Lactic Acid Production from High Concentration of Invert Sugar in Semi-Batch Reactor

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Abstract The objective in this study is the production of high yield of lactic acid production from invert sugar in alkaline degradation at mild reaction condition. The invert sugar was immediately and continuously added into an alkaline solution in a batch and semi-batch reactor, respectively. The effect of batch and semi-batch, initial concentration of sodium hydroxide and initial of concentrated raw materials were studied. Semi-batch provided better yield than batch reactor. High sodium hydroxide concentration with low invert sugar in semi-batch operation was found to have high lactic acid. The feed concentration of invert sugar at 30, 45 and 70% provided lactic acid yield around 65-68% for excess sodium hydroxide base at 50°C and 10 hr.

Keywords: lactic acid; invert sugar; semi-batch reactor

1. Introduction

Lactic acid is a potential acid, it is used in many industries such as food industry, medical, cosmetics, packaging. Currently, it is more interesting because it is monomer for polylactic acid (PLA), which is a bioplastic replace to plastic from petroleum. Plastic from polylactic acid is easy to degrade, so it is environmentally friendly material. Lactic acid can be produced either fermentation or chemical synthesis. In present, the most lactic acid was produced with fermentation because it got high yield of lactic acid more than 90% (Abdel-Rahman et. al., 2013). However, the control of growth of microorganism was difficult and took long fermentation time.

Alkaline degradation, a chemical synthesis, was a simple technique, low cost and took short time of reaction. There have been few studies of alkaline degradation because it got lower yield than fermentation. Nimela (1990) got 30 and 32% yield of lactic acid at 175 and 190 °C, respectively from 1.5 g xylan/50 ml which was treated with 3 M sodium hydroxide for 2 hrs. Yang and Montgomery (1996) got 40 and 20.4% yield of lactic acid at 1.8 and 50% w/w glucose at 100 °C. Brujin et.al (1986) studied the reaction between 0.025M monosaccharide with 0.01 M potassium hydroxide at 78 °C, it got about 6-7% lactic acid and other hydroxyacid such as saccharinic acid, glycolic acid, 2,4-dihydroxybutyric acid and >C6 acids.

From the previous research, low concentration of raw material was used as raw material and the batch reactor was used for this reaction. The reaction between alkaline and low concentration of monosaccharide can be reacted in batch reactor. When high concentration of monosaccharide was used and reacted with high concentration of alkaline, the heat was generated and the temperature increased more than 110 °C, that is resulting from exothermic reaction (Yang and Montgomery, 2007). When the high concentration of both raw materials were used, the high temperature occurred and the batch was
not suitable for this reaction. The semi-batch reactor that have been used for this reaction before, will be studied in this experiment.

Several biomass such as bagasse, starch and xylan, can be used as raw material in alkaline degradation process. Those biomass can be easily founded in Thailand and low cost, but it used high temperature to get lactic acid. When high temperature condition was used, it might degrade the lactic acid, so the yield of lactic acid will decreased. Monosaccharide is good raw material for alkaline degradation (Yang and Montgomery, 2007), the reaction temperature when monosaccharide was used was low. This research will used sucrose that is disaccharide and can be easily hydrolyzed to invert sugar as raw material. After that it was treated with alkaline to produce lactic acid at mild temperature.

2. Material and Method

2.1 Material and Experiment setup

Sucrose was purchased from supermarket in Thailand. Sodium hydroxide (NaOH) and sulfuric acid (H₂SO₄) are analytical grade while methanol is HPLC grade.

The in-house reactor was equipped with glass jacket to circulate water at desired temperature. The solution was welled-mix magnetic stirrer. For semi-batch test, the metering pump, Dosser model MPA 0120PV, was used to feed invert sugar solution into the reactor.

2.2 Method

2.2.1 The effect of batch and semi-batch

The 8.5 M sodium hydroxide was added into reactor first and heated at desired temperature. After that 30% w/v of invert sugar was immediately feed (batch) and continuously feed (semi-batch) at 0.2 ml/min into sodium hydroxide solution. The ratio of invert sugar : sodium hydroxide is 1:1 by volume. For batch experiment, the samples were taken periodically until 8 hr while the samples from semi-batch were taken at 8.3 hr (finish feed) and 10 hr. All samples were taken for analyse for the yield of lactic acid. Some samples were analysed the reducing sugar for the percent of conversion.

2.2.2 The effect of initial concentration of sodium hydroxide

The concentration of sodium hydroxide were varied at 3.5, 5.5, 8.5 and 10.2 M sodium hydroxide. The sodium hydroxide solution was heated at 50 °C after that 30% w/v of invert sugar was gradually feed into sodium hydroxide. The samples were taken at 8.3, 10 and 24 hr.

2.2.3 The effect of high concentration of raw material

All experiments were run at 50 °C in semi-batch reactor. Three raw materials were prepared. First raw material was 30% w/v invert sugar and 8.5 M sodium hydroxide, second was 45% w/v and 10.2 M NaOH, and third raw material was 70% w/v and 15 M sodium hydroxide. The samples were taken at 8.3 and 10 hr.

2.2.4 Analytical Procedure

Samples were taken at desired time and acidified to pH 2. High performance liquid chromatography with Plastisil ODS column was used to analyze lactic acid concentration. The detector was UV at 210 nm. The 5 mmol sulfuric acid was mobile phase at flow rate of 0.8 ml/min.
Samples were taken and stopped the reaction at 4 °C. Reducing sugar content was determined by DNSA method.

3. Result and Discussion

The batch experiments were tested at 50 and 70 °C. The samples were taken at 10, 30, 60, 120, 240, 360, 480 min as shown in figure 1. It could be observed that the conversion about 90% within 60 and 10 min at 50 and 70 °C, respectively. The conversion of both samples about 97% at 240 min.

The yield of lactic acid were increased when time increased. The reaction at 70 °C was faster than 50 °C, it showed that the reaction rate increase with temperature. The yield of lactic acid at 70 °C about 50% at 10 min and gradually increased until 55% at 8 hr. While the yield was only 16% at 50 °C and 10 min. At 50 °C, when time increased the yield was 57% at 120 min after that the yield almost constant and the highest yield was 60% at 8 hr. At 8 hr, the yield of lactic acid at 70 °C were lower yield than at 50 °C might cause the highest temperature after reaction started were 58 and 93 °C at 50 and 70 °C, respectively. At high temperature, the formation of C_7 or high molecular weight species takes place. This is a result of recombination in fragments of pyruvaldehyde and C_6 α-dicarbonyl (Bruijin et al., 1986; Shaffer and Friedemann, 1930). Therefore the yield of lactic acid was decreased at high temperature.

The lactic acid yield of in batch and semi-batch reactor were showed in figure 2. For batch reactor after 2 hr the yield was almost constant when time increased. At 50 °C, the yield was 57.2 and 59.5% at 2 and 8 hr, respectively. At 70 °C, the yield was 52.3 and 55.3% at 2 and 8 hr, respectively. The yield was almost constant during 2-8 hr because of the conversion of invert sugar was 96% at 2 and 1 hr for the temperature of reaction as 50 and 70 °C, respectively. The yield of semi-batch were 64.5 and 67.5% at 50 °C and 72.3 and 71.9% at 70 °C. Although the reaction time of semi-batch took longer time than batch, but the yield were high at both temperature. It showed that the semi-batch is better than batch. It might be the control temperature in semi-batch reactor was better, the temperature of mixture increased less than 1 °C. Moreover, the semi-batch can control high concentration of sodium hydroxide all the time reaction which improved yield of lactic acid (Bruijin et al., 1986).

![Figure 1](image1.png)

**Figure 1.** The yield of lactic acid and conversion of invert sugar at various time in batch reactor.
The 30% w/v invert sugar was reacted with the concentration of sodium hydroxide of 3.5, 5.5, 8.5 and 10.2 mol/L. The results were shown in figure 3. When initial concentration of sodium hydroxide increase, the lactic acid will increase. This results correspond with previous research. Bruijin et.al. (1986) found that as concentration of OH⁻ increase, the lactic acid and saccharinic acid were high whereas the acetic acid, formic acid and >C6 acid were low. The yield of lactic acid at 24 hr were 52, 65, 71 and 73% at 3.5, 5.5, 8.5 and 10.2 M of sodium hydroxide, respectively. The yield between 8.5 and 10.2 M sodium hydroxide were little difference, so in this experiment, 8.5 M of sodium hydroxide is suitable for 30% w/v invert sugar.

When the concentrated invert sugar was used, the initial concentration of sodium hydroxide should increase to maintain high OH⁻ in the mixture. Therefore the initial concentration of sodium hydroxide will increase as the concentration of invert sugar increased.

When 70% invert sugar as raw material and reacted with 15 M sodium hydroxide in batch reactor, it is runaway because it was highly exothermic when adding invert sugar in alkaline solution. Therefore, the batch reactor was impossible for alkaline degradation with high concentration of raw
material. The semi-batch was suitable for high concentration of raw material because it can controlled temperature of reaction and controlled OH in the system.

The three experiments in semi-batch reactor were set and compared the lactic acid yield as shown in figure 4. The yield of lactic acid at 10 hr. were 68, 66 and 65% at 30, 45 and 70% of invert sugar, respectively. When high concentration of sugar was used, the lactic acid will decrease and saccharinic acid will increase (Bruijin et.al., 1986). Therefore the yield was decrease as the concentration of invert sugar was increased. However, this research used very high concentrated invert sugar that has not been investigated before and got high yield. Moreover, the high concentrated of raw material, it had low water, was suitable for the further purification step after lactic acid production.

![Figure 4. The effect of concentrated raw material on yield of lactic acid](image)

4. Conclusions

The semi-batch reactor was suitable for the alkaline degradation of concentrated invert sugar. When high concentration of sodium hydroxide was used, the yield of lactic acid will increased. The yield of lactic acid got about 65-68% as 30, 45 and 70% invert sugar was used at 50 °C.

5. References

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