CHAPTER IV
RESULTS AND DISCUSSION

4.1 Optimization of Microwave-assisted Alkali Pretreatment

4.1.1 Effect of Time and Temperature

Ammonia-based pretreatment was used to remove lignocellulosic part, as described by Salvi *et al.* (2010). Ammonia, being a selective reagent for lignin and noncorrosive, serves as nitrogen source for microbes in downstream processes (Teymouri *et al.*, 2005). Moreover, cellulose and hemicelluloses are well preserved in the ammonia-based pretreatment process, with little or no degradation (Han *et al.*, 2009). The main effect of NH_4OH pretreatment is to delignify by breaking the ester bonds of cross-linking lignin (Demirbas, 2008) and the inhibitors were not created by NH_4OH pretreatment (Han *et al.*, 2009). *Miscanthus Sinensis* was pretreated with NH_4OH under the following conditions: 0.5% volume of NH_4OH, 15:1 of liquid to solid ratio, temperature in the range of 60–160 °C and time in the range of 5–60 min. In this study *Miscanthus Sinensis*, pretreated with NH_4OH at the temperature range of 60–160 °C for 5–60 min, resulted in the highest amount of monomeric sugars (about 1.37–1.47 g/100 g biomass) at 120 °C for 15 min (Table 4.1 and Figure 4.1e). Hu *et al.*, (2008) also showed that an increase of temperature resulted in not only a significant increase in lignin removal but also a decrease in the total sugar yield, indicating the decomposition of sugars. Therefore, the optimal temperature and time selected for the NH_4OH pretreatment were at 120 °C for 15 min to give the maximum glucose, xylose, and arabinose yields of 1.42, 0.66 g, and 0.66 g per 100 g biomass, respectively.

Table 4.1 Momomeric sugar yields of *Miscanthus Sinensis* hydrolyzed with 0.5 % (w/v) NH_4OH using 15:1 LSR under different times and temperatures (g sugar/100 g biomass)

Temperature (°C)	Pretreatment Time (min)	Glucose	Xylose	Arabinose	Total Monomeric Sugars
60	5	0.26 ± 0.02	0.07 ± 0.02	0.07 ± 0.02	0.40 ± 0.06
	10	0.36 ± 0.04	0.17 ± 0.05	0.19 ± 0.10	0.73 ± 0.19
	15	0.62 ± 0.10	0.39 ± 0.08	0.06 ± 0.08	1.06 ± 0.26
	30	1.19 ± 0.05	0.47 ± 0.07	0.05 ± 0.04	1.71 ± 0.16
	60	1.13 ± 0.08	0.23 ± 0.11	0.03 ± 0.02	1.38 ± 0.21
80	5	0.38 ± 0.03	0.41 ± 0.05	0.14 ± 0.03	0.93 ± 0.11
	10	0.48 ± 0.12	0.51 ± 0.07	0.20 ± 0.05	1.19 ± 0.24
	15	1.12 ± 0.09	0.61 ± 0.02	0.26 ± 0.08	1.89 ± 0.20
	30	1.35 ± 0.06	0.69 ± 0.10	0.42 ± 0.13	2.46 ± 0.29
	60	1.21 ± 0.05	0.58 ± 0.08	0.16 ± 0.06	1.95 ± 0.19
100	5	0.38 ± 0.11	0.13 ± 0.06	0.13 ± 0.05	0.63 ± 0.22
	10	0.71 ± 0.15	0.32 ± 0.10	0.25 ± 0.07	1.28 ± 0.26
	15	1.14 ± 0.08	0.53 ± 0.09	0.46 ± 0.11	2.12 ± 0.17
	30	1.10 ± 0.09	0.51 ± 0.16	0.49 ± 0.04	2.09 ± 0.22
	60	0.69 ± 0.03	0.16 ± 0.04	0.13 ± 0.05	0.98 ± 0.12
120	5	0.28 ± 0.05	0.16 ± 0.03	0.09 ± 0.04	0.54 ± 0.12
	10	0.91 ± 0.15	0.48 ± 0.09	0.10 ± 0.08	1.49 ± 0.32
	15	1.42 ± 0.05	0.66 ± 0.05	0.66 ± 0.08	2.74 ± 0.18
	30	1.09 ± 0.08	0.53 ± 0.06	0.45 ± 0.05	2.07 ± 0.19
	60	0.82 ± 0.06	0.33 ± 0.11	0.06 ± 0.02	1.21 ± 0.19
140	5	0.14 ± 0.04	0.02 ± 0.03	0.11 ± 0.06	0.28 ± 0.13
	10	1.10 ± 0.06	0.03 ± 0.02	0.14 ± 0.08	1.26 ± 0.16
	15	1.19 ± 0.10	0.07 ± 0.03	0.17 ± 0.07	1.42 ± 0.20
	30	1.32 ± 0.08	0.06 ± 0.02	0.09 ± 0.04	1.47 ± 0.14
	60	1.03 ± 0.21	0.04 ± 0.02	0.06 ± 0.04	1.13 ± 0.27
160	5	1.31 ± 0.11	0.01 ± 0.01	0.04 ± 0.03	1.37 ± 0.15
	10	1.62 ± 0.17	0.02 ± 0.01	0.05 ± 0.03	1.69 ± 0.21
	15	1.53 ± 0.15	0.02 ± 0.02	0.04 ± 0.01	1.59 ± 0.18
	30	1.41 ± 0.10	0.02 ± 0.01	0.03 ± 0.02	1.46 ± 0.13
	60	1.45 ± 0.23	0.01 ± 0.01	0.03 ± 0.02	1.49 ± 0.26

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

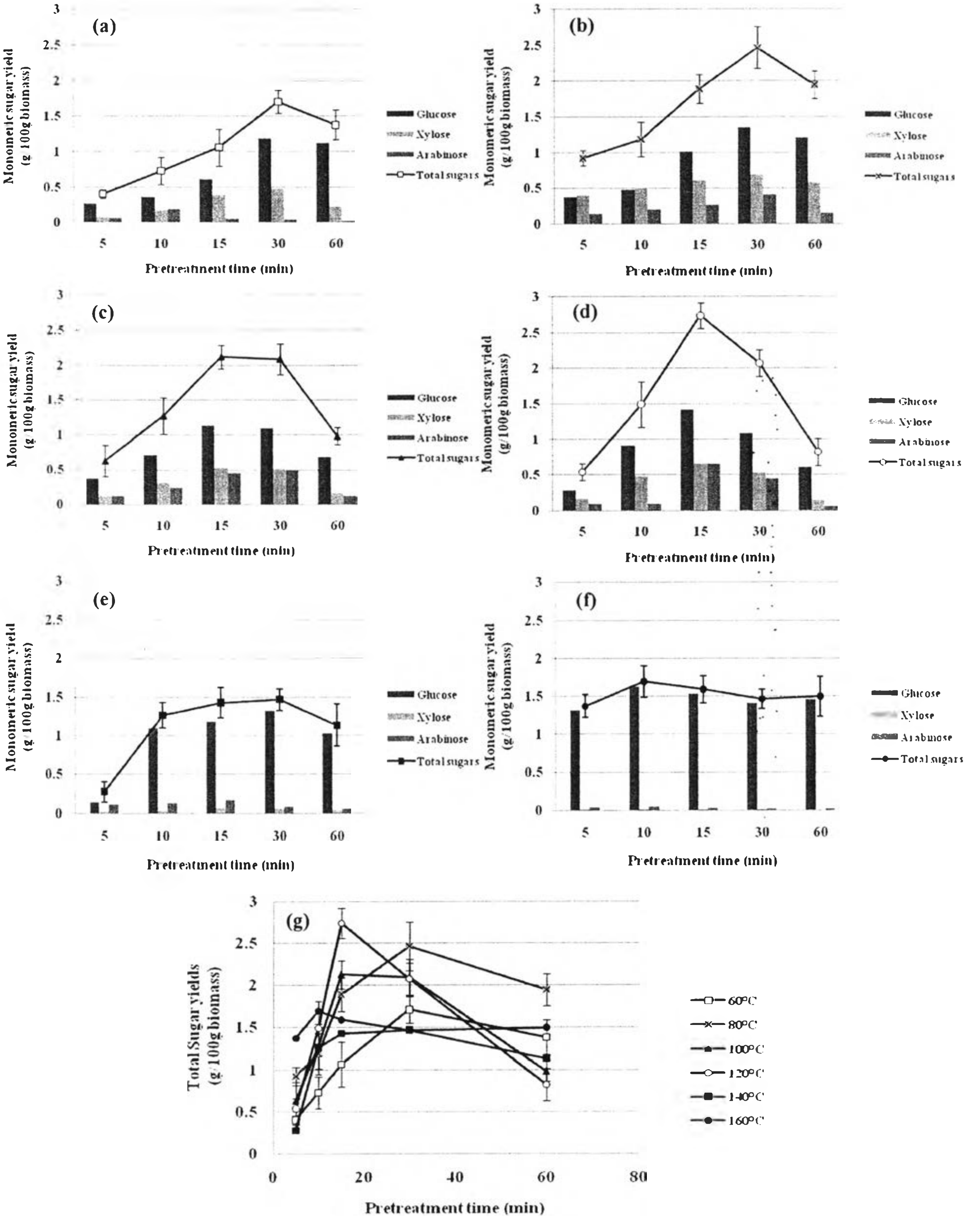

Figure 4.1 The main components of *Miscanthus Sinensis* hydrolysate using 0.5 % (w/v) NH_4OH and different times and temperatures: (a) 60 °, (b) 80 °, (c) 100 °, (d) 120 °, (e) 140 °C, (f) 160 °C, and (g) comparison of the total yield of monomeric sugars at different temperatures.

4.1.2 Effect of Alkali Concentration

To study the effect of alkali concentration on the release of monomeric sugars, *Miscanthus Sinensis* was pretreated with various NH_4OH concentrations (0.5 to 5.0 % w/v) at 120 °C for 15 min. It was found that NH_4OH concentration had a significant influence on the sugar yield, as shown in Table 4.2 and Figure 4.2. The yield of the sugar released from the pretreated *Miscanthus Sinensis* was increased from 0.5 to 1.0 % (w/v) NH_4OH with the highest total monomeric sugar yield of 2.93 g/100 g biomass, using 1.0 % (w/v) NH_4OH. Severe conditions of this pretreatment method caused a high degree of degradation of the released sugars, indicated by the low yields of glucose, xylose and arabinose (Hu *et al.*, 2008).

Table 4.2 Monomeric sugar yields of NH_4OH-pretreated *Miscanthus Sinensis* using 15:1 LSR at 120 °C for 15 min with different NH_4OH concentrations (g sugar/100 g biomass)

Alkali Concentration (% w/v)	Glucose	Xylose	Arabinose	Total Monomeric Sugars
0.5	1.42 ± 0.05	0.66 ± 0.05	0.66 ± 0.08	2.74 ± 0.18
1.0	1.37 ± 0.08	0.86 ± 0.03	0.70 ± 0.07	2.93 ± 0.18
2.0	1.37 ± 0.08	0.38 ± 0.09	0.41 ± 0.11	2.16 ± 0.28
3.0	0.76 ± 0.07	0.07 ± 0.13	nd	0.83 ± 0.20
4.0	0.47 ± 0.04	nd	nd	0.47 ± 0.04
5.0	0.35 ± 0.15	nd	nd	0.35 ± 0.15

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

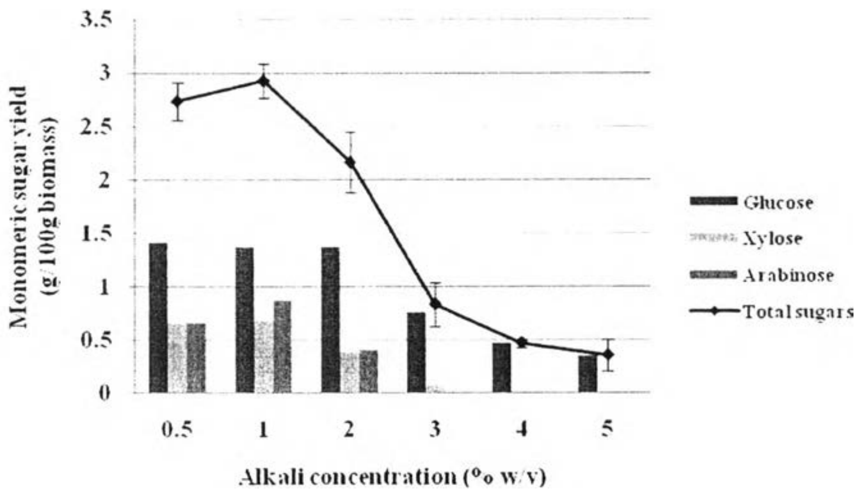

Figure 4.2 Effect of NH_4OH concentration on the release of monomeric sugars of NH_4OH- pretreated *Miscanthus Sinensis* using 15:1 LSR at 120 °C for 15 min.

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

The LSR effect on the monomeric sugar production of *Miscanthus Sinensis* was evaluated using LSR of 15:1, 30:1, and 45:1 at 1.0 % (w/v) NH_4OH, 120 °C for 15 min since LSR lower than 15:1 cause non- homogeneous because of less liquid being present. Table 4.3 and Figure 4.3 demonstrate that the release in the monomeric sugars decreased with increasing the LSR due to the more dilution of the mixture (Cara *et al.*, 2007; Kim *et al.*, 2007). The highest amount of the total monomeric sugar yield of 2.93 g/100 g biomass, containing 1.37 g of glucose, 0.86 g of xylose, and 0.70 g of arabinose (per 100 g biomass), was obtained at 15:1 LSR. Therefore, the LSR of 15:1 was selected for further study.

Table 4.3 Monomeric sugar yields of Miscanthus Sinensis using 1.0 % (w/v) NH_4OH at 120 °C for 15 min with different LSRs (g/100 g biomass)

Liquid to Solid Ratio (LSR)	Glucose	Xylose	Arabinose	Total Monomeric Sugars
15:1	1.37 ± 0.08	0.86 ± 0.03	0.70 ± 0.07	2.93 ± 0.18
30:1	0.90 ± 0.09	0.41 ± 0.04	0.48 ± 0.04	1.79 ± 0.17
45:1	0.48 ± 0.05	0.29 ± 0.07	0.25 ± 0.04	1.01 ± 0.16

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

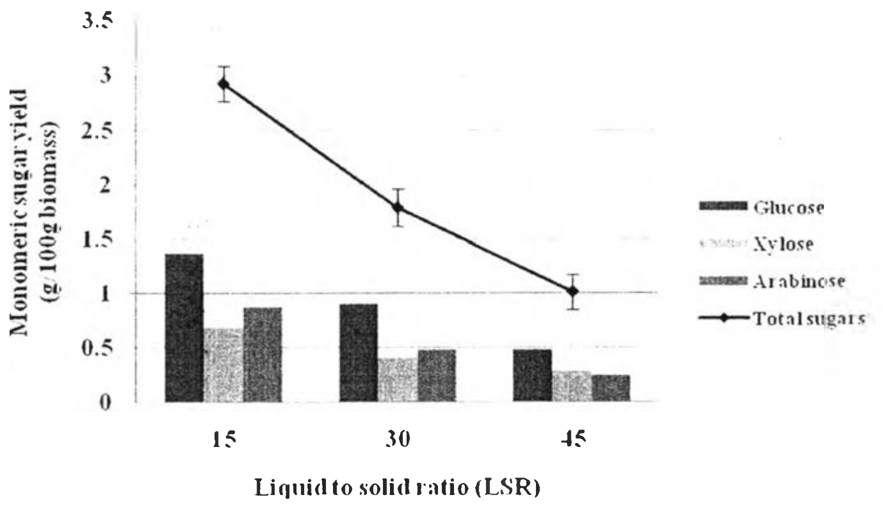

Figure 4.3 Effect of LSR on the release of the monomeric sugars of NH_4OH-pretreated Miscanthus Sinensis (conditions: 1.0 % (w/v) NH_4OH at 120 °C for 15 min).

4.2 Optimization of Microwave-assisted Acid Pretreatment

4.2.1 Effect of Time and Temperature

Since H_2SO_4 is the most common acid used to digest biomass, thus, was the first choice to digest *Miscanthus Sinensis* in this study at the temperature range of 60–160°C for 5–60 min, using 0.5 % w/v and 15:1 LSR. Table 4.4 and Figure 4.4 show that xylose was the main product, meaning that the main reaction occurs during the acid pretreatment is the hydrolysis of xylan in hemicellulose. It was observed that the longer pretreatment time gave the higher monomeric sugar yield (Figure 4.4 a-f). The highest total monomeric sugar yield, 24.58 g/100 g biomass, was achieved at 140 °C for 30 min. It is worth noting that under these conditions, the highest xylose yield of 18.98 g/100 g biomass was detected, but the highest glucose and arabinose yields of 6.87 and 2.76 g/100 g biomass, were achieved at 160 °C for 60 and 5 min, respectively (Figure 4.4 e-f). It is due to the fact that glucose was the main component released from cellulose, which has more ordered structure than hemicelluloses, thus, requires longer pretreatment time and higher reaction temperature for glucose production (Liao *et al.*, 2006). The decreasing trends in the monomeric sugar yield at too long pretreatment time was due to the occurrence of the decomposition reaction of sugar to inhibitory compounds, such as furfural, hydroxymethylfurfural generated in acid condition (Palmqvist *et al.*, 2000; Demirbas, 2008). Noureddini and Byun, (2010) showed the decreasing trend in the formation of monomeric sugars at severe condition correlated well with the formation of furfural. For the rate of sugar decomposition under dilute-acid conditions, xylose is more sensitive to the acidity and high temperature conditions and glucose is more resistant to harsh conditions (Taherzadeh and Karimi, 2007). Therefore, the optimal temperature and time selected for the H_2SO_4 pretreatment were at 140 °C for 30 min to give the maximum glucose, xylose, and arabinose yields of 2.87, 18.98, and 2.73 g per 100 g biomass, respectively.

Table 4.4 Monomeric sugar yields of *Miscanthus Sinensis* hydrolyzed, using 0.5 % (w/v) H_2SO_4, 15:1 LSR under different times and temperatures (g sugar per 100 g biomass)

Temperature (°C)	Pretreatment Time (min)	Glucose	Xylose	Arabinose	Total Monomeric Sugars
60	5	0.30 ± 0.05	0.38 ± 0.12	0.17 ± 0.08	0.85 ± 0.25
	10	0.77 ± 0.07	0.92 ± 0.04	0.25 ± 0.07	1.94 ± 0.18
	15	0.95 ± 0.11	1.12 ± 0.05	0.42 ± 0.04	2.49 ± 0.20
	30	1.02 ± 0.14	1.94 ± 0.08	0.43 ± 0.03	3.39 ± 0.25
	60	0.92 ± 0.05	1.10 ± 0.12	0.15 ± 0.11	2.17 ± 0.28
80	5	0.60 ± 0.09	0.21 ± 0.04	0.14 ± 0.05	0.95 ± 0.18
	10	0.80 ± 0.13	1.00 ± 0.02	0.76 ± 0.08	2.57 ± 0.23
	15	2.00 ± 0.06	2.72 ± 0.05	1.60 ± 0.08	6.33 ± 0.19
	30	1.16 ± 0.12	1.36 ± 0.14	0.97 ± 0.02	3.48 ± 0.28
	60	0.90 ± 0.05	0.92 ± 0.08	0.74 ± 0.11	2.55 ± 0.24
100	5	1.36 ± 0.06	1.30 ± 0.11	1.21 ± 0.70	3.87 ± 0.87
	10	1.03 ± 0.28	2.00 ± 0.26	1.58 ± 0.22	4.61 ± 0.76
	15	1.62 ± 0.28	3.26 ± 0.22	1.58 ± 0.26	6.46 ± 0.76
	30	1.51 ± 0.25	10.84 ± 0.35	1.86 ± 0.24	14.20 ± 0.84
	60	0.99 ± 0.30	6.25 ± 0.30	1.74 ± 0.29	8.98 ± 0.89
120	5	0.47 ± 0.28	2.87 ± 0.73	1.30 ± 0.29	4.64 ± 1.30
	10	0.84 ± 0.23	7.39 ± 0.72	1.45 ± 0.10	9.68 ± 1.05
	15	1.58 ± 0.24	11.49 ± 0.35	1.82 ± 0.27	14.89 ± 0.86
	30	2.74 ± 0.04	18.63 ± 1.01	2.39 ± 0.07	23.77 ± 1.12
	60	2.88 ± 0.26	18.14 ± 0.48	2.27 ± 0.25	23.29 ± 0.99
140	5	0.98 ± 0.23	10.63 ± 0.65	1.36 ± 0.32	12.98 ± 1.20
	10	1.96 ± 0.12	15.37 ± 0.74	2.48 ± 0.24	19.80 ± 1.12
	15	2.28 ± 0.12	16.72 ± 1.08	2.71 ± 0.30	21.70 ± 1.50
	30	2.87 ± 0.18	18.98 ± 0.82	2.73 ± 0.30	24.58 ± 1.30
	60	2.69 ± 0.14	17.47 ± 0.67	2.53 ± 0.19	22.69 ± 1.00
160	5	4.00 ± 0.35	16.49 ± 0.70	2.76 ± 0.20	23.25 ± 1.20
	10	4.18 ± 0.25	17.58 ± 0.32	1.96 ± 0.29	23.72 ± 0.86
	15	4.07 ± 0.35	16.51 ± 0.85	2.23 ± 0.30	22.82 ± 1.50
	30	6.16 ± 0.27	13.82 ± 0.73	2.11 ± 0.20	22.09 ± 1.20
	60	6.87 ± 0.39	8.20 ± 0.43	0.08 ± 0.07	15.15 ± 0.89

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

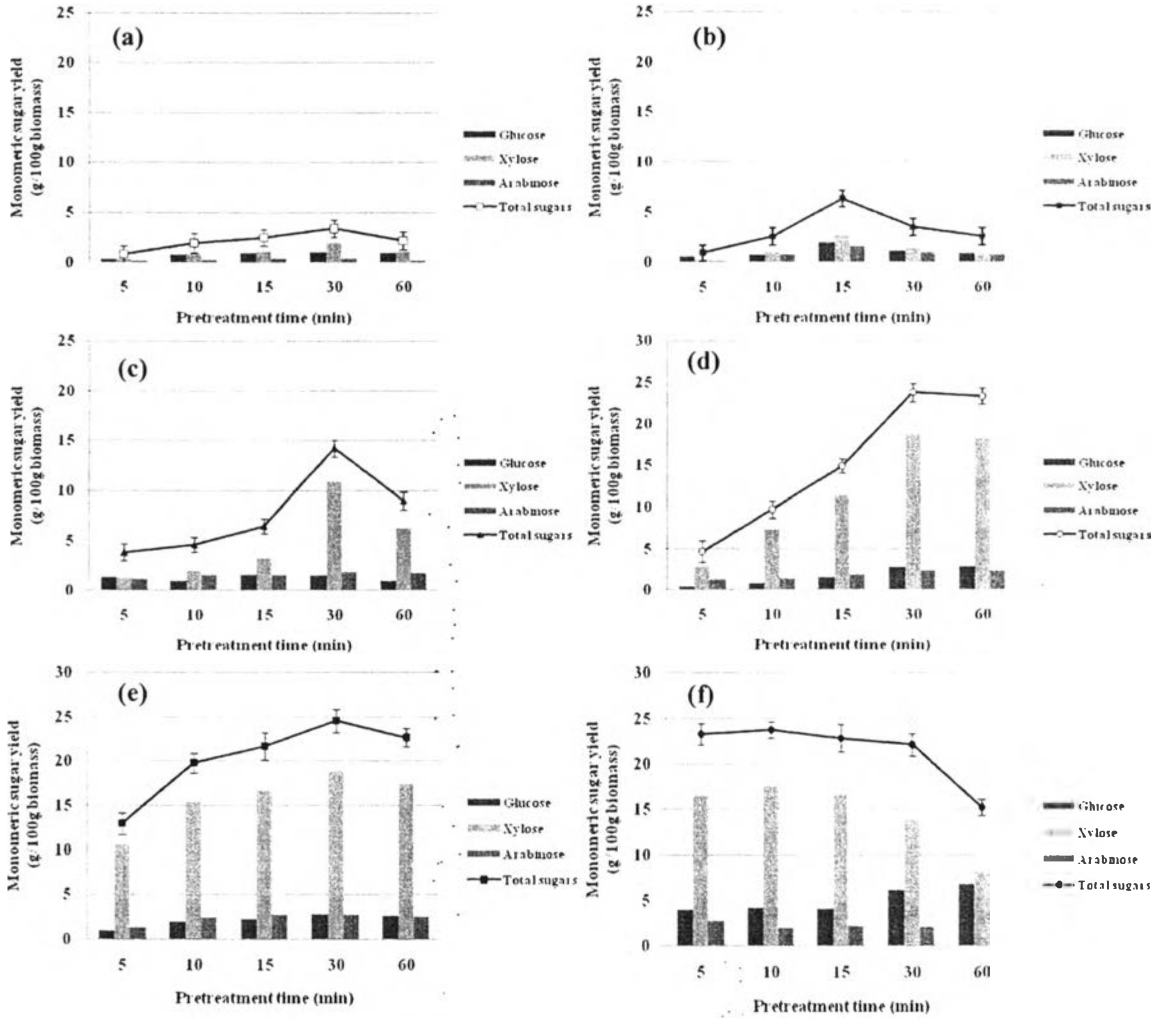

Figure 4.4 The main components of *Miscanthus Sinensis* hydrolysate using 0.5 % (w/v) H_2SO_4, 15:1 LSR, and different times and temperatures: (a) 60 °, (b) 80 °, (c) 100 °, (d) 120 °, (e) 140 °, and (f) 160 °C.

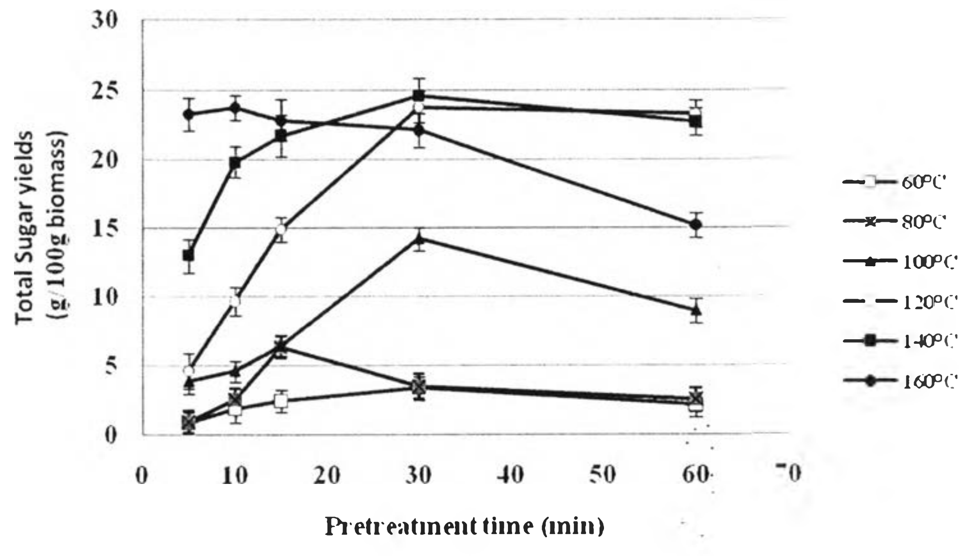

Figure 4.5 Comparison of the total yields of monomeric sugars using 0.5 % (w/v) H_2SO_4, 15:1 LSR, and different times and temperatures.

4.2.2 Effect of Acid Concentration

Miscanthus Sinensis was pretreated using various concentration of H_2SO_4 (0.5, 1.0, 2.0, 3.0, 4.0 and 5.0 % w/v), 15:1 LSR at 140 °C for 30 min. Table 4.5 and Figure 4.6 show an increase in releasing monomeric sugars when increasing acid concentration from 0.5 to 3.0 % w/v and it decreased thereafter owing to too concentrated acid causing the decomposition of sugars, as discussed earlier. The highest total monomeric sugar yield of 29.25 g/100 g biomass, containing glucose, xylose, and arabinose yields of 8.77, 18.98, and 5.06 g/ 100 g biomass, respectively, was obtained. The reason for achieving xylose as the highest product is because hemicelluloses, mainly composing of xylan, are easier to break down in acid pretreatment than cellulose (Wang *et al.*, 2010; Liao *et al.*, 2006). The yields of arabinose and glucose progressively increased with the increase in severity.

Table 4.5 Monomeric sugar yields of *Miscanthus Sinensis* pretreated with different H_2SO_4 concentrations using 15:1 LSR at 140 °C for 30 min (g sugar per 100 g biomass)

Acid Concentration (% w/v)	Glucose	Xylose	Arabinose	Total Monomeric Sugars
0.5	2.87 ± 0.18	18.98 ± 0.82	2.73 ± 0.30	24.58 ± 1.30
1.0	5.43 ± 0.28	16.32 ± 0.80	2.85 ± 0.42	24.61 ± 1.50
2.0	6.73 ± 0.32	16.15 ± 0.71	3.58 ± 0.07	26.47 ± 1.10
3.0	8.77 ± 0.37	15.42 ± 0.62	5.06 ± 0.29	29.25 ± 1.28
4.0	8.62 ± 0.24	15.21 ± 0.75	4.28 ± 0.24	28.11 ± 1.23
5.0	7.49 ± 0.24	15.08 ± 0.65	4.33 ± 0.32	26.90 ± 1.21

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

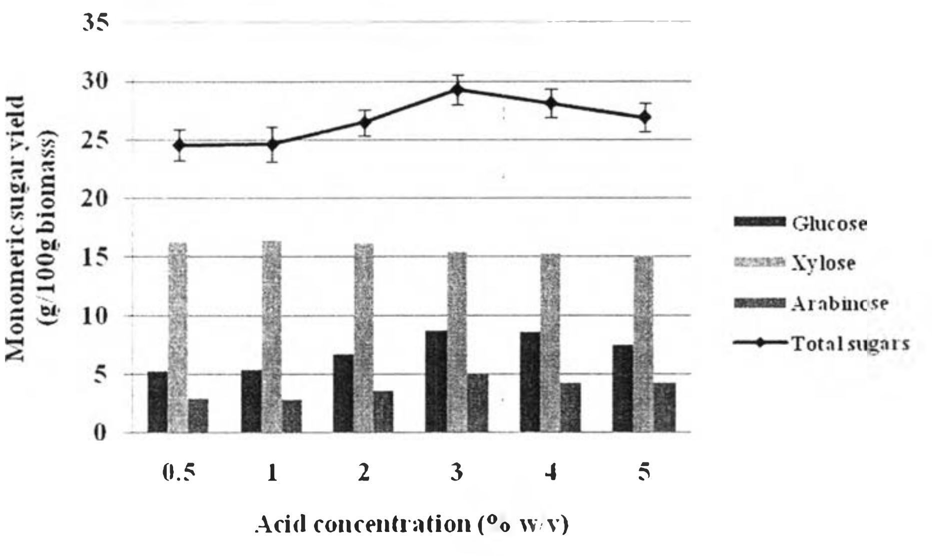

Figure 4.6 Effect of H_2SO_4 concentration on the release of monomeric sugars of H_2SO_4- pretreated *Miscanthus Sinensis* using 15:1 LSR at 140 °C for 30 min.

4.2.3 Effect of Liquid-to-Solid Ratio

In this case, *Miscanthus Sinensis* was pretreated in various LSRs with 3.0 % (w/v) H_2SO_4 at 140 °C for 30 min, see Table 4.6 and Figure 4.7, revealing that an increase in LSR resulted in a decrease in the release of the monomeric sugars, similar to the NH_4OH pretreatment. The highest amount of the total monomeric sugar yield of 29.25 g/100 g biomass, containing the maximum glucose, xylose, and arabinose yields of 7.60, 17.09, and 4.56 (g/100 g biomass), was obtained at the 15:1 LSR. Again, the pretreatment was not performed with the LSR lower than 15:1 because of non-homogeneity of the system. As a result, the optimal conditions of the H_2SO_4 pretreatment for treating *Miscanthus Sinensis* were; 3.0 % (w/v) H_2SO_4 with 15:1 LSR at 140 °C for 30 min, giving the highest glucose, xylose, and arabinose yields of 8.77, 15.42, and 5.06 g/100 g biomass, respectively.

Table 4.6 Monomeric sugar yields of *Miscanthus Sinensis* using 3.0 % (w/v) H_2SO_4 at 140 °C for 30 min with different LSRs (g/100 g biomass)

Liquid to Solid Ratio (LSR)	**Glucose**	**Xylose**	**Arabinose**	**Total Monomeric Sugars**
15:1	8.77 ± 0.37	15.42 ± 0.62	5.06 ± 0.29	29.25 ± 1.28
30:1	4.21 ± 0.21	12.37 ± 0.59	1.61 ± 0.32	18.18 ± 1.12
45:1	3.27 ± 0.30	7.17 ± 0.55	2.09 ± 0.13	12.53 ± 0.98

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

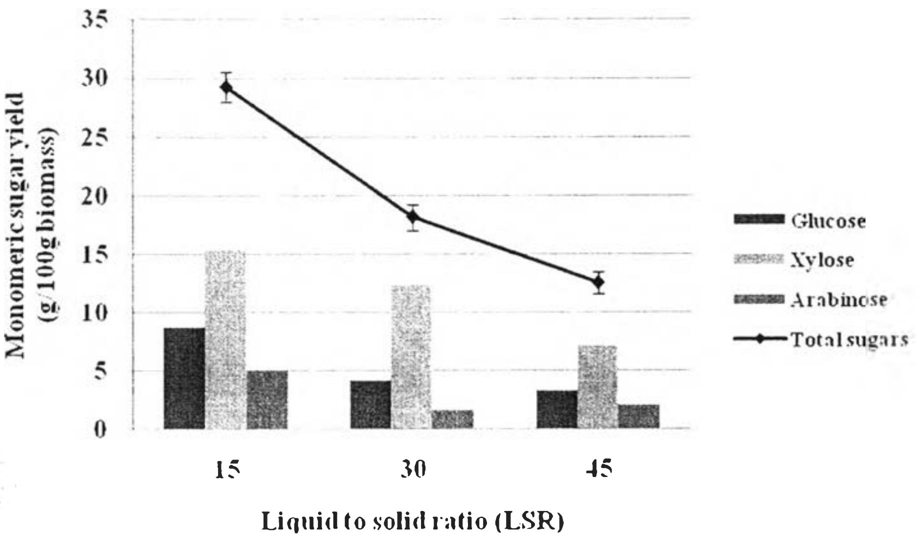

Figure 4.7 Effect of LSR on the release of monomeric sugars of H_2SO_4-pretreated *Miscanthus Sinensis* (conditions: 3.0 % (w/v) H_2SO_4 at 140 °C for 30 min).

4.3 Comparison of Dilute-acids

The optimal condition obtained from the H_2SO_4 pretreatment of *Miscanthus Sinensis* was used to study the effect of acid type, using HCl, HNO_3, and H_3PO_4, as can be seen in Table 4.7 and Figure 4.8. Remarkably, the total monomeric sugar produced from the H_3PO_4 pretreatment was much higher than the others, giving the highest total monomeric sugar yield of 62.28 g/100 g biomass, while H_2SO_4, HCl, and HNO_3 gave 29.25, 23.89, and 11.24 g/100 g biomass, respectively. The level and composition of the monomeric sugars released depends on the type of acid and its concentration (Hernández-Salas *et al.*, 2009). According to Whitmore and Atalla (1985), used phosphoric acid for the regeneration of cellulose I. Studied show that cellulose swells rapidly in phosphoric acid solutions. Wei *et al.* (1996) show the potential of using phosphoric acid on decrystallization and dissolution of cellulose. The highest amount of the total monomeric sugar yield, 62.28 g/100 g biomass, from the H_3PO_4 pretreatment contains the maximum glucose, xylose, and arabinose yields of 41.05, 18.69, and 2.53 g biomass, respectively. Interestingly, in the case of H_3PO_4 after neutralization of hydrolysates with NaOH (for fermentation process using microorganisms), the salt formed is sodium phosphate which can be used as a nutrient for microorganisms. Therefore, an operation of filtration to remove the salts, which is another advantage to improve the economic process, is not needed. Moreover, it is environmental friendly since the salt formed is not a waste (Gámez *et al.*, 2006).

Table 4.7 Monomeric sugar yields of *Miscanthus Sinensis* pretreated with 3.0 % (w/v) of different dilute acids using 15:1 LSR at 140 °C for 30 min (g/100 g biomass)

Types of Acid	Glucose	Xylose	Arabinose	Total Monomeric Sugar
H_2SO_4	8.77 ± 0.37	15.42 ± 0.62	5.06 ± 0.29	29.25 ± 1.28
HCl	4.10 ± 0.37	17.13 ± 0.74	2.66 ± 0.34	23.89 ± 1.45
HNO_3	3.13 ± 0.33	7.28 ± 0.47	0.83 ± 0.32	11.24 ± 1.12
H_3PO_4	41.05 ± 1.12	18.69 ± 0.86	2.53 ± 0.52	62.28 ± 2.54

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

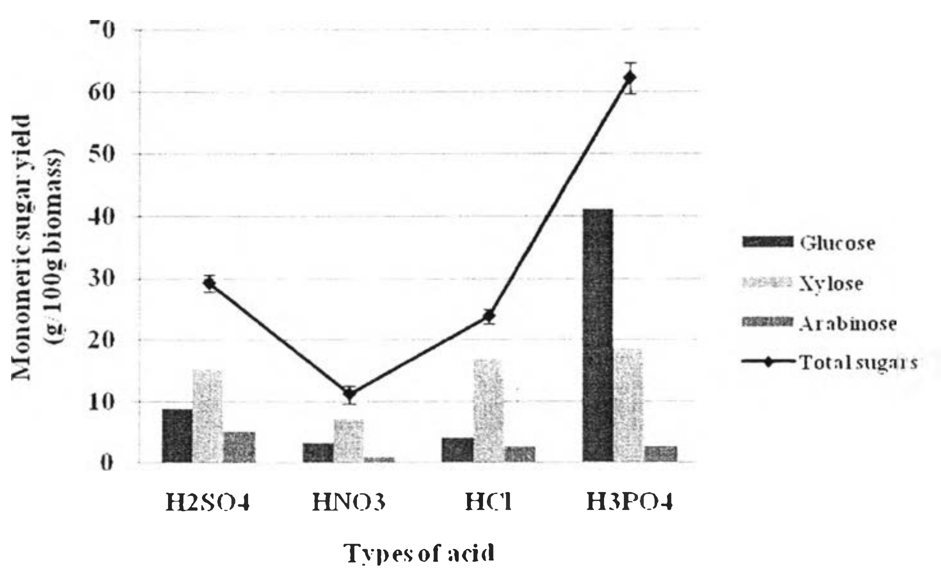

Figure 4.8 The effect of different types of dilute acid on the release of monomeric sugar of *Miscanthus Sinensis*.

4.4 Two-stage Pretreatment (Dilute Ammonium Hydroxide Followed by Dilute Phosphoric Acid Pretreatment)

After alkali pretreatment using following conditions: 15:1 LSR at 1.0 % (w/v) NH_4OH, 120 °C for 15 min, the solid was washed with water until neutrality and then the pretreated solid was further treated with 3.0 % (w/v) H_3PO_4 using 15:1 LSR at 140 °C for 30 min. Wang *et al.* (2010) show the two-stage pretreatment significantly enhanced cellulose digestibility due to a partial removal of hemicelluloses and lignin after first stage pretreatment, the cellulose fibers were exposed. The sugar yields obtained from the two stage pretreatment compared to the single stage pretreatment, as presented in Table 4.8 and Figure 4.9, were the highest, 71.64 g/100 g biomass.

Table 4.8 The effect of pretreatment processes on the release of monomeric sugar yields of *Miscanthus Sinensis*

Pretreatment Process	Glucose	Xylose	Arabinose	Total Monomeric Sugar
NH_4OH	1.37 ± 0.08	0.86 ± 0.03	0.70 ± 0.07	2.93 ± 0.18
H_3PO_4	41.05 ± 1.12	18.69 ± 0.86	2.53 ± 0.52	62.28 ± 2.54
Two-stage	29.66 ± 1.01	15.13 ± 0.97	2.97 ± 0.14	71.64 ± 2.12

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

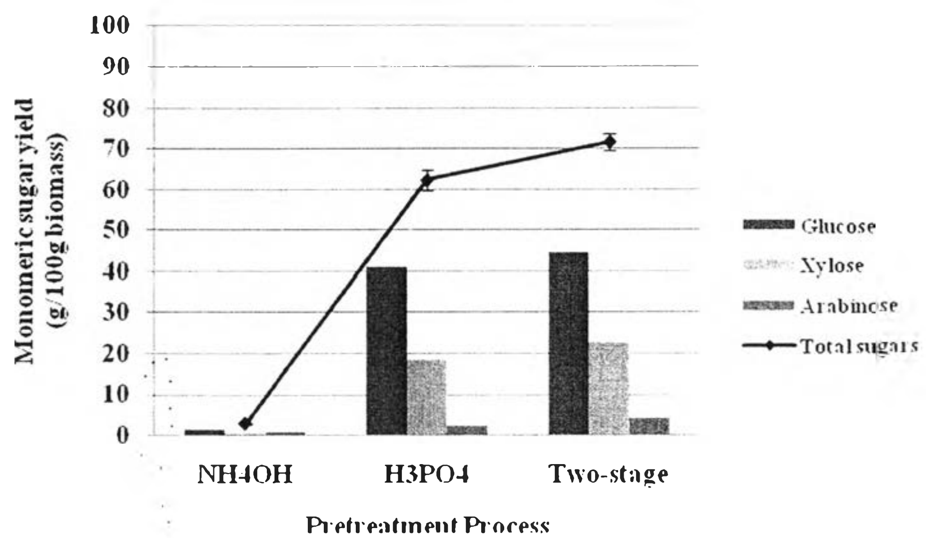

Figure 4.9 The effect of pretreatment processes on the release of monomeric sugar yields of *Miscanthus Sinensis*.

4.5 Effect of Pretreatment on Chemical Composition

The chemical composition change of *Miscanthus Sinensis*, after each pretreatment, is listed in Table 4.9. The *Miscanthus* treated with the two-stage pretreatment via microwave irradiation composed of the lowest extractives, ash, lignin, and hemicellulose contents, indicating that the two-stage pretreatment efficiently removed extractives, ash, lignin, and hemicellulose in Miscanthus Sinensis. All these results show that the alkali pretreatment removed most of the lignin and the acid pretreatment mainly removed hemicelluloses, consistent with Zhu *et al.* (2006) and Han *et al.* (2009).

Table 4.9 Chemical composition of the residues after pretreatment for three microwave/chemical pretreatment processes

Pretreatment Method	Composition, %			
	Extractives	Ash	Hemicellulose	Lignin
Untreated	8.6	7.7	31.3	17.4
Microwave/alkali	1.8	5.0	32.9	12.2
Microwave/acid	2.0	5.6	20.7	15.8
Microwave/alkali/acid	1.5	4.2	18.4	10.4

4.6 FT-IR Analysis

Figure 4.10 shows FTIR spectra of untreated and pretreated *Miscanthus*. By comparing the FTIR spectra of alkali, acid, and two-stage pretreated *Miscanthus* (Figure 4.10B-D, respectively) with that of untreated *Miscanthus* (Figure 4.10A). According to Wang *et al.* (2010), the peak at 1734 cm^{-1} represents the complex linkages between hemicelluloses and lignin, such as ester-linked acetyl, feruloyl, and p-coumaroyl groups, and the spectrum of the *Miscanthus* the alkali pretreatment (Figure 4.10B) shows no peak at this position, implying that the linkages were broken. The lignin peak around 1253 cm^{-1} was also diminished. After alkali and two-stage pretreatment (Figure 4.10B and 4.10D), the polysaccharides peaks (898, 1108, 1164, 1260, 1325, and 1378) became sharper, as compared with untreated *Miscanthus*, corresponding well to the increase in polysaccharides content after the pretreatment.

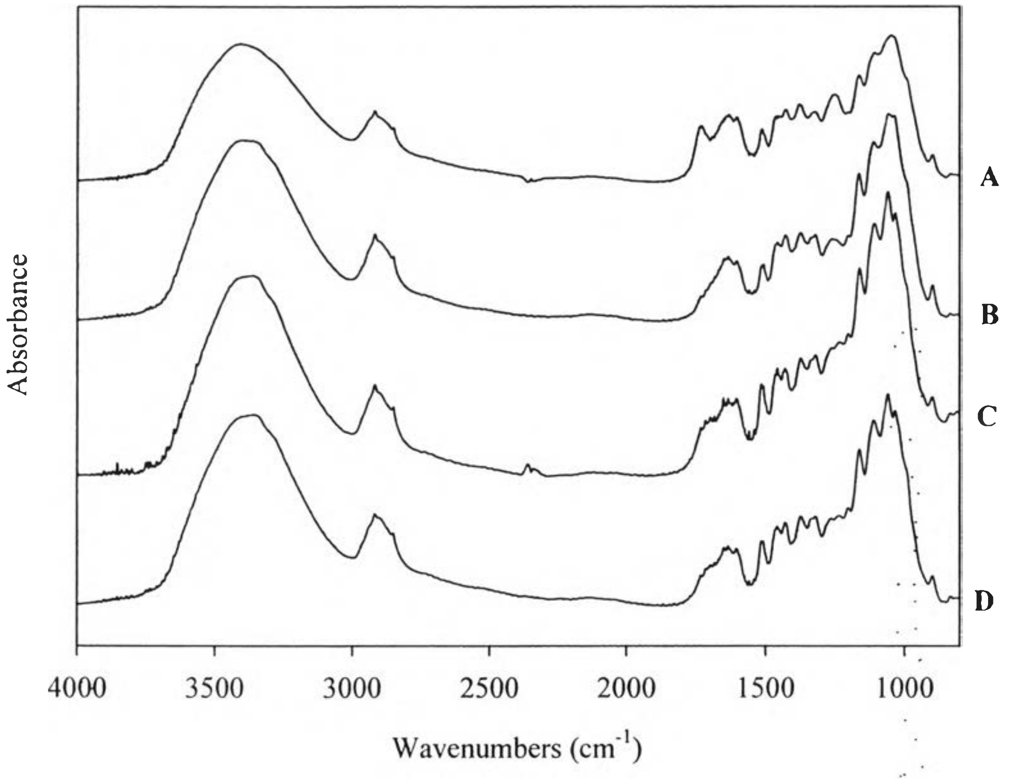

Figure 4.10 FTIR spectrum of (A) raw *Miscanthus*, (B) alkali-preteated *Miscanthus*, (C) acid-pretreated *Miscanthus*, and (D) Two-stage pretreated *Miscanthus*.