CHAPTER IV
STUDY OF MONOMERIC SUGAR PRODUCTION FROM SUGARCANE BAGASSE USING MICROWAVE/CHEMICAL PRETREATMENT PROCESS

4.1 Abstract

Sugarcane bagasse are mainly composed of cellulose and hemicellulose which can be converted to monomeric sugars (glucose and xylose), and then further fermented to biofuel. Interestingly, microwave heating techniques are now widely used in many applications of chemical research due to their fast heating, homogeneous temperature, reduced reaction time, and increased reaction rate. The objective of the work is to investigate the suitable pretreatment conditions of converting sugarcane bagasse to the highest amount of monomeric sugars via microwave heating combined with sulfuric acid or sodium hydroxide. The effects of time, temperature, concentration of sulfuric acid and sodium hydroxide, and liquid-to-solid ratio were also determined. The suitable conditions of the NaOH pretreatment were 0.5 % (w/v) NaOH with a 15:1 liquid- to- solid ratio at 40 °C for 5 min. In the case of the H_2SO_4 pretreatment, the optimal conditions were 2.0 % (v/v) H_2SO_4 with a 15:1 liquid-to-solid ratio at 120 °C for 15 min. Under optimal conditions, the sulfuric pretreatment released the highest total monomeric sugar yield of 51.50 g/100 g biomass (21.89 g of glucose and 29.61 g of xylose), which was much higher than those pretreated using NaOH.

Keyword: Sugarcane bagasse/ Microwave technique/ Acid hydrolysis/ Alkali hydrolysis/ Pretreatment

4.2 Introduction

Suagarcane bagasse is a renewable, cheap and widely abundant in Thailand. It has a complex structure, and is mainly consisted of 40% cellulose, 24% hemicellulose and 25% lignin [1]. Pretreatment is required to convert sugarcane bagasse into fermentable sugars for further alcohol production.

Acid and alkali can be used as catalysts for treating lignocellulosic residues [2-6]. Acid can break down heterocyclic ether bonds between sugar monomers in the polymeric chains, which are formed by hemicellulose and cellulose [4]. H_2SO_4 and HCl are potential acid used to hydrolyze sugarcane bagasse. The amount of the sugars released depends on acid concentration, reaction time and temperature [7-8]. However, alkali pretreatment of sugarcane bagasse digests of the lignin matrix and makes cellulose and hemicellulose available to enzyme degradation [9].

Microwave is a new effective technique for pretreatment of cellulosic materials [10]. It results in a much higher switchgrass digestibility than conventional heating [11]. Besides, the microwave- assisted alkali pretreatment removes more lignin and hemicellulose from wheat straw with shorter pretreatment time compared with the conventional alkali one [12]. The microwave technique also offers the advantage of performing acidic hydrolysis reaction [13].

The objectives of this work are to evaluate practical benefits of microwave/chemical pretreatment, to investigate the optimal conditions of monomeric sugar production from sugarcane bagasse pretreated with dilute-alkaline and dilute-acid using microwave technique, and to compare the amounts of monomeric sugars obtained from each condition.

4.3 Experimental

4.3.1 Materials

Sugarcane bagasse was obtained from Ratchaburi, Thailand. Before any pretreatment, sugarcane bagasse was washed with tap water and dried under sunlight. It was then milled to obtain small particles using herb grinder before further pretreatment.

Sodium hydroxide (NaOH), hydrochloric acid (HCl) and nitric acid (HNO_3) were purchased from Labscan Asia Co., sulfuric acid (H_2SO_4) and phosphoric acid (H_3PO_4) were supplied by Merck Co. (Germany), D-glucose anhydrous was obtained from Ajax Finechem (Aus.) and DL-xylose (minimum, 99 %) were purchased from Sigma Aldrich Chemicals Co. Inc. (USA). These chemicals were directly used without purification.

4.3.2 Equipments

4.3.2.1 Microwave Solvent Extraction Lab Station

Pretreatment of sugarcane bagasse was conducted in Microwave Solvent Extraction Lab Station (Ethos Sel, Milestone). Samples were heated in a Teflon-vessel sealed with a Teflon using with time-to-temperature program.

4.3.2.2 High Performance Liquid Chromatography (HPLC)

Monomeric sugars (glucose and xylose) were determined by HPLC with a refractive index detector (Series 200 LC/S/N291N5060508, Perkin Elmer) using an Aminex-HPX 87H column (300 mm x 7.8 mm, Bio-Rad Lab, USA) and a guard column (30 mm x 4.6 mm, Bio-Rad Lab, USA) under the following conditions: mobile phase 0.005 M of H_2SO_4 and a flow rate of 0.60 mL/min. Sample injection was 20 μL.

4.3.3 Methodology

4.3.3.1 Pretreatment of sugarcane bagasse by microwave heating

The microwave/chemical pretreatment process of sugarcane bagasse in this project was investigated from NaOH/H_2SO_4 hydrolysis, using water as a controlled experiment. Prior to microwave pretreatment, sugarcane bagasse was suspended in NaOH (0.5-5 % (w/v)) or H_2SO_4 solution (0.5-3 % (v/v)) with 15:1-

40:1 liquid-to-solid ratio (LSR) (mL of solution/g of bagasse by weight). The mixture was stirred until homogeneous, and transferred to Teflon-vessel sealed with a Teflon cap. The microwave pretreatment was conducted under reaction temperature and time in the range of 40 to 120 °C and 5-120 min, respectively (Adapted from [14]).

4.3.3.2 Pretreatment of sugarcane bagasse by conventional heating

Sugarcane bagasse were suspended in 0.5-2.0 % (v/v) H_2SO_4 solution with a 30:1 liquid-to-solid ratio (LSR) (mL of solution/g of bagasse by weight) in a 1000 mL flask and kept boiling at 120 °C for 90 min using oil bath (Adapted from Hu *et al.* (2008)). It is estimated that the solution mixture took about 5 min to be heated up from room temperature to the required temperature (120 °C), therefore the recording of time was started after the flask was placed in the oil bath for 5 min. During the heating period, the solution mixture was stirred to homogenize the solid and liquid fractions.

4.3.3.3 Monomeric sugar analysis

After pretreatment, the mixture was filtered to separate the solid residues and the filtrate fraction. The liquid fraction was collected for monomeric sugar analysis using HPLC.

4.4 Results and Discussion

4.4.1 Water Pretreatment

Table 4.1 and Figure 4.1 demonstrated that the differences in time at 40 °C, 60 °C, and 80 °C slightly affect to the release of monomeric sugar containing in the sugarcane bagasse. The total yields of monomeric sugars obtained under different conditions were around 3.04–4.27 g/100 g biomass. It can be seen that the temperature had no significant effect on the release of monomeric sugars. Thus, it is not necessary to perform the reaction at higher temperature. Although microwave treatment can be utilized to pre-treat sugarcane bagasse, the amounts of monomeric sugars released from the water hydrolysis reaction were still low. It was noticed that water/microwave pretreatment could not completely break down the resistant structure of sugarcane bagasse [11, 13].

Table 4.1 Total yield of monomeric sugars of water-pretreated sugarcane bagasse under different times and temperatures

Reaction time (min)	Total yield of monomeric sugars (g/100 g biomass)		
	40 °C	60 °C	80 °C
30	3.58 ± 0.58	3.88 ± 0.88	3.04 ± 0.05
60	4.20 ± 0.24	4.07 ± 0.72	3.60 ± 0.59
90	4.27 ± 0.08	3.62 ± 0.10	3.38 ± 0.69

Data are mean values ± S.D. of two replicates.

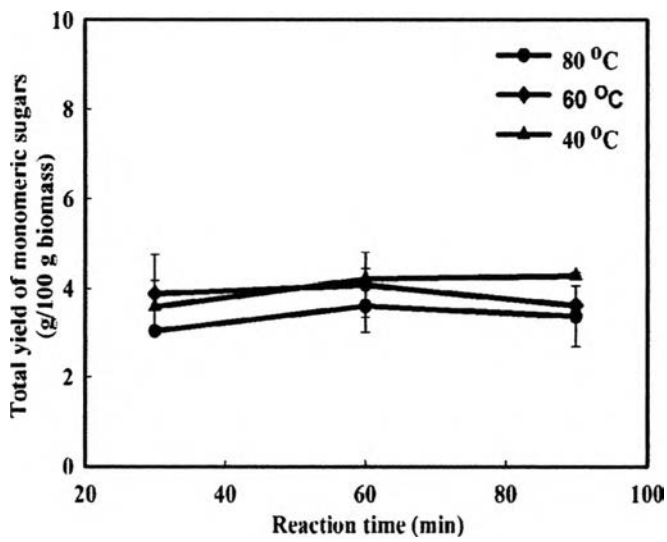

Figure 4.1 Effects of time and temperature on the release of the monomeric sugars from water-pretreated sugarcane bagasse.

Based on the fact that alkali is capable of removing the lignin barrier in the biomass and acid can hydrolyze the heterocyclic ether bond between sugar monomers to enhance higher sugar yields [3, 4], acid or alkali combined with

microwave pretreatment was studied using sugarcane bagasse by varying heating time, heating temperature, acid or alkali concentration, and liquid-to-solid ratio.

4.4.2 Dilute Alkaline Pretreatment

4.4.2.1 Effects of Reaction Time and Temperature

The effects of reaction time and temperature were investigated by pretreating sugarcane bagasse with 0.5 % (w/v) NaOH using a 30:1 liquid-to-solid ratio. Table 4.2 and Figure 4.2 (a) showed that 40 °C temperature resulted in the highest glucose content with less amount of xylose due to the fact that xylose would be released from hemicellulose easily hydrolyzed using the acid pretreatment. The NaOH treatment is to delignify by breaking the ester bonds of lignin [15]. The obtained lignin is solubilized in hemicellulose still recovered as oligomers [4]. As a result, glucose was easier released from sugarcane bagasse by the NaOH pretreatment.

At 60 °C and 80 °C, the NaOH pretreatment resulted in very low total monomeric sugar yields (about 0.82–1.69 g/100 g biomass), and the reaction time did not affect to the monomeric sugar production remaining its yield at almost constant levels, as shown in Table 4.2 and Figure 4.2 (b-d). Generally, alkali pretreatment process utilizes lower temperatures and pressures, as compared to other pretreatment processes [3], and therefore, that is the reason why the high heating efficiency and homogeneous temperature of microwave technique combined with sodium hydroxide might significantly affect to the process by degrading or retarding the sugar production [11].

In the case of the reaction time, even at 40 °C, the highest total monomeric sugar was obtained at 5 min, and then the total monomeric sugar decreased with increasing reaction time (Table 4.2 and Figure 4.2 (a and d)). These results could be explained that the parallel reactions, which were decomposition of sugar to inhibitor, reversion and enolization of monomeric sugar, took place [16]. Reversion is the phenomenon of monomeric sugar polymerization to di- and oligosaccharides [16], and dissolution of monosaccharides in aqueous base causes them to undergo enolization reaction, leading to unstable intermediate formation [17]. The reaction time at 75 min gave a higher yield of monomeric sugars again probably due to the reversed reactions of reversion and enolization reactions to

monomeric sugars [16-18]. Therefore, the optimal temperature and time selected for the NaOH pretreatment were at 40 °C for 5 min to give the maximum glucose of 3.71 g and xylose yields of 1.46 g (per 100 g biomass)

Table 4.2 Momomeric sugar yields of sugarcane bagasse hydrolyzed with 0.5 % (w/v) NaOH using 30:1 LSR under different times and temperatures (g sugar per 100 g biomass)

Temperature (°C)	Reaction time (min)	Glucose	Xylose	Total monomeric sugars
40	5	3.71 ± 0.35	1.46 ± 0.23	5.18 ± 0.59
	15	3.66 ± 0.21	1.65 ± 0.03	5.31 ± 0.25
	30	2.05 ± 0.10	1.09 ± 0.04	3.14 ± 0.14
	45	2.08 ± 0.25	1.21 ± 0.05	3.29 ± 0.31
	60	1.75 ± 0.16	0.85 ± 0.0002	2.59 ± 0.16
	75	2.22 ± 0.20	2.19 ± 0.36	4.41 ± 0.57
	90	2.06 ± 0.04	1.01 ± 0.02	3.06 ± 0.05
60	30	1.69 ± 0.51	nd	1.69 ± 0.51
	45	1.58 ± 0.41	nd	1.58 ± 0.41
	60	1.54 ± 0.02	nd	1.54 ± 0.02
	75	0.98 ± 0.21	nd	0.98 ± 0.21
	90	0.82 ± 0.02	nd	0.82 ± 0.02
80	30	1.25 ± 0.25	nd	1.25 ± 0.25
	45	0.99 ± 0.22	nd	0.99 ± 0.22
	60	0.91 ± 0.05	nd	0.91 ± 0.05
	75	1.10 ± 0.06	nd	1.10 ± 0.06
	90	1.09 ± 0.11	nd	1.09 ± 0.11

Data are mean values ± S.D. of two replicates.
nd = not detected

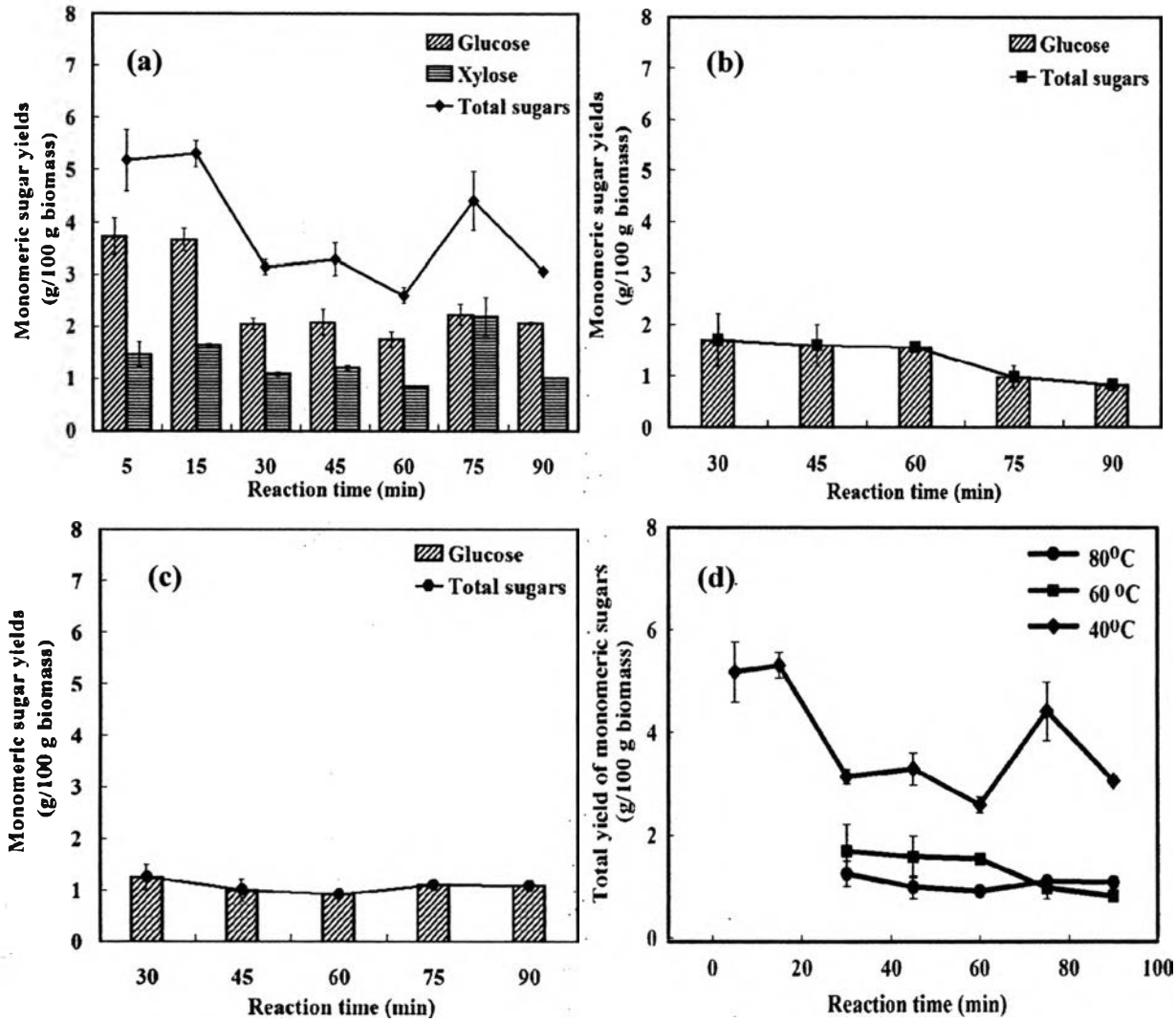

Figure 4.2 The main components of sugarcane bagasse hydrolysate using 0.5 % (w/v) NaOH, 30:1 LSR, and different times and temperatures: (a) 40 °C, (b) 60 °C, (c) 80 °C, and (d) comparison of the total yield of monomeric sugars at different temperatures.

4.4.2.2 Effect of Alkaline Concentration

In order to study the effect of alkali concentration on the release of monomeric sugars, sugarcane bagasse was pretreated using 0.5 to 5.0 % (w/v) NaOH with a 30:1 liquid-to-solid ratio at 40 °C for 15 min. As shown in Table 4.3 and Figure 4.3, an increase in NaOH concentration caused a lower total monomeric sugar yield. This was probably due to the partial degradation of the

hemicellulose at high alkali concentration, implying too severe condition [11, 19]. The highest total monomeric sugar yield, 5.31 g/100 g biomass, was achieved using 0.5 % (w/v) NaOH. Under these conditions, 3.66 g glucose and 1.65 g xylose per 100 g biomass were obtained. In addition, the highest amount of glucose, 4.50 g/100 g biomass, was released using 1.0 % (w/v).

Table 4.3 Momomeric sugar yields of NaOH-pretreated sugarcane bagasse using 30:1 LSR at 40 °C for 15 min with different NaOH concentrations (g/100 g biomass)

Alkali concentration (%w/v)	Glucose	Xylose	Total monomeric sugars
0.5	3.66 ± 0.21	1.65 ± 0.03	5.31 ± 0.25
1.0	4.50 ± 0.17	nd	4.50 ± 0.17
3.0	2.58 ± 0.62	nd	2.58 ± 0.62
5.0	2.08 ± 0.08	nd	2.08 ± 0.08

Data are mean values ± S.D. of two replicates.
nd = not detected

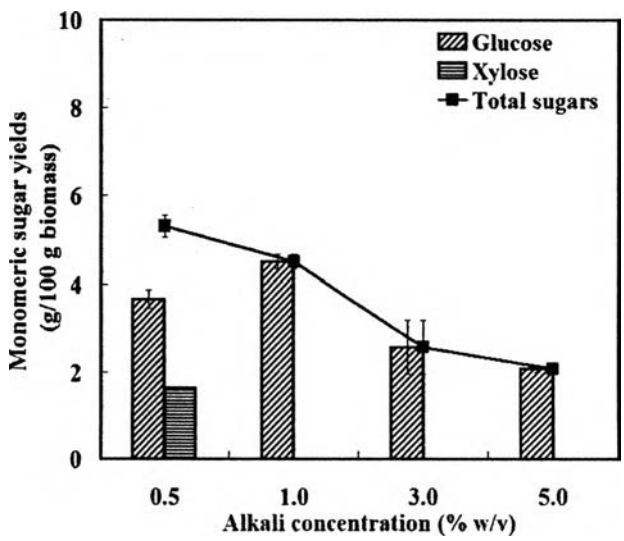

Figure 4.3 Effect of NaOH concentration on the release of monomeric sugars of NaOH- pretreated sugarcane bagasse using 30:1 LSR at 40 °C for 15 min.

4.4.2.3 Effect of Liquid-to-Solid Ratio (LSR)

The effect of LSR on the monomeric sugar production of sugarcane bagasse was evaluated by treating sugarcane bagasse with 15:1 to 40:1 of LSR at 0.5 % (w/v) NaOH at 40 °C for 15 min. Table 4.4 and Figure 4.4 demonstrated that the release in the monomeric sugars decreased with increasing the LSR. It can be explained that the higher the LSR, the more dilute the mixture [20]. The highest amount of the total monomeric sugar yield of 8.74 g/100 g biomass, containing 5.05 g glucose and 3.69 g xylose (per 100 g biomass), was obtained at 15:1 LSR. The LSR of 20:1 gave the maximum glucose yield, 5.42 g/100 g biomass, but it was not significantly different from the glucose yield obtained at 15:1 LSR. Therefore, the LSR of 15:1 was selected for further study. However, when LSR was lower than 15:1, it became hard to keep the reaction system homogeneous because of less liquid being present [15, 21].

Table 4.4 Monomeric sugar yields of sugarcane bagasse using 0.5 % (w/v) NaOH at 40 °C for 15 min with different LSRs (g/100 g biomass)

Liquid to solid ratio (LSR)	Glucose	Xylose	Total monomeric sugars
15: 1	5.05 ± 0.41	3.69 ± 0.40	8.74 ± 0.81
20: 1	5.42 ± 0.70	nd	5.42 ± 0.70
30: 1	3.66 ± 0.21	1.65 ± 0.03	5.31 ± 0.25
40: 1	4.59 ± 0.48	nd	4.59 ± 0.48

Data are mean values ± S.D. of two replicates.
nd = not detected

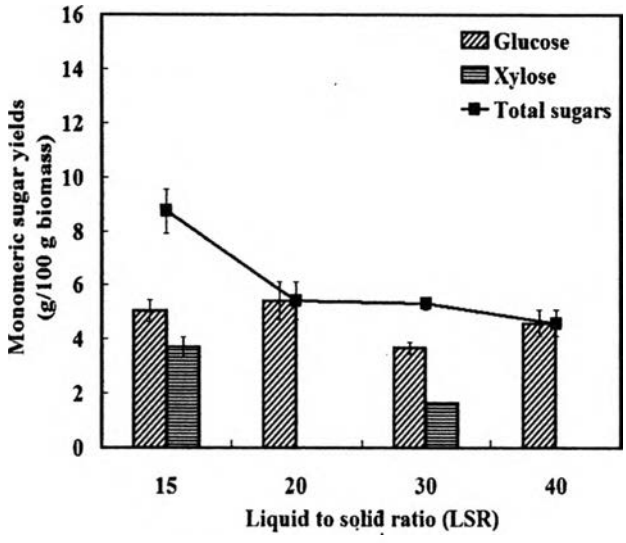

Figure 4.4 Effect of LSR on the release of the monomeric sugars of NaOH-pretreated sugarcane bagasse (other conditions: 0.5 % (w/v) NaOH at 40 °C for 15 min).

4.4.3 Dilute Acid Pretreatment

4.4.3.1 Effects of Time and Temperature

The main components of the sugarcane bagasse hydrolyzed by 0.5 % (v/v) H_2SO_4 using 30:1 LSR under different times and temperatures are shown in Table 4.5 and Figure 4.5. The results showed that xylose was the main product. The high content of xylan makes this sugarcane bagasse adequate for xylose production [2], and the main reaction that occurs during acid pretreatment is the hydrolysis of hemicellulose, especially xylan [5]. For the reaction time within 90 and 60 min at 60 °C and 80–100 °C, respectively, it was observed that the longer reaction time gave the higher monomeric sugar yield (Figure 4.5 a-c). Interestingly, at 120 °C the maximum total monomeric sugar yield, 26.58 g/100 g biomass, was obtained within 15 min and decreased thereafter. Under these conditions, the highest xylose yield, 16.80 g/100 g biomass, were detected, but the highest glucose yield, 11.05 g/100 g biomass, were achieved at 120 °C for 30 min (Table 4.5 and Figure 4.5 d-e). It is due to the fact that glucose was the main component released from cellulose, which has more ordered structure than hemicelllose [22], thus, requires longer pretreatment time for glucose production. A decrease in the monomeric sugar yield at too long reaction time was due to the occurrence of decomposition reaction of sugar to inhibitor, such as furfural [7, 11, 23]. Moreover, the reaction time together with

the temperature caused the severity, and consequently, the effectiveness of biomass pretreatment [11].

Table 4.5 Monomeric sugar yields of sugarcane bagasse hydrolyzed, using 0.5 % (v/v) H_2SO_4, 30:1 LSR under different times and temperatures (g sugar per 100 g biomass)

Temperature (°C)	Reaction time (min)	Glucose	Xylose	Total monomeric sugars
60	30	2.28 ± 0.56	2.42 ± 0.78	4.70 ± 1.34
	60	3.35 ± 1.05	3.53 ± 0.12	6.89 ± 1.17
	90	6.61 ± 1.08	7.23 ± 1.00	13.84 ± 2.08
	120	4.61 ± 0.16	5.15 ± 0.26	9.76 ± 0.42
80	30	5.43 ± 0.65	5.60 ± 0.68	11.03 ± 1.33
	60	7.07 ± 0.20	8.68 ± 0.46	15.75 ± 0.67
	90	6.35 ± 0.19	7.36 ± 0.88	13.71 ± 1.07
	120	6.87 ± 1.03	8.27 ± 0.70	15.14 ± 1.74
100	30	2.90 ± 0.43	4.67 ± 0.55	7.57 ± 0.98
	60	8.85 ± 0.35	16.38 ± 1.33	25.23 ± 1.68
	90	7.04 ± 0.55	13.43 ± 0.80	20.46 ± 1.35
	120	7.24 ± 1.25	12.90 ± 0.43	20.14 ± 1.68
120	5	7.87 ± 0.23	11.97 ± 0.52	19.84 ± 0.75
	10	8.44 ± 0.19	13.79 ± 0.58	22.23 ± 0.77
	15	9.78 ± 0.46	16.80 ± 1.17	26.58 ± 1.63
	30	11.05 ± 0.13	14.93 ± 1.77	25.97 ± 1.89
	60	9.68 ± 1.54	10.92 ± 0.15	20.60 ± 1.69
	90	7.30 ± 0.87	8.50 ± 0.85	15.80 ± 1.73
	120	10.48 ± 0.59	11.37 ± 0.73	21.85 ±1.33

Data are mean values ± S.D. of two replicates.

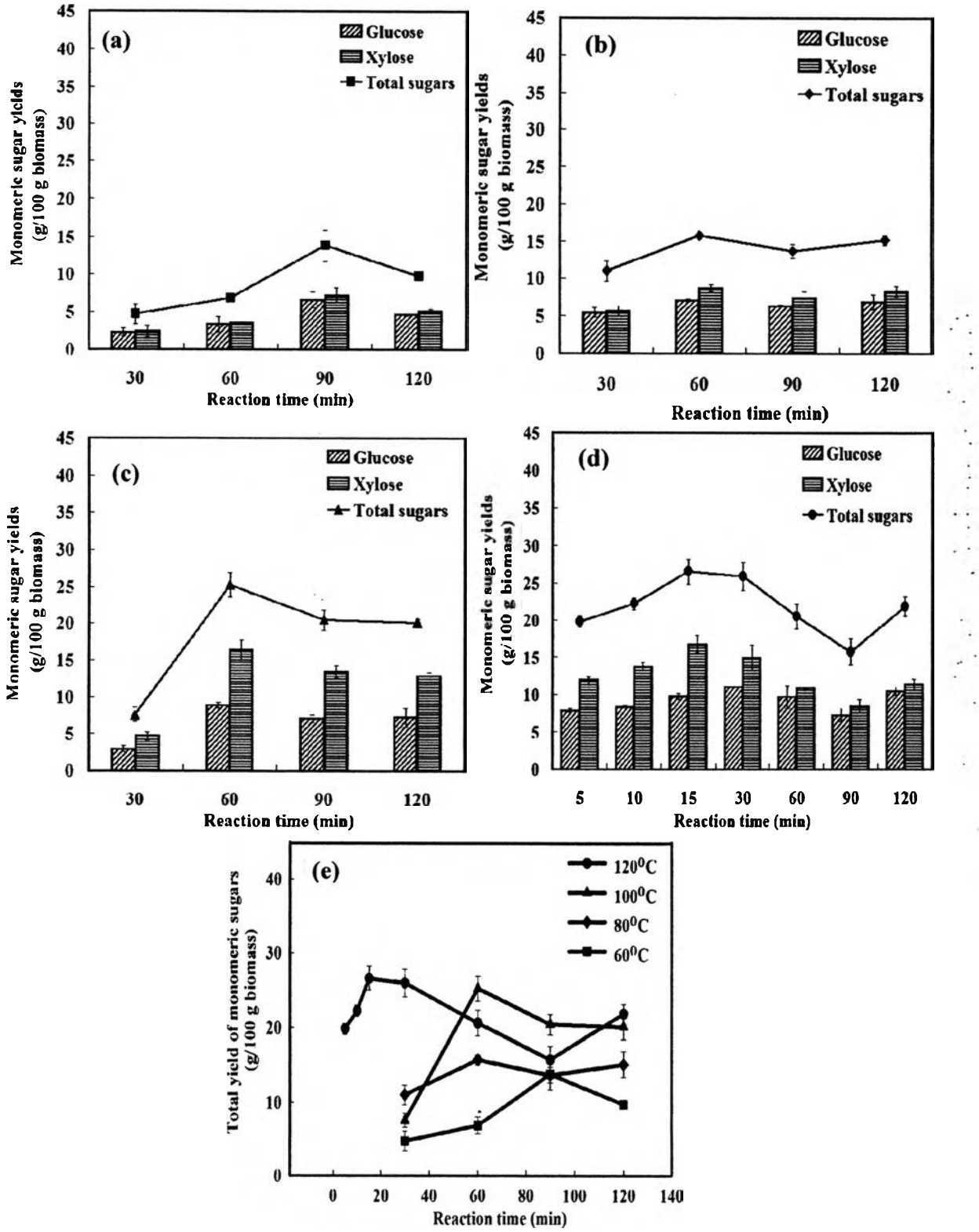

Figure 4.5 The main components of sugarcane bagasse hydrolysate under 0.5 % (v/v) H_2SO_4, 30:1 LSR, and different times and temperatures: (a) 60 °C, (b) 80 °C,

(c) 100 °C, (d) 120 °C, and (e) comparison of the total yield of monomeric sugars at different temperatures.

4.4.3.2 Effect of Acid Concentration

The sugarcane bagasse hydrolyzed with different concentrations of dilute H_2SO_4 using 30:1 LSR at 120 °C for 15 min (Table 4.6 and Figure 4.6) showed an increase in releasing monomeric sugars when increasing acid concentration from 0.5 to 2.0 % (v/v), and a decrease beyond 2.0 % (v/v). Due to the synergistic interaction effect of acid concentration and temperature [8], the highest total monomeric sugar yield of 36.62 g/100 g biomass was obtained when the sugarcane bagasse was treated, using 2.0 % (v/v) H_2SO_4 at 120 °C for 15 min. Under these conditions, the maximum glucose and xylose yields, which were 15.33 and 21.29 (in g/100 g biomass), respectively, were detected. This phenomenon can be explained that any further increase in acid concentration caused the increase in the release of some toxic compounds or inhibitors, resulting in a decrease of monomeric sugar yield [7, 24]. Therefore, 2.0 % (v/v) was selected for further study.

Table 4.6 Monomeric sugar yields of sugarcane bagasse pretreated with different H_2SO_4 concentrations using 30:1 LSR at 120 °C for 15 min (g sugar/ 100 g biomass)

Acid concentration (% v/v)	Glucose	Xylose	Total monomeric sugars
0.5	9.78 ± 0.46	16.80 ± 1.17	26.58 ± 1.63
1.0	15.12 ± 1.56	19.20 ± 0.18	34.32 ± 1.74
1.5	14.21 ± 0.73	18.75 ± 1.29	32.97 ± 2.02
2.0	15.33 ± 1.02	21.29 ± 0.70	36.62 ± 1.72
2.5	14.60 ± 1.22	17.19 ± 0.96	31.79 ± 2.18
3.0	15.10 ± 0.76	18.90 ± 0.97	34.00 ± 1.73

Data are mean values ± S.D. of two replicates.

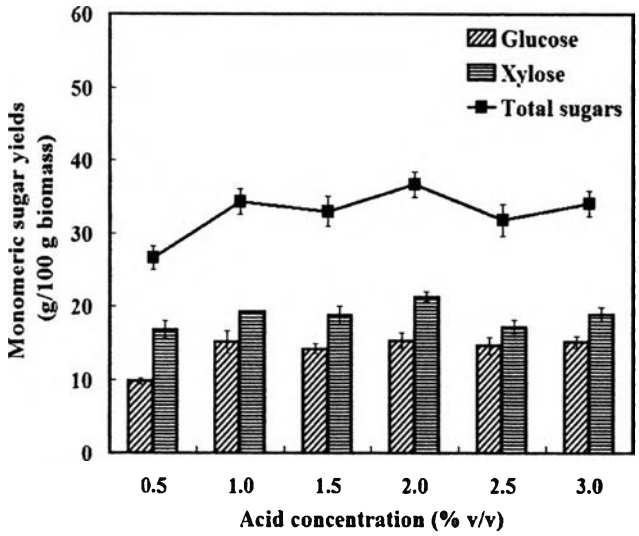

Figure 4.6 Effect of H_2SO_4 concentration on the release of monomeric sugars of sugarcane bagasse hydrolysates (Other conditions: 30: 1 LSR at 120 °C for 15 min).

4.4.3.3 Effect of Liquid-to-Solid Ratio (LSR)

To investigate the effect of LSR on the release of monomeric sugars, sugarcane bagasse was pretreated in various LSR with 2.0 % (v/v) H_2SO_4 at 120 °C for 15 min. Table 4.7 and Figure 4.7 revealed that an increase in LSR resulted in a decrease in the release of the monomeric sugars. Again, similar to the NaOH pretreatment, the increase in LSR makes the mixture more dilute, thus less efficiency in producing the monomeric sugars [20]. The highest amount of the total monomeric sugar yield, 51.50 g/100 g biomass, containing the maximum glucose yield of 21.89 and the maximum xylose yield of 29.61 (in g/100 g biomass), was obtained at 15:1 LSR. Again, the pretreatment was not performed with the LSR lower than 15:1 because a smaller LSR made the system less homogeneous [15, 21].

Table 4.7 Monomeric sugar yields of sugarcane bagasse using 2.0 % (v/v) H_2SO_4 at 120 °C for 15 min with different LSRs (g/100 g biomass)

Liquid to solid ratio (LSR)	Glucose	Xylose	Total monomeric sugars
15:1	21.89 ± 3.67	29.61 ± 4.34	51.50 ± 8.00
20:1	20.63 ± 0.95	27.86 ± 1.11	48.49 ± 2.06
30:1	15.33 ± 1.02	21.29 ± 0.70	36.62 ± 1.72
40:1	10.83 ± 1.09	16.00 ± 0.90	26.83 ± 1.99

Data are mean values ± S.D. of two replicates.

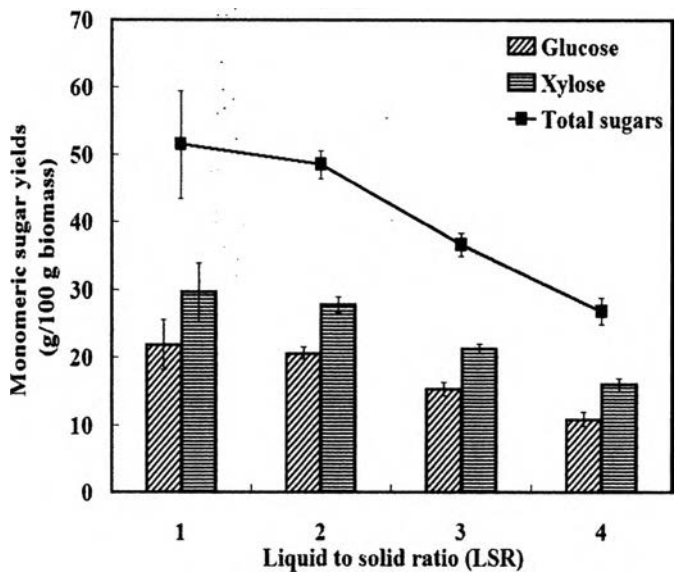

Figure 4.7 Effect of LSR on the release of monomeric sugars of H_2SO_4-pretreated sugarcane bagasse (Other conditions: 2.0 % (v/v) H_2SO_4 at 120 °C for 15 min).

As a result, the optimal conditions of H_2SO_4 pretreatment used to treat sugarcane bagasse were 2.0 % (v/v) H_2SO_4 with 15:1 LSR at 120 °C for 15 min, giving the highest glucose and xylose yields, which were 21.89 and 29.61 (in g/100 g biomass), respectively. Comparing between the optimal conditions of NaOH and H_2SO_4 pretreatments, it was found that the total monomeric sugar yield released from the H_2SO_4 pretreatment was much higher than that obtained from the NaOH pretreatment. Therefore, it can be concluded that the H_2SO_4 pretreatment combined with microwave heating is more suitable to pre-treat sugarcane bagasse.

4.4.4 Comparison of dilute-acids

Several dilute acids were used as catalysts for the hydrolysis of sugarcane bagasse. The efficiency of H_2SO_4 determined in this work was compared with HCl, HNO_3, and H_3PO_4 under the same operating conditions (2.0 % (v/v) acid with 15:1 LSR at 120 °C for 15 min)

Table 4.8 and Figure 4.8 show that the amount of the total monomeric sugars produced from the H_2SO_4 pretreatment was still higher than those from other acids. The total monomeric sugar yields were decreased from 51.50 to 45.71, 39.07, and 20.28 g/100 g biomass when sugarcane bagasse was pretreated with H_2SO_4, HCl, HNO_3, and H_3PO_4, respectively. The level and composition of the monomeric sugars released depends on the type of acid and its concentration [9]. H_2SO_4 and HCl were potential acids used for pre-treating sugarcane bagasse, and it was also found that the release of the monomeric sugars of the HCl-pretreated sugarcane bagasse was nearly to that of the H_2SO_4-pretreated sugarcane bagasse [7]. In case of HNO_3 and H_3PO_4 pretreatments, they could not release the monomeric sugars as well as H_2SO_4 and HCl. It was also reported that HNO_3 needed lower time under optimal conditions than H_2SO_4 or HCl [23], thus, the severity of conditions may resulted in lowering the monomeric sugar yield of nitric acid pretreatment [11]. For the H_3PO_4 pretreatment, it could also be owing to the weak character of the catalyst [8].

Table 4.8 Monomeric sugar yields of sugarcane bagasse pretreated with 2.0 % (v/v) of different dilute acids using 15:1 LSR at 120 °C for 15 min (g/100 g biomass)

Types of acid	Glucose	Xylose	Total monomeric sugar
H_2SO_4	21.89 ± 3.67	29.61 ± 4.34	51.50 ± 8.00
HCl	18.51 ± 1.21	27.20 ± 0.03	45.71 ± 1.24
HNO_3	18.30 ± 0.01	20.77 ± 0.08	39.07 ± 0.09
H_3PO_4	nd	20.28 ± 0.73	20.28 ± 0.73

Data are mean values ± S.D. of two replicates.
nd = not detected

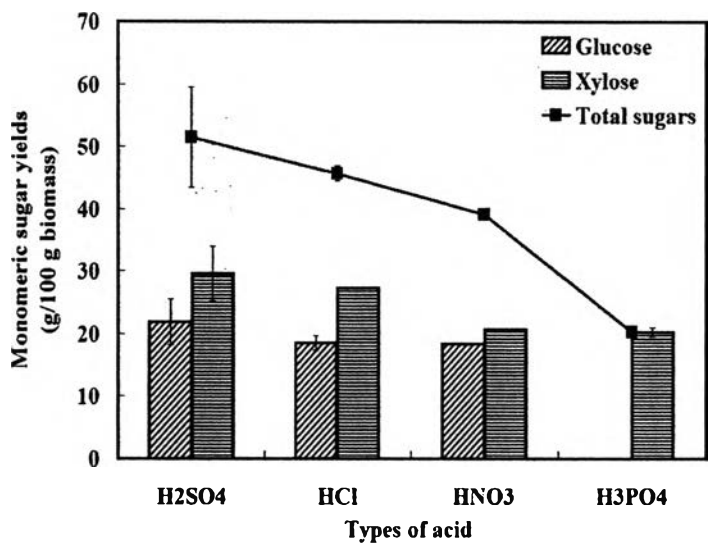

Figure 4.8 The effect of different types of dilute acid on the release of monomeric sugar of sugarcane bagasse.

4.4.5 Comparison of microwave- and conventional-heating assisted sulfuric acid pretreatment

In this part of work, sugarcane bagasse was pretreated with different H_2SO_4 concentrations using 30:1 LSR at 120 °C for 90 min under conventional heating. As shown in Table 4.9 and Figure 4.9, the highest glucose and xylose yields were obtained in 1.0–1.5 % (v/v) H_2SO_4. At the same operation conditions (0.5 % (v/v) H_2SO_4, 30:1 LSR, 120 °C and 90 min), the total sugar yield from microwave heating (15.80 g/100 g biomass, as shown in Table 4.5) was higher than that from conventional heating (13.67 g/100 g biomass, as shown in Table 4.9). Besides, at certain sulfuric acid level, microwave-heating gave a much higher total sugar yield with shorter reaction time than the conventional heating, as shown in Figure 4.10. The results revealed that microwave was an efficient heating method, leading to a much higher sugar yield with a shorter pretreatment time, as compared with the conventional heating.

Table 4.9 Monomeric sugar yields of sugarcane bagasse pretreated with different H_2SO_4 concentrations using 30:1 LSR at 120 °C for 90 min under conventional heating (g sugar/ 100 g biomass)

Acid concentration (% v/v)	Glucose	Xylose	Total monomeric sugars
0.5	4.69 ± 0.12	8.98 ± 0.21	13.67 ± 0.33
1.0	7.94 ± 0.02	13.07 ± 0.25	21.01 ± 0.27
1.5	7.06 ± 0.54	13.77 ± 0.66	20.83 ± 1.20
2.0	6.02 ± 0.35	10.09 ± 0.32	16.12 ± 0.67

Data are mean values ± S.D. of two replicates.

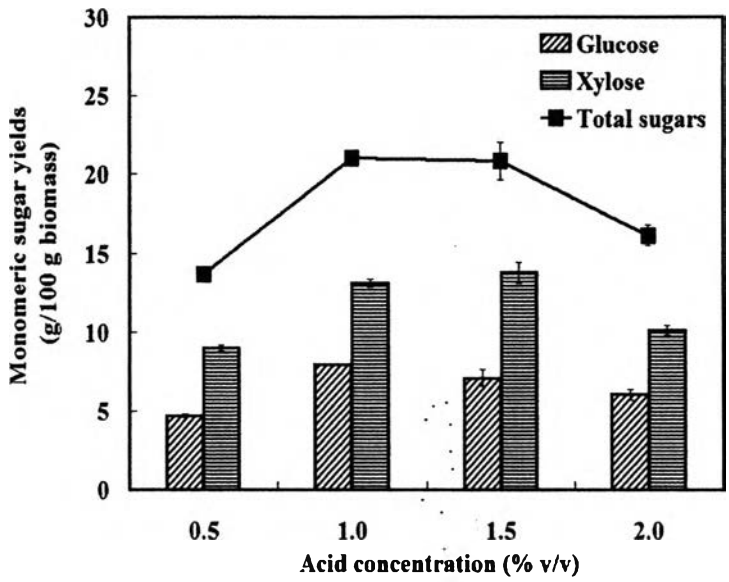

Figure 4.9 Effect of H_2SO_4 concentration on the release of monomeric sugars of sugarcane bagasse hydrolysates under conventional heating, using 30:1 LSR at 120 °C for 90 min.

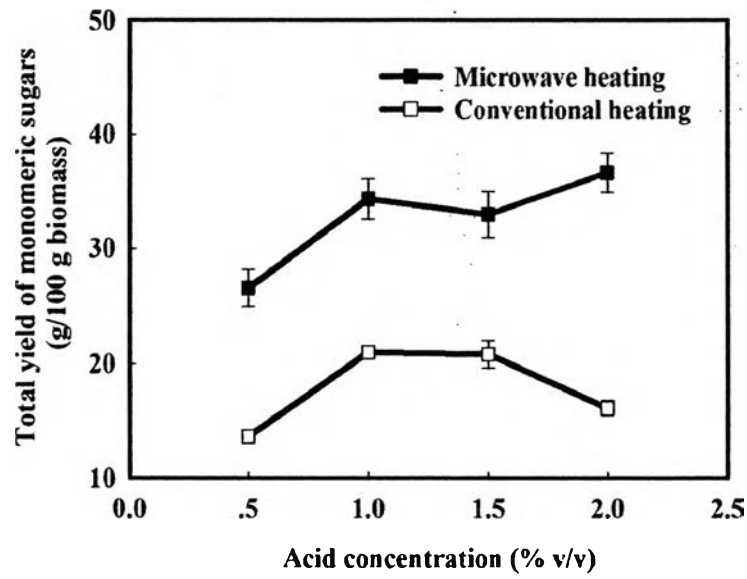

Figure 4.10 Comparison of the total monomeric sugar yields of sugarcane bagasse hydrolysates obtained from microwave- (Conditions: 30:1 LSR at 120 °C for 15 min) and conventional-heatingsnd conventional- (Conditions: 30:1 LSR at 120 °C for 90 min) heatings.

4.5 Conclusions

Sugarcane bagasse has a potential to be utilized in the sugar production. This work showed the benefit of using microwave as an efficient heating method in the pretreatment of sugarcane bagasse to produce monomeric sugars. Sugarcane bagasse could be hydrolyzed by NaOH and H_2SO_4 combined with microwave heating. Sugarcane bagasse pretreated using 2.0 % (v/v) H_2SO_4 with a 15:1 liquid-to-solid ratio at 120 °C for 15 min resulted in the highest monomeric sugar yields (21.89 g glucose and 29.61 g xylose per 100 g biomass), which was much higher than that pretreated using NaOH. As a result, the use of microwave was lower the hydrolysis time due to its high heating efficiency. The results of this work can serve as a step for further work of this area.

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