RESEARCH ARTICLE OPEN ACCESS

Production of Xylose from Corncobs

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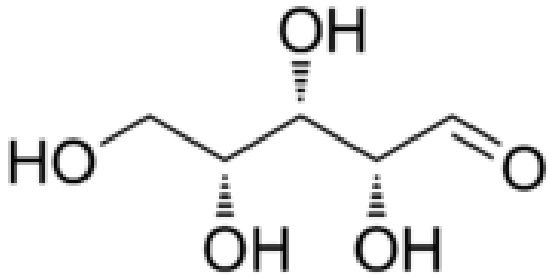
I. INTRODUCTION

A. *Xylose:*

Xylose (*xylon*, "wood") 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.

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

Structure:



The acyclic form of xylose has chemical formula $HOCH_2(CH(OH))_3CHO$. The cyclic hemiacetal isomers are more prevalent in solution and are of two types: the pyranoses, which feature six-membered C_5O rings, and the furanoses, which feature five-membered C_4O rings (with a pendant CH_2OH group). Each of these rings subject to further isomerism, depending on the relative orientation of the anomerichydroxy group.

Crystalline Xylose is composed of white crystals which are odorless and slightly hygroscopic.

Figure-Crystalline xylose

Physical Properties of Xylose:

Specific Gravity: 1.525
Other Name : Wood Sugar
Density : 1.525g/cm^3 (at 20°C)
Colour : White
Melting Point : 153-158^{o}C
Solubility : Water -125% w/w

Occurrence

Xylose is the main building block for the hemicellulose xylan, which comprises about 30% of some plants (birch for example), far less in others (spruce and

pine have about 9% xylan) it is the first saccharide in biosynthetic pathways of most anionic polysaccharides such as heparan sulfate and chondroitin sulfate

Hydrolysis of Xylan:

$C_5H_8O_4 + H_2O \xrightarrow[120°C]{\triangle} C_5H_{10}O_5$

Xylan Water H_2SO_4 Xylose

Uses of Xylose:

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 used as a non-nutritive sweetener in pharmaceutical industry.

II. EQUIPMENTS

Xylose:

A. Autoclave

- High torque zero leakage magnetic drive coupling.
- Maximum working pilot plant upto 210kg/cm^2 & temperature upto 673K.
- It is complete pilot plant with gas charging liquid closing, vaccum plant, Auto cooling system chiller, condenser for distillation or reflux, thermic fluid heating system etc.
- Size of the vessel used is 2lit capacity.
- The vessel which is used is made up of SS-316. Hastelloy B/C., M.O.C-S.S-316, Monel, Nickel, Titanium, Alloy 20 and Teflon coated SS-316.
- The autoclave system is continuously monitors, control and record various parameters.
- Fixed head reactor with bomb raising and lowering arrangements.
- GMP(Good Manufacture Practice) model panel & trolley of SS.

Applications:

It is used for reactions like Alklation, Amination, Carboxylation, Catalytic reduction, Chlorination, Dehyrogenation, Esterification, Etoxylation halogenations, Hyrgenation, Methylation, Nitration, Oxidation, Polymerisation, Sulphonation etc., at high pressure and temperature.

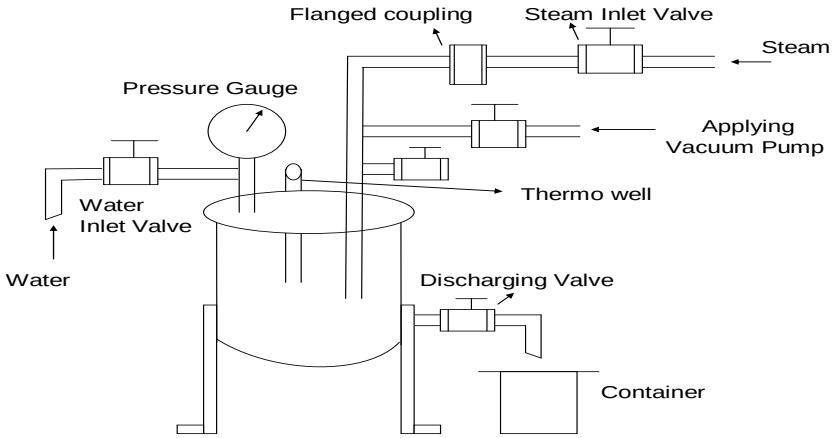
- To invent chemicals.
- To produce chemicals in small quantity.
- For pilot plant purpose.
- For quality control and process improvements.
- To study reaction parameters.
- For heterogenous mixing.

B. Vacuum pump:

A compressor that takes suction at a pressure below atmospheric and discharges against atmospheric pressure, it is called a vacuum pump.

As the absolute pressure at the suction decreases, the volumetric efficiency drops and approaches at the lowest absolute pressure attainable at the pump. It consists of a diaphragm pump in which reciprocating member is a flexible diaphragm, which is made of metal, plastic, or rubber. It can develop a discharge pressure of $3.0m^3/hr$.

Reactor:



The Diameter of the reactor = 31cm = 1.2 ft
The Height of the reactor = 68. 58cm = 2.3 ft

C. Resin Column

Description:

Resin is a macro-porous weakly basic anion resin having a tertiary amine functionality attached to polymeric styrene Di-vinyl benzene matrix, Resin is supplied in free base form ready for immediate use. The combination of macro-porous structure and tertiary amine functionality results in fast reaction rates, high regeneration efficiency, low rinse requirements and excellent chemical and physical stability. Resin can be effectively regenerated with sodium hydroxide, water and hydrogen peroxide, Resin can be used to treat water/process stream to remove free mineral acidity (Chlorides, Sulphate and nitrates). Resin particle size range from 0.3 to 1.2mm.

Packing: Resin are supplied in 50 lts, plastic bags. It can be supplied in 180 lt mild steel drums with plastic liner. The resin is always supplied in the free base form.

Purpose of Resin: Here, Resin is used for removal of acid content, color from the xylose reaction mixture and after passing through the resin, the xylose reaction mixture is neutral, color less and resin absorbs other sugars except xylose.

The resin is to be regenerated, for purpose of regeneration, taken 225gms of sodium hydroxide (NaOH) pellets and diluted in 7 lts of water (manjeera) thoroughly. Then after the dilution, the NaOH solution was passed through the 12 lts of resin in a resin column, air compressor (fluidization) also given to the resin in a resin column and kept for 30 min because for the proper mixing of the resin with NaOH solution in the presence of the fluidization. Then it is discharged slowly into a container. The purpose of using NaOH solution is to remove the colour, from the resin which is absorbed during the Xylose reaction mixture passed through the resin. To it, several washing with water is given to remove alkali and finally it is regenerated with H_2O_2. Take approximately 100ml H_20_2 which is diluted in 6lts of water and it is passed through the 12 lts of resin in a resin column, after discharging the H_20_2 solution from the resin column. Again several washings is done with water, until we get the resin to be neutral in nature. The neutral nature which is obtained, after regeneration of resin can be check by using P^H paper ranges (2 – 11).

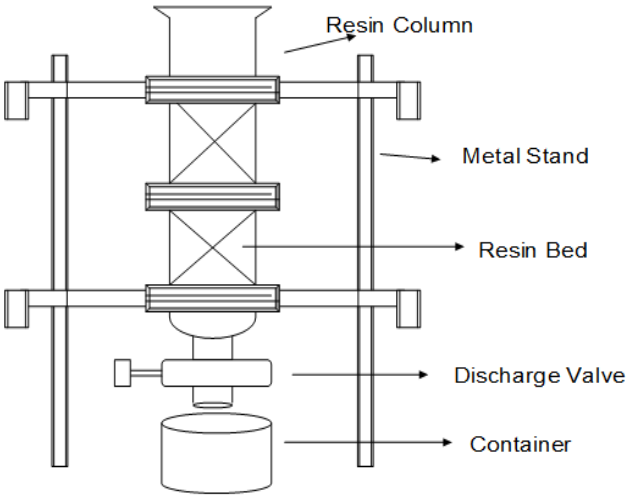


Figure: Resin column

D. Rotary Evaporation:

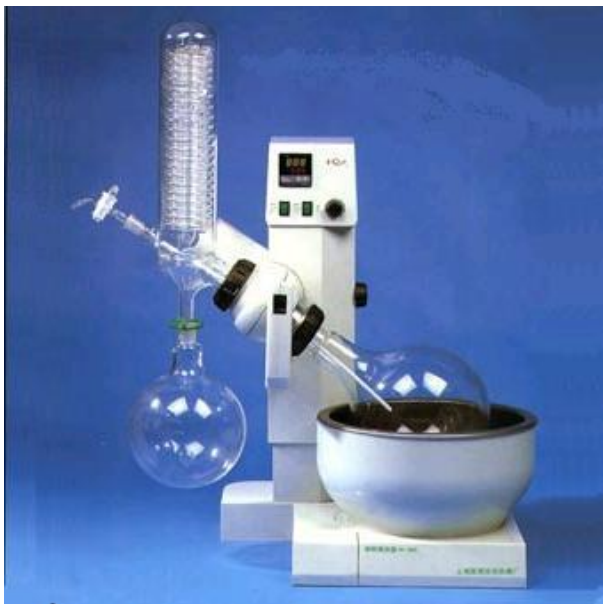


Figure- rotary evaporator

Rota vapor is used for the removal of Solvent (water, methanol) from the solute by vaporization. It can also used for other purposes like Recrystallisation, synthesis and cleaning of fine chemicals and Soxhlet extractions.

Principle:

With a vacuum rotary evaporator, single step distillations are performed and in a product friendly manner. The basis of this procedure is the evaporation and condensation of solvents using a rotating evaporating flask.

Distillation can be performed under vacuum. This increases performance and helps to protect products. Distillations can be performed under vacuum and atmospheric pressure.

Operation:

- **Evaporation area:**

The solvent is heated over a heating bath. In the rotating evaporating flask, a thin solvent film forms on the inside of the flask, resulting in increased evaporation rate. Rotation also results in even mixing of the material and thus, prevents stationary over heating in the flask.

- **Rotation drive:**

The drive unit makes sure that the evaporating flask rotates evenly with the resulting advantages.

- **Cooling area:**

The solvent vapor flows very quickly in the condenser. Here, the energy in the solvent vapor is transferred to cooling medium (mostly water) and the solvent condenses.

- **Receiving flask:**

The receiving flask receives the condensing solvent.

- **Vacuum:**

Vacuum reduces the boiling temperature and thus increases performance of distillation. Evaporating performance is influenced by distillation, pressure (vacuum), heating bath temperature, rotation speed and size of evaporating flask.

E. Cryostat (Julabo):

The julabo-refrigerated circulators employ a circulator head and a cooling machine with bath tank, and have been designed for heating and cooling of liquids in a bath tank. Besides the cooling aggregate, the main functional elements are the heater, circulation pump and control electronics. The medium used is water and the temperature range is 5^0C-80^0C and has a capacity of 5litres of water.

In the experiments, the Cryostat (julabo) is used to circulate chilled water in the condensers of rotavapor, Soxhlet extractor and stirred vessel. The purpose of the Cryostat (julabo)is to supply the chilled water to the condenser, to condense the vapours of the solvent.

III. MATERIALS & METHODOLOGY

A. Materials Used For Xylose:

- ***Corncobs:***

A **corn cob**, is the central core of an ear of maize (*Zea mays* ssp. *mays*). 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

Figure : Corn cobs

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.

Corncobs contain: 39.1% cellulose,42.1% hemicellulose,9.1% lignin,1.7% protein and 1.2% ash .

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.

Uses

Corncobs find use in the following applications:

- Industrial source of the chemical furfural
- Fiber 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
- Raw material for bowls of corncob pipes
- As a bio-fuel
- Charcoal production

- **Sulphuric Acid:**

Sulfuric acid is a highly corrosive strong mineral acid with the molecular formula H_2SO_4 and molecular weight 98.079 g/mol. It is a pungent-ethereal, colorless to slightly yellow viscous liquid that is soluble in water at all concentrations.

- **Resin:**

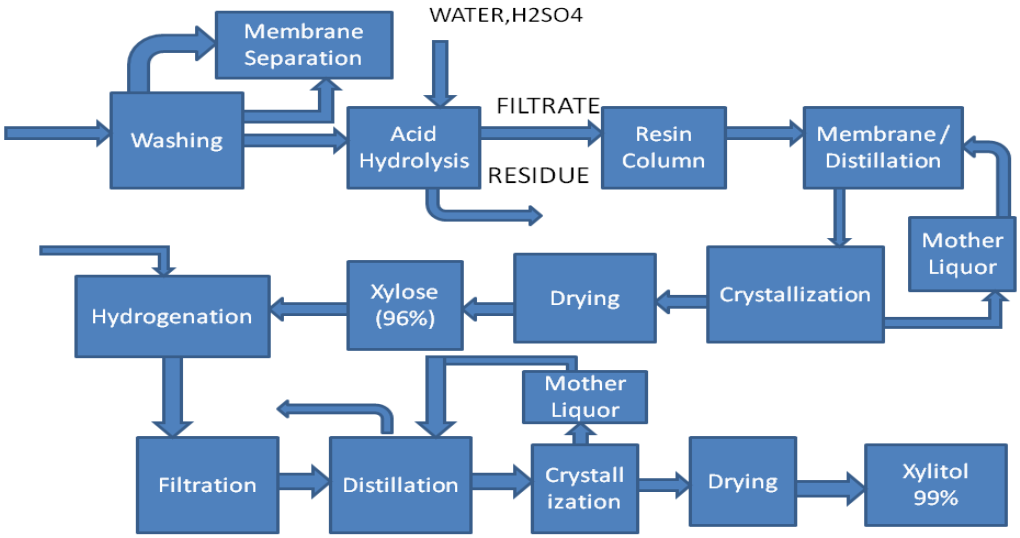
Resin the removes the color when a fluid is passed through it.it acts as a good adsorber.

- **Methanol:**

Methanol, also known as methyl alcohol, wood alcohol, wood naphtha, methyl hydrate, or wood spirits, is a chemical with the formula CH_3OH.
Boiling point: 64.7 °C
Density: 792 kg/m³
Formula: CH_3OH
Molar mass: 32.04 g/mol
Melting point: -97.6 °C
Vapor pressure: 13.02 kPa

Procedure:

BLOCK DIAGRAM FOR A PREPARATION OF XYLITOL



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)

1. Washing:

2kgs of the corn cobs (dry) was fed into the reactor and to it 8lts of water (manjeera) was added. Then the heat is supplied to the reactor by means of the steam and maintained at a pressure of 1 - 1.2kg/cm^2. After obtaining the temperature of 120^{o}C, it was maintained for ½ hr i.e., 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.

2. Acid hydrolysis:

To the above wet corn cobs, 120 gms of concentrated sulphuric acid (98.9%) was diluted in 8lts of water (manjeera), 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 ½ hr 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.

3. Extraction:

To the above reacted corn cobs, 8lts 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. 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.

5. De-Colourization & Neutralization:

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

6. Rota evaporation:

The filtrate neutralized from resin column is taken as a feed to the Rota Vapor. 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.

7. 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.

8. 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 bottom and filtered xylose at the top.

9. Drying (finished product):

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

Figure-Powder xylose

IV. ANALYSIS

Material Balance for Xylose:

Experiment:

Material input	Quantity (kgs)	Material output	Quantity (kgs)
Washing: Wet corn cobs + Water	16.8	Crude Xylose Extract	16
Wet corn cobs+ Water	15.6	Crude Xylose Extract	15.2
Acid Hydrolysis: Water + H_2S0_4ACID+ Corn cobs	10kgs + 0.18kgs+ 6.41kgs=16.59	Reaction Mixture	16

V. RESULTS

Washing:
Wash-1:
Input:
Weight of dry corn cobs = 7.3kgs
Amount of water used for washing = 40kgs
Output:
Weight of filtrate = 35.5kgs
LOSS = INPUT-OUTPUT
= 40-35.5 → 4.5kgs

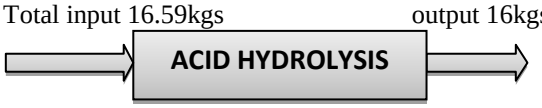
Wash-2:
Input:
Amount of water used for washing = 35.5kgs
Output:
Weight of filtrate = 30kgs
LOSS = INPUT-OUTPUT
=35.5-30 → 5.5kgs

Wash-3:
Input:
Amount of water used for washing = 30kgs
Output:
Weight of filtrate = 20kgs

LOSS = INPUT-OUTPUT
=30-20 → 10kgs

Wash-4:
Input:
Amount of water used for washing =20kgs
Output:
Weight of filtrate = 13.5kgs
LOSS = INPUT-OUTPUT
=20-13.5 → 6.5kgs
Weight of wet corn cobs=15.6kg
Weight of dry corncobs per kg=2.13kg

Acid hydrolysis:
Total input 16.59kgs output 16kgs



Weight of corn cobs = 6.41kg
Weight of water = 10kgs
Weight of acid = 0.18kg
Others = 0.59kg

VI. CONCLUSION

During the process development for the preparation of xylose maximum amount of yield was achieved.

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