Production of Xylose from Corn Cobs using Sulphuric Acid

G. Shalini[1], Vanam Sudhakar[2], B. V. Satya Ramesh[3], Dr. M. Shyam Sundar[4]

[1]Project Assistant, [3]Principal Scientist , [4]Senior Technician, CSIR-Indian Institute of Chemical Technology, Hyderabad, India.

[2]Mtech Student, University College of Technology, Osmania University, Hyderabad, India.

Abstract*-Xylose is classified as a monosaccharide of the aldopentose type, which means that it contains five carbon atoms and includes an aldehyde functional group. It is derived from hemicellulose, one of the main constituents of biomass. Like most sugars, it can adopt several structures depending on conditions. With its free aldehyde group, it is a reducing sugar.Corn cobs are the cheaply available raw material containing 20% of xylan. The conversions of xylose at different temperatures are discussed in this work, in which we got optimum conversion of 90% at 120^{o}C. 2.0 kgs of corn cobs which are synthesized by acid hydrolysis was converted to xylose.During acid hydrolysis 120gm of concentrated sulphuric acid was diluted in 8L of water, the pressure was maintained at 1- 1.2 kg/cm^{2} throughout the reaction period of 3hrs.The reaction material after acid hydrolysis was passed through the centrifuge and Ion-Exchanger resin for the removal of suspended solids and acid, colour, salts from the reaction material respectively. The efficiency of product has been improved by using rota evaporation, there by improving quality of product by producing 200gm of pure xylose.

***Keywords:* Corn cobs, Acid hydrolysis, Conversion, Ion-Exchange and rota evaporation.**

I. INTRODUCTION

Xylose is a sugar first isolated from wood, and named for it. Xylose is a pentose processing, the molecular formulae $C_5H_{10}O_5$, which is found in Hemi-cellulose of natural substance of fibrous vegetables and fruit, as well as in Corn Cobs, Bagasse and various hard wood trees like Birch. Although xylose taste and looks exactly like sugar, which is really sugars mirror image. A corn cob is the central core of an ear of maize. It is the part of the ear on which the kernels grow. The ear is also considered a "cob" or "pole" but it is not fully a "pole" until the ear is shucked, or removed from the plant material around the ear. Young ears, also called baby corn, can be consumed raw, but as the plant matures the cob becomes tougher until only the kernels are edible.The innermost part of the cob is white and has a consistency similar to foam plastic.

Corncobs are an important by product of the sweet corn processing industry in Egypt, where they represent about 15% of the total corn production and the total volume of this by-product generated from the total volume of corn was estimated to be 54,424 ton in 2008 (personal communication, Egyptian Directorate of Agriculture).Worldwide, corncobs are either used as animal feed or returned to the harvested field as fertilizer.

Fig.1 Dry corn cobs

TABLE 1. Compositional Analysis Percentage.

Raw Material Compositional Analysis %	
Cellulose	39.1
Hemi-Cellulose	42.1
Lignin	9.1
Protein	1.7
Ash	1.2
Total	**100**

Due to their chemical composition, corn residues show great potential as a renewable raw material for producing a variety of added-value chemicals, such as lactic acid, citric acid, sugars, and ethanol. On the other hand, the secondary metabolites and constituents of corncobs remain unclear. Development of an efficient way to utilize corncobs will require additional research into the chemical nature of this environmental agro-waste and its potential application to the production of valuable chemicals and pharmaceuticals. Corncobs find use in the following applications:

- Industrial source of the chemical furfural
- Fibre in fodder for ruminant livestock (despite low nutritional value)
- Water in which corncobs have been boiled contains thickeners and can be added to soup stock or made into traditional sweetened corncob jelly
- Bedding for animals – cobs absorb moisture and provide a compliant surface

- A mild abrasive for cleaning building surfaces, when coarsely ground
- As a bio-fuel
- Charcoal production

Xylose is the main building block for the hemicellulose xylan. The corn cobs materials containing 20 % of xylan, which is synthesis by acid hydrolysis and converted to xylose having a low calorific value and exhibiting a sweetening power, having approximately equivalent to 67% that of sucrose. Xylose has 40 per cent fewer calories and 75 per cent fewer carbohydrates then sugar and it is slowly metabolized, resulting in very negligible changes in insulin.

Xylose is a versatile product having applications in various fields.

- Xylose is used as a source to prepare food for diabetic patients.
- It is used as an environmental friendly non-toxic water solution cleaning component.
- It is used in dying i.e., textile industry.
- It is used in tanning i.e., leather industry.
- It is in pharmaceutical industry.
- It is used as additive in colour photography.
- It is used as a brightener in acid zinc electroplating.
- It is used in the production of ethanol by fermentation.
- It is the starting point of one of the important derivatives like xylitol.
- It is used in the removal of chlorine from drinking water.
- It is used as an active oxygen generating source.
- It is used as an inhibitor in pharmaceutical industry.
- It is used as a raw material for producing xylitol.

II. OBJECTIVE OF THE WORK

In present process xylose produced from corn cobs after passing through many stages where corncobs are activated in acid hydrolysis to produce xylose and there by impurities are removed from Ion-exchange process. Maximum recovery of product is done in rotary evaporation step followed by crystallization and drying. 2kgs of corncobs producing 200grams of pure xylose. Xylose is also used as main raw material for xylitol process.

III. MATERIALS AND METHODOLGY

3.1 Raw Materials

Raw materials used in preparation of xylose are corncobs, sulphuric acid, Indion-890 (resin) and methanol.

3.2 Methodology

- Extraction of xylose from corn cobs

1. Washing
2. Acid Hydrolysis
3. Extraction
4. Centrifuge
5. De-colorization & Neutralization
6. Rota evaporation
7. Crystallization
8. Filtration
9. Drying (Finished Product)

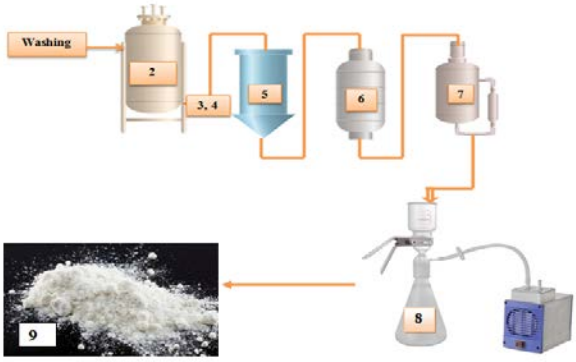


Fig.2 Process Flow Diagram

IV. EXPERIMENTAL PROCEDURE

4.1 Experimental Setup

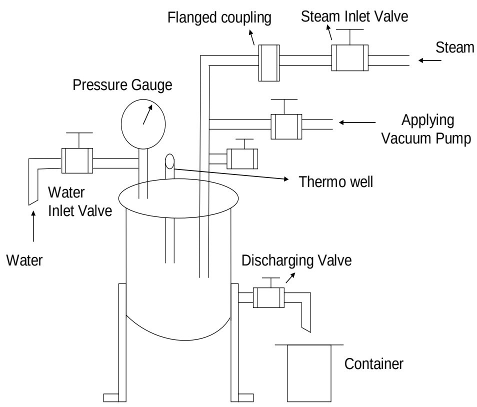


Fig.3 Stainless Reactor

4.2 Washing

Stainless reactor consists of the diameter 31cm and height of the reactor 68.58cm. 2 kgs of the corn cobs (dry) was fed into the reactor and to it 8 litres of water was added. Then the heat is supplied to the reactor by means of the steam and maintained at a pressure of 1 - 1.2 kg/cm^2. After obtaining the temperature of 120 ^{o}C, it was maintained for 30 min, it is washed for several times for removal of impurities such as dust, lignin, sand and other foreign bodies, after washing the discharge is collected out.

4.3 Acid Hydrolysis

To the above wet corn cobs, 120 grams of concentrated sulphuric acid (98.9%) was diluted in 8lts of water after dilution the concentration is 1: 6 ratio with respective to Corn cobs basis, the diluted sulphuric acid was fed to the reactor by means of vacuum pump, heating is given by means of steam and maintained at 120 ^{o}C at a pressure of 1-1.2 kg/cm^2 throughout the reaction period i.e., 3hrs in the mean for every 30 min reaction we have collected samples which were sent for HPLC analysis.After completion of the reaction period, the reaction material was discharged and it was collected in a container.

4.4 Extraction

To the above reacted corn cobs, 8 litres of the water was charged into the reactor for Crude Xylose Extraction, it was collected and recycled for about 30 min. After completion of extraction, the extracted material was collected in a container.

4.5 Centrifuge

The above reaction material (Acid Hydrolysis + 1st, 2nd extraction) was passed through the centrifuge for the removal of suspended particles and makes the material to be clear.

Fig.4 Centrifuge

4.6 De-Colourization and Neutralization

After completion of the centrifuge, it is then passed through the Ion-Exchanger resin. Resin particle size range from 0.3 to 1.2 mm for the removal of acid, colour, other sugar and salts from the reaction material and makes the material to be neutral and Colour less.

Fig.5 Ion-exchange column

4.7 Rotary Evaporation

The 10% Concentrated material which is obtained from the reverse osmosis is taken as a feed to the Rota Vapour. Here complete distillation or evaporation of water from the feed takes place and to it a methanol wash is given so that to remove the left-out moisture from the product. Thus after completion of the Rota Evaporation, Crude product is obtained.

Fig.6 Viscous Mass after Rotary Evaporation

4.8 Crystallization

To the Crude Product a measured quantity of methanol is added which is about one third of the crude product obtained and mixed it thoroughly, a pinch of fine powdered pure Xylose is seeded and kept aside for crystallization process.

4.9 Filtration

After completion of the crystallization, it is filtered under G3 filter funnel by using a measured quantity of methanol, in the presence of vacuum pump for filtration purpose. Mother liquor is collected at the bottomand filtered xylose at the top.

Fig.7 Vacuum Filtration

4.10 Drying (Finished Product)

After completion of filtration, the finished product is kept under vacuum, to make the product complete dry.

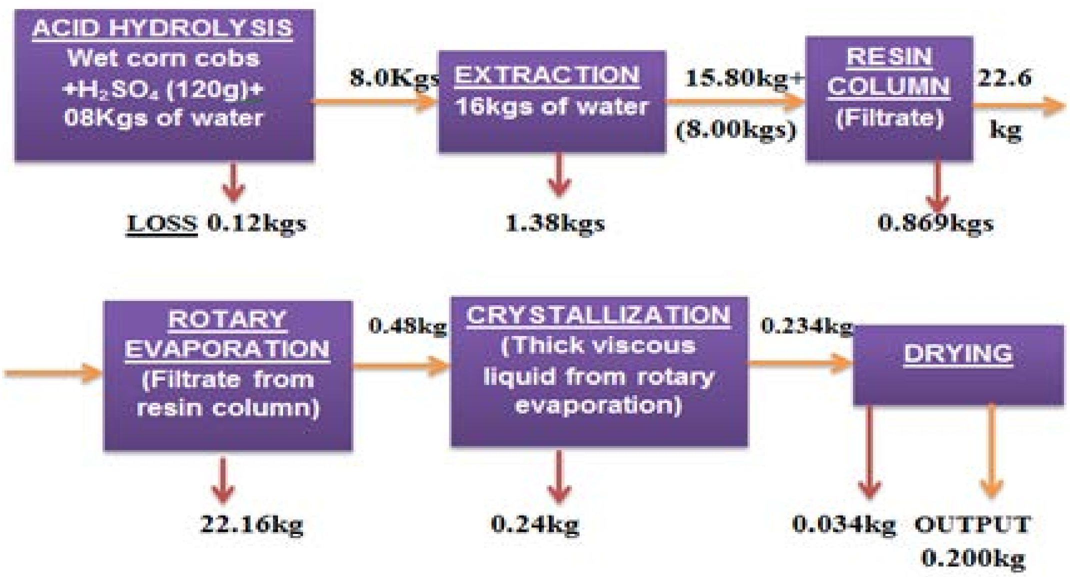
Fig.8 Dry powder of xylose

V. RESULTS AND DISCUSSIONS

Corn cob can be hydrolyzed to their sugar constituents using dilute acid under mild conditions that is necessary for cellulose hydrolysis. To selectively obtain xylose from corn cob for xylitol production, it is important to establish rapid hydrolysis conditions that conversion of xylose-rich substrates that do not require further treatment. Therefore, in this study, the effect of various temperatures as shown in Table.3 on the acid hydrolysis of the hemicellulose fraction of corn cob were evaluated. xylose production in Corn cob hemicellulosic medium and synthetic medium in shake flasks using D-xylose as a major carbon source.

TABLE 2.Washing of 2 Kgs Corn Cobs at 120°C

Washing	Input	Quantity (Kgs)	Output	Quantity (Kgs)	Loss (Kgs)
1.	Corn cobs+water	2kgs+10 kgs	Water	1.8	10.2
2.	2nd Extract Water	08	Filtrate	2.8	5.2
3.	3rd Extract: Water	08	Filtrate	4.9	3.1
4.	4th Extract: Water	08	Filtrate	5.91	2.09



$$\% \text{ Conversion} = \frac{\text{Amount In Feed Stream} - \text{Amount In Product Stream}}{\text{Amount In Feed Stream}} \times 100$$

Amount of corn cobs in feed stream = 2000 g

Amount of product obtained = 200 g

$$\% \text{ Conversion} = \frac{2000-200}{2000} \times 100 = 90.0\ \%$$

TABLE 3. Conversion of Xylose at Different Temperatures

Experiment Number	Weight Of Corn Cobs (Kgs)	Conversion % of Xylose at Different Temperatures		
		100 °C	110 °C	120 °C
1.	2	76	82	90
2.	2	78	84	91.1
3.	2	77.9	83.6	90.8

VI. CONCLUSION

This study demonstrated that xylose yield of 90% is possible with a very low generation of byproducts and xylitol can be produced effectively in corn cob hydrolysate (hemicellulose) instead of a complex chemically defined medium.

Xylose production from corn cob has some advantages such as the reduction in the cost of the medium, cheaper and easily available raw material and the ease of purification in the culture broth. The results of this present study are of considerable importance to the commercial manufacture of xylose using agricultural resources.

ACKNOWLEDGMENT

The authors thank Mr. K.Ravindranath, Chief Scientist and Director IICT for their permission to carry out the work at IICT, Tarnaka, and Hyderabad.

REFERENCES

[1] N. J. Cao , M. S. Krishnan,J. X. Du,C. S. Gong,"Ethanol Production From Corn Cob Pretreated By The Ammonia Steeping Process Using Genetically Engineered Yeast", Biotechnology Letters,Volume.18, No.9,Pg.1013-1018, September 1996.

[2] Attilio Converti,Patrizia Perego,Jose Manuel Domíngue," Xylitol production from hardwood hemicellulose hydrolysates by Pachysolen tannophilus, Debaryomyces hansenii, and Candida guilliermondii", Applied Biochemistry and Biotechnology,Volume 82, Issue 2, Pg.141–151, November 1999.

[3] Jose M. Dominguez,Cheng S. Gong, And George T.Tsao, "Pre-treatment of Sugar Cane Bagasse Hernicellulose Hydrolysate for Xylitol Production by Yeast", Applied Biochemistry And Biotechnology, Volume.57/58, Pg.49-50,1996.

[4] Name of Au Archana Sharma , "Production of xylitol by catalytic hydrogenation of xylose", The Pharma Innovation – Journal,Volume. 2, No.12,Pg.1-6,Feb 2014.

[5] A.Herrera,J.A.Ramírez, M.Vazquez,"Production of Xylose from Sorghum Straw Using Hydrochloric Acid" ,Journal of Cereal Science,Volume 37, Issue 3, Pages 267-274, May 2003.

[6] R. B. L. Mathur by Hand Book of Cane Sugar Technology.

[7] Kirk & Othmer by Encyclopedia of Chemical Technology.

[8] Perry's Chemical Engineering Hand Book.

[9] Warren. L. Mc Cabe by Unit Operations of Chemical Engineering.

[10] Octave Levenspiel, Chemical Reaction Engineering Third Edition.