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APPENDICES

Appendix A Analytical Procedures

1. Detergent Analysis

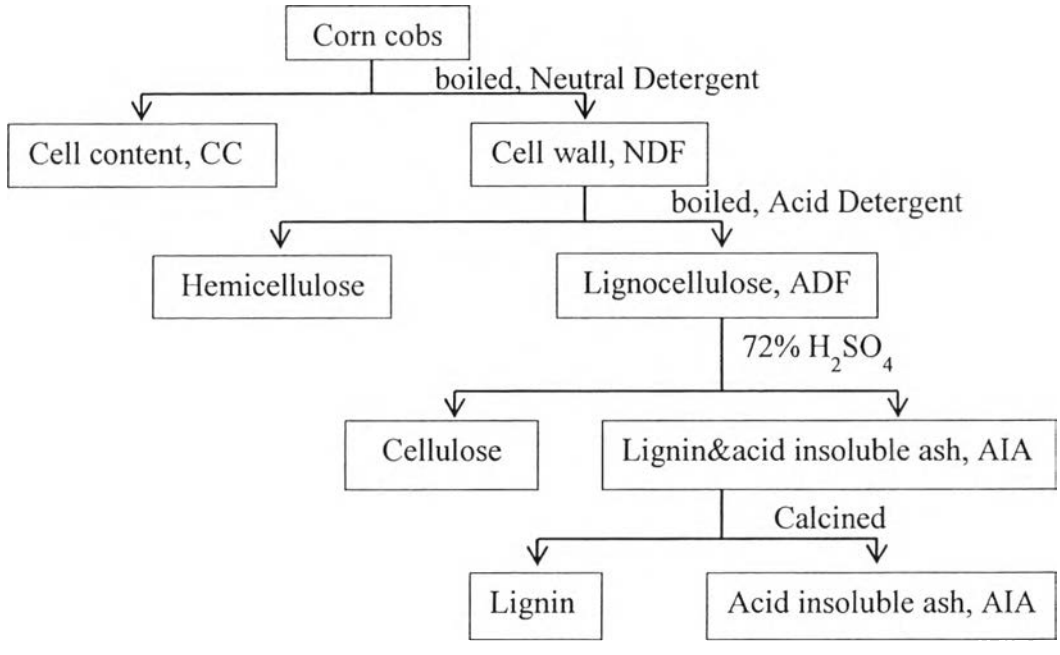


Figure A1 Schematic of detergent analysis procedure flow diagram.

1.1 Neutral detergent fiber (NDF)

Neutral Detergent Fiber (NDF) is cell wall constituents which are not dissolve in neutral solution. NDF consists of hemicellulose, cellulose, lignin, cutin, and keratin.

Reagents

1. Distiled or deionized water
2. Acetone (AR grade)
3. Sodium sulphite
4. Neutral detergent

Procedures

1. Dry crucible at 100 °C for 1 h, after that cool to room temperature at desiccators, and then weigh (W_1).

2. Weigh 1.02 g of dried sample and weigh (W_2).

3. Add 0.5 g of sodium sulphite into crucible.

4. Place crucible from no.3 in Hot Extraction Unit 1020

5. Add 100 mL of neutral detergent into column and reflux for 60 minutes.

6. Rinse sample into crucible with minimum of hot (90°C–100°C) water until the pH of solution reach neutral pH.

7. Wash sample in crucible with 25 mL of acetone for 3 times.

8. Dry sample at 100 °C for 5 h, after that cool to room temperature at desiccators, and then weigh (W_3).

9. Ash residue in crucible for 2 h at 550 °C, after that cool to room temperature at desiccators, and then weigh (W_4).

10. Determine %NDF:

$$\%NDF = \frac{W_4 - W_3}{W_2} \times 100$$

1.2 Acid detergent fiber (ADF)

Acid detergent fiber (ADF) is cell wall constituents which are not dissolve in acid solution. ADF mainly consists of cellulose and lignin, and small amount of cutin. Therefore, loss of product in acid solution should be hemicellulose.

Reagents

1. Distiled or deionized water

2. Acetone (AR grade)

3. Sulfuric acid (H_2SO_4)

Procedures

1. Dry crucible at 100 °C for 1 h, after that cool to room temperature at desiccators, and then weigh (W_1).

2. Weigh 1.02 g of dried sample and weigh (W_2).

3. Add 0.5 g of sodium sulphite into crucible.

4. Place crucible from no.3 in Hot Extraction Unit 1020

5. Add 100 mL of acid detergent into column and reflux for 60 minutes.

6. Rinse sample into crucible with minimum of hot (90°C–100°C) water until the pH of solution reach neutral pH.

7. Wash sample in crucible with 25 mL of acetone for 3 times.

8. Dry sample at 100 °C for 5 h, after that cool to room temperature at desiccators, and then weigh (W_3).

9. Ash residue in crucible for 2 h at 550 °C, after that cool to room temperature at desiccators, and then weigh (W_4).

10. Determine %ADF:

$$\%ADF = \frac{W_4 - W_3}{W_2} \times 100$$

11. Determine % Hemicellulose:

$$\%Hemicellulose = \%NDF - \%ADF$$

1.3 Acid detergent lignin (ADL)

Acid detergent lignin (ADL) is cell wall constituents which are not dissolve in acid solution. ADF mainly consists of cellulose and lignin, and small amount of cutin. Therefore, loss of product in acid solution should be hemicellulose.

Reagents

1. Distiled or deionized water

2. 72% Sulfuric acid (H_2SO_4)

Procedures

1. Fill crucible, which contains the sample from ADF analytical, about half full with 72% H_2SO_4 and stir.

2. Refill with 72% H_2SO_4 and stir at hourly intervals as acid drains away.

3. After 3 h, filter off as much acid as possible with vacuum and wash contents with hot water until free from acid.

4. Dry sample at 100 °C for 5 h, after that cool to room temperature at desiccators, and then weigh (W_3).

5. Ignite crucible in muffle furnace for 2 h at 550 °C, after that cool to room temperature at desiccators, and then weigh (W_4).

6. Determine %Lignin:

$$\%ADL = \frac{W_4 - W_3}{W_2} \times 100$$

7. Determine % Cellulose:

$$\%Cellulose = \%ADL - \%ADF$$

2. Monosaccharide Analysis

1. Prepare a series of sugar calibration standards in HPLC grade water at concentrations appropriate for creating a calibration curve for each sugar of interest.

2. Prepare a set of multi-component standards containing glucose, xylose, and arabinose in the range of 0.2 - 20.0 g/L.

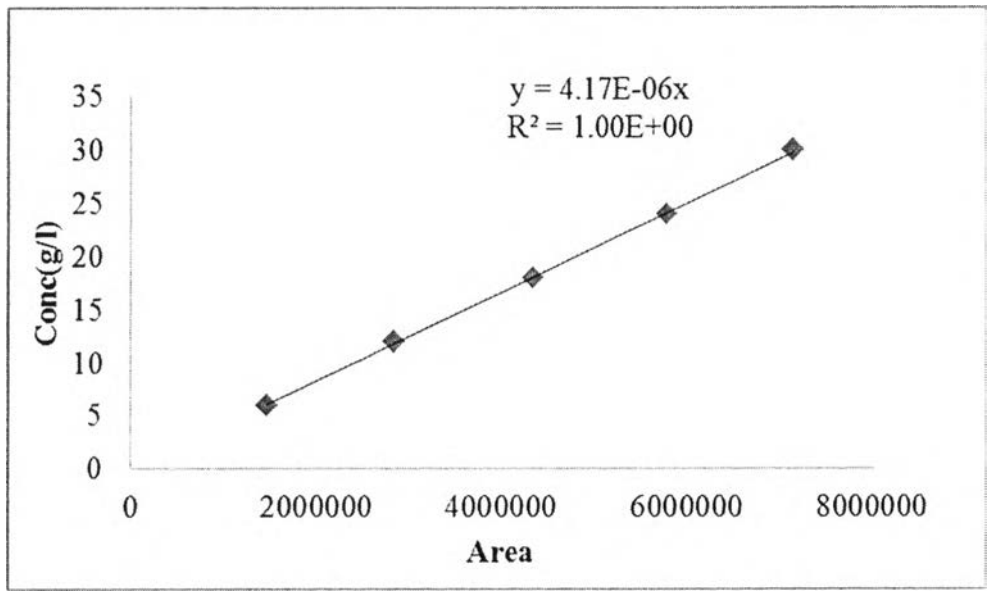
3. Analyze the calibration standards, the calibration verification standards, and the samples by HPLC using an organic acid column (Aminex HPX- 87H column, Bio-Rad Lab, USA) for glucose, xylose, and arabinose. HPLC system equipped with a refractive index detector (Model 6040 XR, Spectra-Physics, USA). The column was used with 0.005 M sulfuric acid solution as a mobile phase. The flow rate was controlled at 0.6 mL min^{-1} for 15 min and the column temperature was 65 °C.

Table A1 The retention time of standard

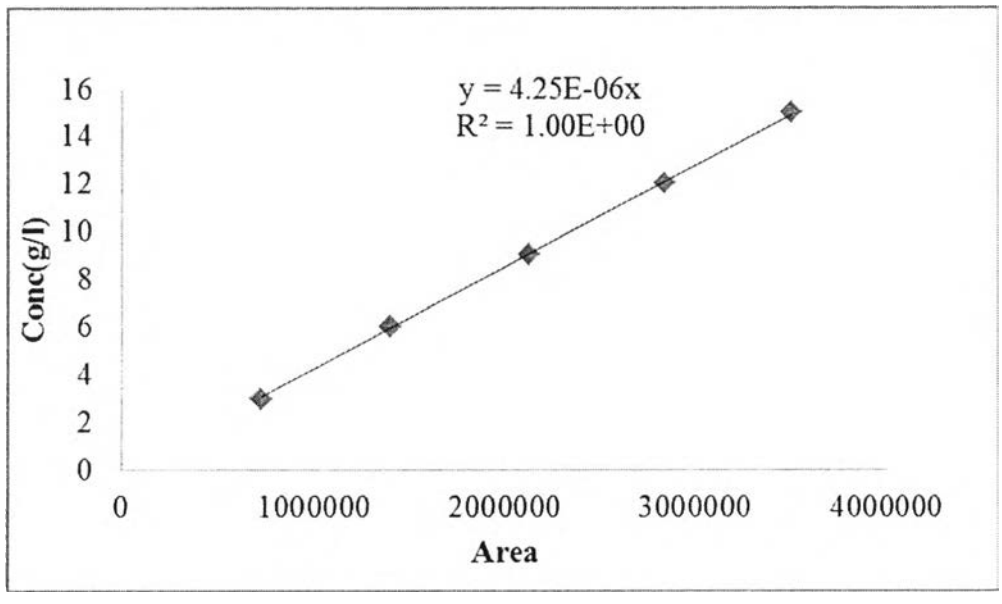
Standard	Aminex column (min)
Glucose	8.638
Xylose	9.216
Mannose	9.114
Galactose	9.137
Cellubiose	7.143
Arabinose	9.939
Rhamnose	N/A
Furfural	N/A

N/D; Not Determine

A) Standard glucose



B) Standard xylose



C) Standard ardinose

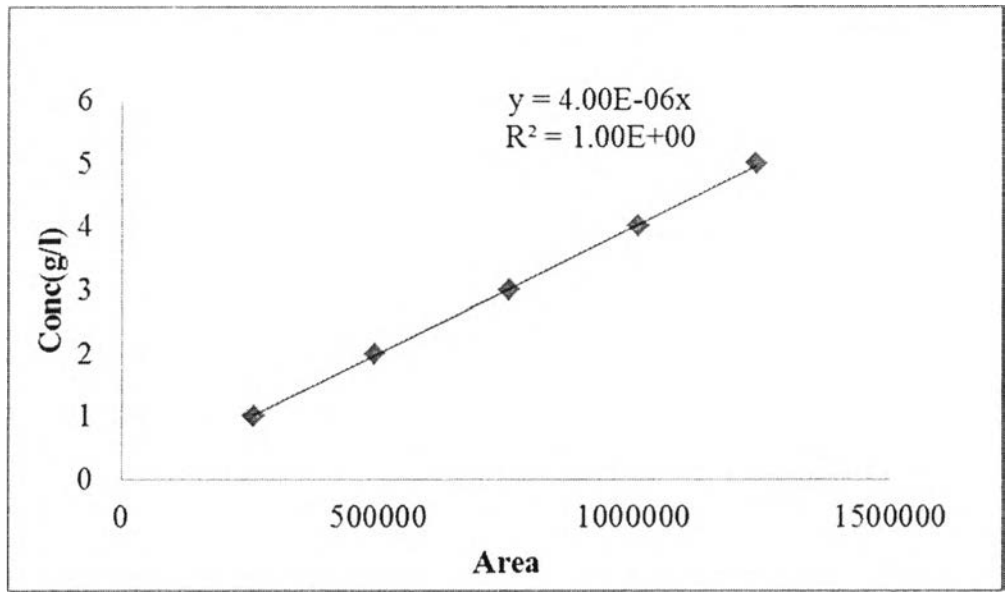


Figure A2 Calibration curve for each sugar: (A) glucose, (B) xylose, and (C) arabinose.

3. Ethanol Production Analysis

The fermentable sugars were produced from only enzymatic hydrolysis step, which they were fermented to ethanol by using *Saccharomyces cerevisiae* yeast from 1 day to 3 days at 37 °C. The solution was analyzed by gas chromatography at faculty of Pharmacy, Chulalongkorn university. The calibration curve of ethanol was showed in Figure A3 for determining the quantity of ethanol in the solution. The standards of ethanol in various concentrations were prepared in the range 0.5 g/L to 10 g/L.

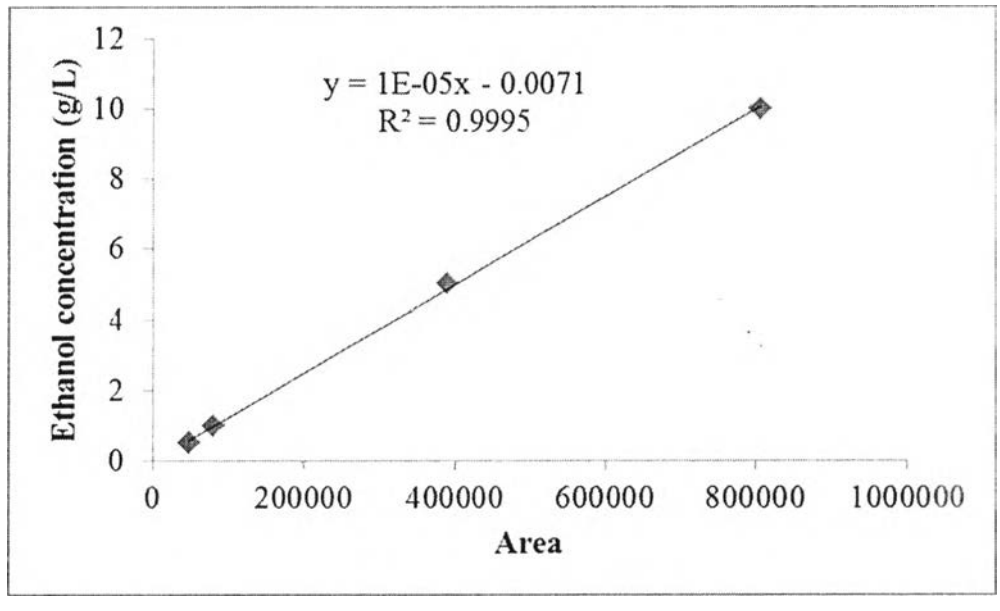


Figure A3 Calibration curve for ethanol production.

Appendix B Experimental Data for Chapter IV

1. Composition of Corn Cobs After Pretreatment and Enzymatic Hydrolysis

Table B1 Content of cellulose, hemicellose, and lignin in corn cobs

	Cellulose (g)	**Hemicellulose (g)**	**Lignin (g)**
Before pretreatment	0.8254	0.9200	0.1480
After pretreatment	0.7216	0.1570	0.0151
After enzymatic hydrolysis	0.1534	0.1471	0.0269

CURRICULUM VITAE

Name: Ms. Boonyisa Wanitwattanarumlug

Date of Birth: October 10, 1987

Nationality: Thai

University Education:

2006 – 2009 Bachelor Degree of Enigineering in Chemical Engineering, Faculty of Engineering, Kasetsart University. Bangkok, Thailand.

Work Experience:

2009 Position: Internship Student

Company: Thai Plastic and Chemicals Company (TPC), Thailand

Proceedings:

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