carbohydrate is high in all of the biomass that we have chosen, especially in bagasse and tapioca starch. Rice straw and palm leaf contain high amounts of inorganic substances resulting in high total ash contents and, thus, lower total carbohydrate contents.

3.2 The extent of biomass hydrolysis observed by solid loss determination (SL)

The effect of catalyst concentration on biomass hydrolysis observed by solid loss

To observe the effect of catalyst concentration on each biomass hydrolysis, the concentrations of the HCl catalyst were varied from 0.2, 0.4, 0.6, 0.8, and 1.0 M. The reaction temperature was fixed at 100 °C and 15 minutes reaction time, and the ratio of catalyst per biomass is 10:1 (mL:g). The tapioca starch biomass was included for comparison. As expected, the starch structure of alpha-1,4 glucose polymer was more accessible for the hydrolysis reaction. The result showed that tapioca starch gives 100 percent of solid loss in every concentration, which is the highest percentage of solid loss. Among cellulosic biomass, the solid loss was the most in the case of bagasse, followed by palm leaf and rice straw, respectively (Figure 2). For bagasse, the solid loss slightly increased from 43.37% to 44.00%, 45.43% to 48.40% from the HCl concentrations of 0.2 M to 0.4 M, 0.6 M, and 0.8 M, then decreased to 47.53% at 1.0 M. In palm leaf, the solid loss decreased from 35.57 at 0.2 M to 34.93 % at 0.4 M and then increased to 36.40%, 38.70%, and 39.67% at 0.6 M, 0.8 M, and 1.0 M. Percent solid loss in rice straw slightly increased in the range of 0.2 – 1.0 M from 27.90%, 29.73%, 34.00%, 24.23% to 35.40%.

Bagasse had the highest percent solid loss implying that it has the highest ability to be broken down by the hydrolysis conditions, followed by palm leaf and rice straw. The concentration of the HCl catalyst had a small impact on the solid loss.

The effect of reaction temperature on biomass hydrolysis observed by solid loss

In this experiment, the reaction temperatures were varied at 100 °C, 150 °C, and 200 °C to investigate the effect of reaction temperature on the hydrolysis of biomass observed by solid loss. The concentration of HCl catalyst was fixed at 0.2 M with 15 minutes reaction time and 10:1 (mL:g) of catalyst per biomass.

The control - tapioca starch, gave the highest solid loss. The percent of the solid loss was 100% at the reaction temperatures of 100 °C and 150 °C. However, at 200 °C, the black residual solid was found on the filter paper resulting in a decrease of percent solid loss to 91.40%. The reaction temperature of 200 °C probably decomposed the soluble carbohydrate products. For cellulosic biomass (Figure 3), the solid loss increased from 43.37%, 57.53% to 79.79% for bagasse and 29.90%, 42.07 to 66.70% for rice straw. The reaction temperature had a significant effect on the solid loss. The higher reaction temperatures increased the solid loss. In palm leaves, the solid loss increased to a smaller extent, from 35.57% to 46.13 and 53.00%. The reaction temperature has a smaller effect on the solid loss of palm leaf than other biomass.

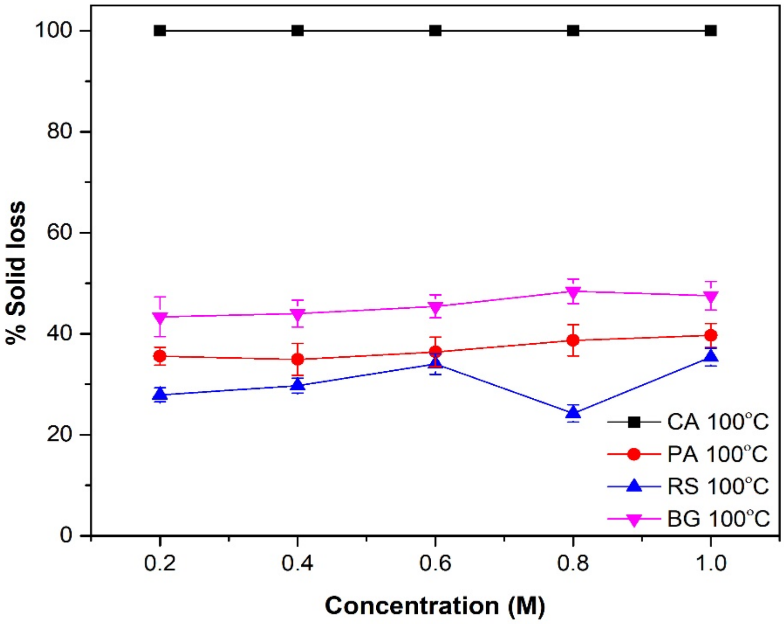

Figure 2 Percent solid loss of each biomass at different HCl catalyst concentrations varies from 0.2 – 1.0 M with 15 min reaction time, 100 °C, and a catalyst per biomass ratio of 10:1 (mL:g). (CA100C is tapioca starch, PA100C is palm leaf, RC100C is rice straw, BG100C is bagasse).

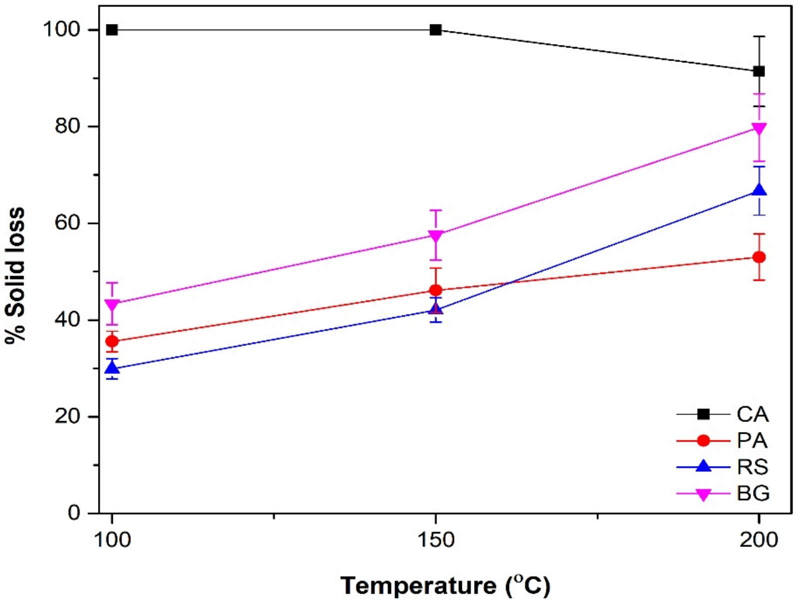

Figure 3 Percent solid loss of each biomass after the hydrolysis at different reaction temperatures (100 °C, 150 °C, and 200 °C), 0.2 M HCl catalyst, 15 minutes reaction time, and 10:1 (mL:g) of catalyst per biomass. (CA is tapioca starch, PA is palm leaf, RS is rice straw, BG is bagasse).

3.3 Reducing sugar determination (RS)

Reducing sugar determination is an inexpensive and convenient method to estimate the concentration of reducing sugar in the samples. All of the monosaccharides and some disaccharides are reducing sugars. The reducing sugar is a basic solution form of an aldehyde or ketone. The aldehyde group of glucose converts 3,5-dinitrosalicylic acid (DNS) to 3-amino-5nitrosalicylic acid, which is the reduced form of DNS. The conversion of absorbance into the concentration of monosaccharides was done by a standard curve of a monosaccharide such as glucose.

The effect of catalyst concentration on biomass hydrolysis observed by reducing sugar

The higher HCl catalyst concentrations until 0.8 M produced the higher reducing sugar concentrations (Figure 4). At 1.0 M concentration, lower reducing sugars were observed in the case of bagasse, rice straw, and palm leaf. At 1.0 M HCl concentration, some of the reducing sugar was possibly decomposed. As expected, tapioca starch gave the highest concentration of reducing sugar. Among cellulosic biomass, in a similar fashion to the solid loss, bagasse gave the highest amount of reducing sugar. The cellulosic structure of bagasse had been the most accessible for acid hydrolysis. On the other hand, palm leaf produced the lowest concentration of reducing sugar.

The acid-catalyzed hydrolysis in the case of HCl and H_2SO_4 are similar. First, the acidic protons dissociate from the acids. The glycosidic oxygens are protonated by the protons and make the corresponding more electrophilic. After that, the oxygen atom of a water molecule attacks the glycosidic carbon resulting in the broken glycosidic bond. Thus, the carbohydrate molecules are fragmented into two smaller molecules, as shown in Figure 5.

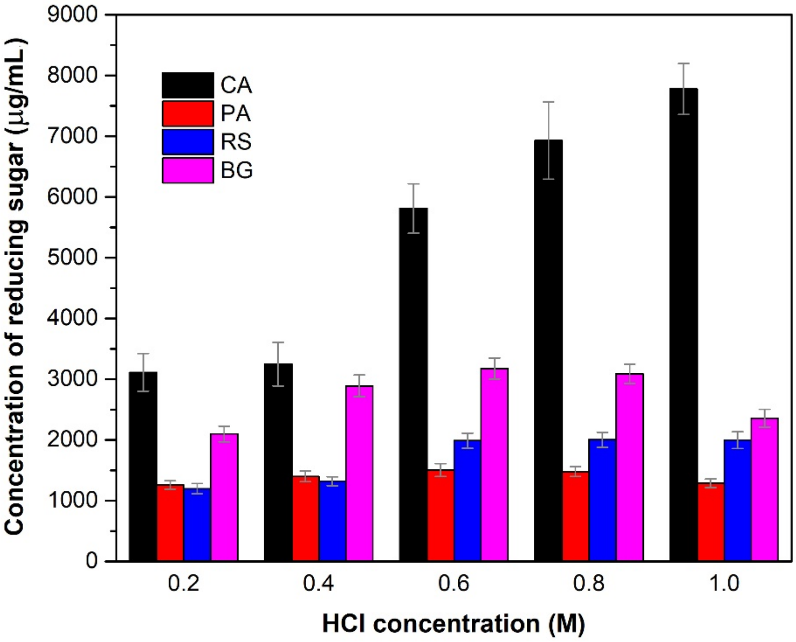

Figure 4 Concentrations of reducing sugar from each biomass at various HCl concentrations, with 15 min reaction time, 100 °C, and a catalyst per biomass ratio of 10:1 (mL:g) (CA is tapioca starch, PA is palm leaf, RS is rice straw, BG is bagasse).

The effect of reaction temperature on biomass hydrolysis observed by reducing sugar

To compare the effect of the reaction temperature on biomass hydrolysis, the reaction temperatures were varied at 100, 150, and 200 °C. The HCl catalyst concentration, reaction time, and ratio of catalyst per biomass were fixed at 0.2 M, 15 minutes, and 10:1 (mL:g), respectively. The reaction temperature significantly affects the reducing sugar

(Figure 6). The medium reaction temperature at 150 °C gave the highest reducing sugar concentrations for bagasse and rice straw. Palm leaf required a lower reaction temperature at 100 °C to produce the highest reducing sugar.

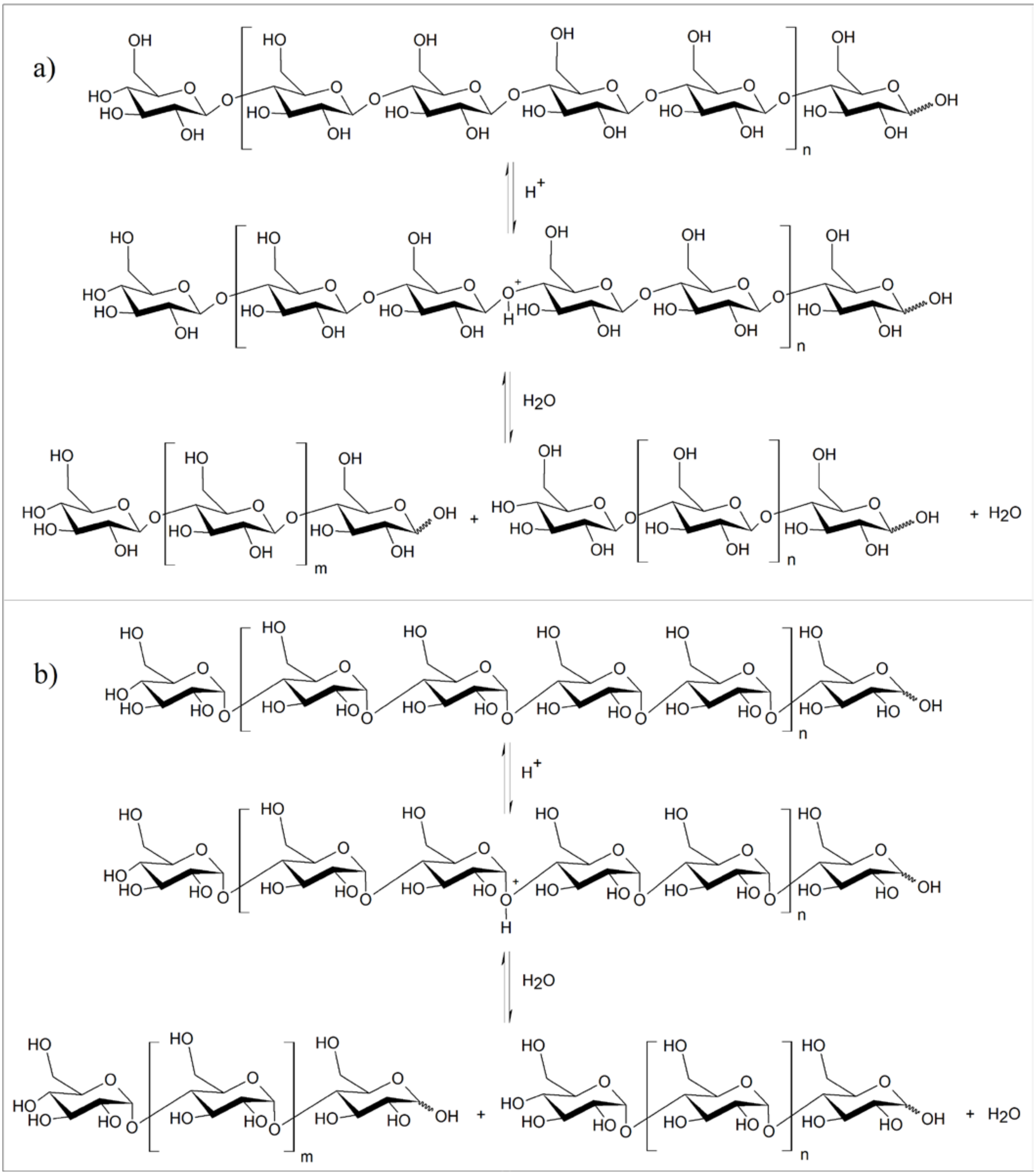

Figure 5 Mechanism of acid hydrolysis of (a) cellulose and (b) starch.

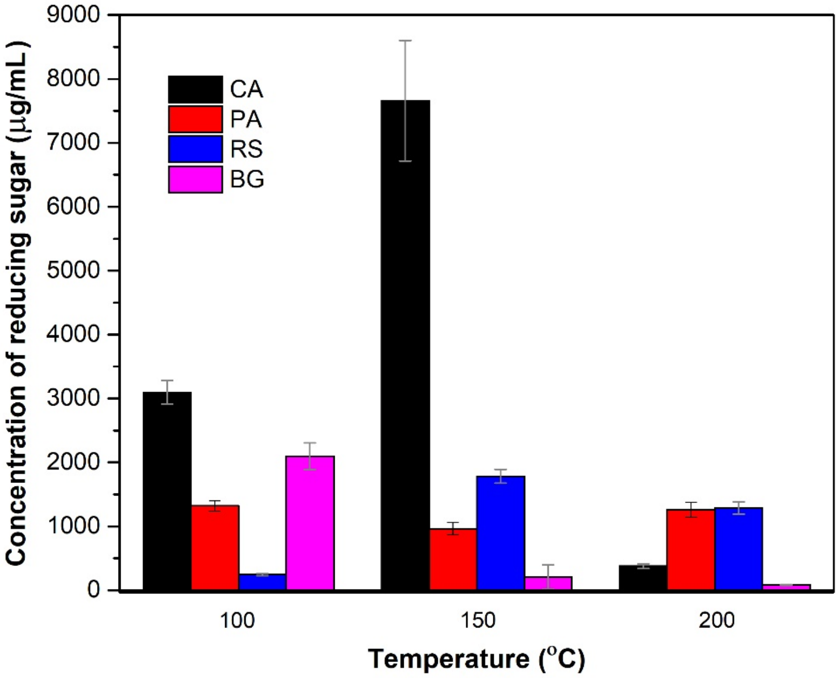

Figure 6 Concentrations of reducing sugar from each biomass at various reaction temperatures with 0.2 M HCl catalyst, 15 minutes reaction time, and 10:1 (mL:g) of catalyst per biomass. (CA is tapioca starch, PA is palm leaf, RS is rice straw, BG is bagasse).

4. Conclusion

In this study, we found that it is feasible to use the combination of microwave irradiation heating and HCl as an acid catalyst to hydrolyze the abundant biomass found in Thailand into soluble carbohydrates. The solid loss and reducing sugar showed that bagasse gave the highest yields of soluble carbohydrates. The reaction temperature and HCl concentration have significant effects on the hydrolysis. The higher reaction temperature and HCl concentration led to a higher solid loss. However, the resulting soluble carbohydrates tend to be partially decomposed. Thus, the medium HCl concentrations at 0.6 M or 0.8 M and the lower reaction temperature at 100°C gave the highest reducing sugar.

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