Value added products from agricultural residue of sweet sorghum bagasse

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Abstract

Lignocellulosic biomasses such as sugarcane bagasse, corn cobs, wheat straw, rice straw etc. are abundantly available, cost effective and economical sources of polysaccharides and renewable energy. There is a search for an alternative and innovative renewable chemical to deplete the fossil resources which is moving forward in the current trend. The main objective of the present study is to synthesize the hemicellulose sugar in monomeric form without degradation of cellulose and lignin from agricultural residue of sweet sorghum bagasse. The effects of the operating temperature and time along with acid concentration were investigated.

The results obtained using 6% sulphuric acid concentration at 120 ^{o}C temperature for 3 h reaction time were calculated. Hydrolysis of lignocellulosic biomass under acidic condition is the most commonly used method of acid hydrolysis, which gets influenced by various process parameters. It can be used in the pharmaceutical and food industries for the preparation of sweeteners for diabetic patients. The obtained xylose can be used as a bio-product as a nutrient.

Keywords: Bagasse, Lignocellulosic Biomass, Hemicellulose, Hydrolysis, Xylose.

Introduction

Biomass is quite simply an organic material that can be derived from plants and animals. Usually, the biomass composition consists of carbon, oxygen, hydrogen, nitrogen and other constituents including alkali and alkaline earth and heavy metals. It is a fossil renewable energy source, which can be produced from landfill gases, agricultural, municipal and wood processing waste. Biomass can be extensively derived from biological organisms or eco-system which can be referred to as lignocellulosic biomass.

The major energy sources for biomass are obtained either by direct combustion or indirect form of biofuel[1]. Biomass was used as a fuel, since hundred years ago[2]. The organic matter of biomass can be stored for a long time as a renewable energy which is similar to the photosynthesis in plants. Sweet sorghum *(Sorghum bicolor)* is the second most abundant millet crop next to rice crop this can be used for sugar, alcohol, syrup, fodder, fuel, bedding, roofing, fencing, paper and chewing. The composition of sweet sorghum bagasse is provided in table 1.

Table 1
Composition of sweet sorghum bagasse

Cellulose	**44.6%**
Hemi- cellulose	27.1%
Lignin	20.7%
Fiber	13%
Ash	0.4%

The raw or utilized sweet sorghum bagasse can be used as a biomass which consists of lignocellulosic material. This lignocellulose biomass is containing 15-30% lignin, 30-45% cellulose and 25–45% hemicellulose constituents[3]. The internal structure and composition of lignocellulosic biomass are provided in fig. 1(a and b). These three constituents arranged in a hetero-matrix form depend on temperature, relative composition, type of species and source of biomass[4]. The other constituent xylan is the second most abundant backbone constituent in sweet sorghum bagasse after cellulose. Xylan is the enzyme, which can undergo hydrolysis by random cleavage of β-1, 4 into xylo-oligosaccharides followed by degradation into xylose monomeric constituent[5,6]. The structure of xylose is provided in fig. 2.

Xylose has 40% low calories of sucrose and less than 75% carbohydrates which can be used as a sweetener for diabetic patients. The taste and physical appearance of the xylose are exactly like sugar. The physical properties of xylose are provided in table 2. Several researchers have worked on the extraction of xylose and hemicellulose from various biomasses. Using high pressure carbon dioxide as a catalyst, hemicellulose and xylose were extracted from wheat straw[7], the dilute sulphuric acid as a catalyst for hydrolysis of sweet sorghum bagasse for the production of xylose[8].

Xylose, arabinose, glucose and other products were synthesized from sugarcane bagasse of hemicellulose hydrolysis by HCl and H_2SO_4[9]. Production of xylose and hemicellulose sugar from sorghum straw was through HCl and H_2SO_4 hydrolysis respectively[10,11].

Inorganic salts were used for pyrolysis of sweet sorghum bagasse and it was observed that the maximum degradation rate decreased for hemicelluloses and cellulose fraction[12]. Recovery of xylose and glucose (total sugar) from sweet sorghum bagasse it by using liquid hot water process by step change flow rate[13]. Studies on pretreatment of sulphuric and hydrochloric acids hydrolysis of sweet sorghum bagasse and analysis of C5 and C6 sugars are carried through surface response methodology[14]. From the literature survey, the

present study deals with the sulphuric acid hydrolysis of hemicellulose from sweet sorghum bagasse for the production of xylose. Initially, the sweet sorghum bagasse was milled for size reduction, then hydrolyzed with sulphuric acid for xylose production. Experiments were conducted with various parameters and analyzed for the xylose retention time using HPLC.

Material and Methods

Materials: Sweet sorghum bagasses were obtained from ICRISAT, Hyderabad, India. Sulphuric acid was purchased from SD Fine Chemicals, Hyderabad, India.

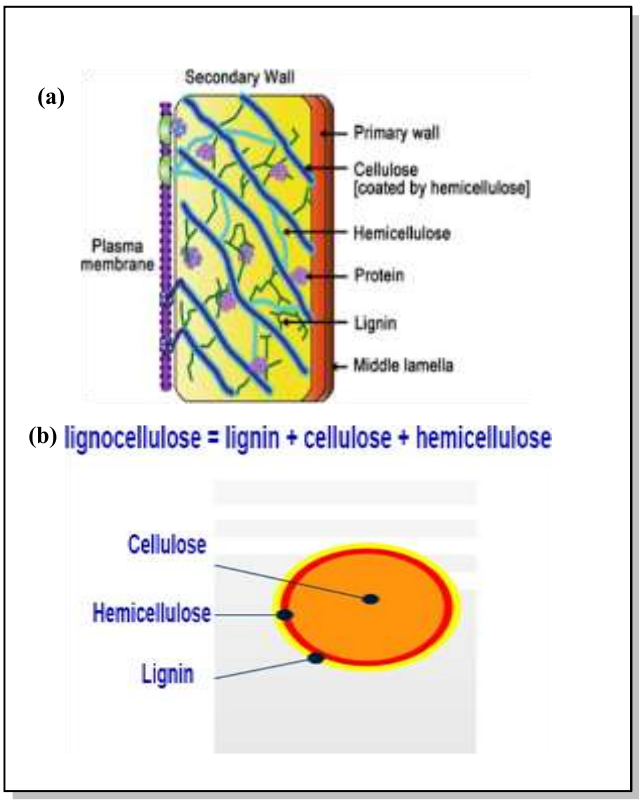

Fig. 1: (a) Internal structure and (b) composition of lignocellulosic biomass (Source; internet)

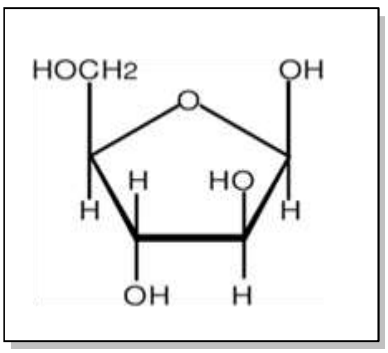

Fig. 2: Cyclic structure of *D*-xylose

Table 2
Physical properties of xylose

Specific Gravity	1.525
Other Name	Wood Sugar
Density	1.525 g/cm^3 (at 20°C)
Colour	White
Melting Point	153-158^{0}C
Solubility	125% w/w in water

The glassware materials such as glass-lined reactor, burette, pipette, conical flask, beakers and thermometers were from BOROSIL, Hyderabad, India. The equipment used for the sample analysis was weighing balance for sample weight, milling equipment for size reduction from Metis Jindal Industries, Punjab, India and Okuma, Japan. High pressure liquid chromatography (HPLC) analytical instrument was procured from Shimadzu, Japan.

The distilled water utilized for the reaction process was generated from the laboratory itself using Reverse Osmosis (RO).

Description of milling Equipment: Biomass particle size reduction is an important operation in many industries. The important reasons for the size reduction of biomass are ease to handle, increase in surface area per unit volume, separation of components etc. The schematic representation of laboratory milling equipment (a) outer view and (b) inner view is shown in fig. 3.

The operation of the milling equipment is energy-intensive for a specific application. In the milling process, the sweet sorghum biomass was initially compressed to undergo high impact and attrition followed by cutting with a blade into the desired size. The collected biomass size was found to be 6 mm at 150-200 rpm. Fig. 4 (a and b) illustrates the sweet sorghum biomass before and after size reduction.

Methods of producing xylose: The method adopted for the production of xylose from easily and abundantly available biomass of sweet sorghum is renewable and has potential for extraction of many useful products. During experimentation, steam was used for giving energy inputs during the process. Acid was used at a 6% concentration to convert the hydrolyzed biomass into the value added product of xylose.

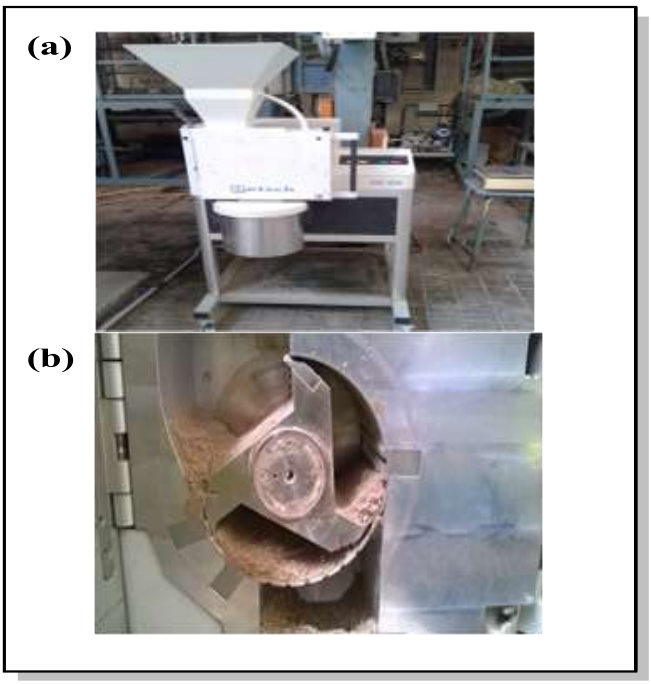

Fig. 3: Photography of milling equipment (a) outer and (b) inner view

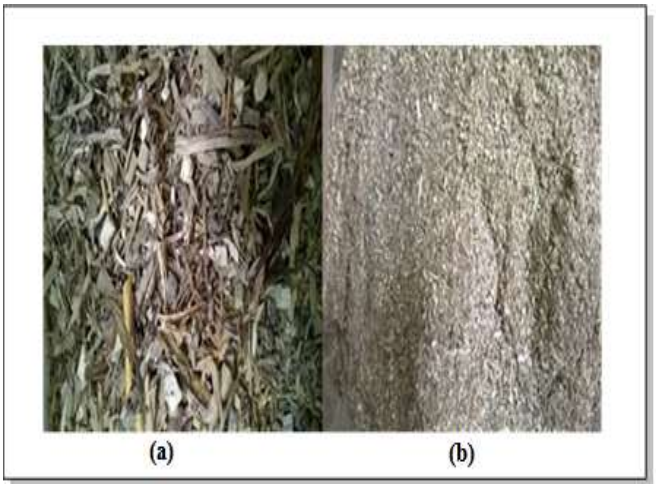

Fig. 4: The sweet sorghum biomass (a) before and (b) after size-reduction

Hydrolysis Methods: The term hydrolysis is applied to reactions in both organic and inorganic chemistry, where in water causes double decomposition and hydrogen getting transferred to one component with hydroxyl to the other.

$$XY + H_2O \longrightarrow HY + XOH$$

$$C_5H_8O_4 + H_2O \xrightarrow[H_2SO_4]{\triangle,\ 120^0C} C_5H_{10}O_5$$

Xylan Water Xylose

The method of hydrolysis can be conducted in either concentrated or dilute acid hydrolysis for the production of xylose.

In the present study, the hydrolysis of biomass was carried out using dilute sulphuric acid for the production of xylose from sweet sorghum bagasse. In this method, the xylose is produced from lignocellulose biomass using dilute acid at an elevated temperature (120^0C). The advantage of the process is that the production of pentose stream can be separated physically from particulate residue. Using of this process is to reduce the capital and operative cost due to the reduction in corrosion and worker hazardous.

Results and Discussion

Experiments were carried out with available sweet sorghum at pilot plant level in 50 L capacity reactor using steam as energy input to break the bonding of biomass by hydrolysis and further acid hydrolysis to convert xylan available in biomass to xylose. The results obtained are encouraging for the production of value added products from a renewable source. In experiment 1, initially 9.3 kg of biomass was washed with 4 kg of water as input. After washing, the weight of biomass was reduced to 7.8 kg as output, which is further washed with water in a second wash. After the second wash, the weight of the biomass again reduced from 7.8 kg to 4.9 kg. After water washing (2^{nd} wash), the biomass was treated with dilute sulphuric acid to produce xylose as a product.

The similar procedure was followed in other experiments such as experiments 2, 3 and 4 by varying the weight of the biomass. After each experiment, the pre-washed biomass was treated with acid hydrolysis. From the all experimental observations (1, 2, 3 and 4), the xylose production was high for experiment 4. The overall experiments were carried out for 3 hours.

The result for each experiment is provided as follows:

Material Balance
Experiment 1

1^{st} Wash of the Biomass:
INPUT:
Weight of wet bagasse (Biomass) = 9.3 kg
Amount of water utilized for 1^{st} washing = 4.0 kg
TOTAL =13.3 kg
OUTPUT:
Weight of bagasse after washing=7.80 Kg
Amount of filtrate = 4.67 kg
TOTAL = 12.47 kg
LOSS = Input - Output
= 13.3 - 12.47 = 0. 83kg

2^{nd} Wash for the Biomass:
INPUT:
Weight of wet bagasse (Biomass) = 7.8kg
Amount of water utilized for 2^{nd} washing = 4.0kg
TOTAL = 11.8kg
OUTPUT:
Weight of bagasse after washing = 4.91 kg
Amount of filtrate = 6.49 kg
TOTAL = 11.4 kg
LOSS = Input - Output
= 11.8 - 11.4 = 0.4 kg

Acid Hydrolysis:
After water washing of the biomass, acid hydrolysis was carried out for further processing.
Wet biomass was taken for acid hydrolysis with 6% acid on basis of dry biomass.
Total input = 12.12 kg
Total output = 11.5 kg
LOSS = Input – Output
= 12.12 - 11.15
= 0.62 kg

Experiment 2

1^{st} Wash for the Bagasse (Biomass)
INPUT
Weight of wet bagasse + Amount of water utilized for 2^{nd} washing = 14.02 kg
OUTPUT
Weight of bagasse after washing + Amount of filtrate= 13.67 kg
LOSS = Input - Output
= 14.02 - 13.67 = 0.35 kg

2^{nd} Wash for the Bagasse (Biomass)
INPUT
Weight of wet bagasse + Amount of water utilized for washing =12.6 kg
OUTPUT
Weight of bagasse after washing +Amount of filtrate = 11.45 kg
LOSS = Input - Output
= 12.6 – 11.45 = 1.15 kg
Acid Hydrolysis
Total input =13.13 kg
Total output=12.37 kg
LOSS = Input – Output
= 13.12 - 12.37= 0.75 kg
Experiment 3

1st Wash for the Biomass:
INPUT:
Weight of wet bagasse + Amount of water utilized for washing = 15.05 kg
OUTPUT:
Weight of bagasse after washing + Amount of filtrate = 14.33 kg
LOSS = Input - Output
= 15.05 -14.33 = 0.72 kg

2nd Wash for the Bagasse (Biomass):
INPUT:
Weight of wet bagasse + Amount of water utilized for washing = 12.01 kg
OUTPUT:
Weight of bagasse after washing + Amount of filtrate = 11.90 kg
LOSS = Input - Output
= 12.01 - 11.9 = 0.11 kg
Acid Hydrolysis:
Total input = 10.93 kg
Total output = 9.17 kg
LOSS = Input – Output
= 10.93 - 9.17 = 1.76 kg
Experiment 4:

1st Wash for the Biomass:
INPUT:
Weight of wet bagasse + Amount of water utilized for washing = 16.1 kg
OUTPUT:
Weight of bagasse after washing + Amount of filtrate= 14.88 kg
LOSS = Input - Output
=16.1 - 14.88 = 1.22 kg

2nd Wash for the Bagasse (Biomass):
INPUT:
Weight of wet bagasse + Amount of water utilized for washing = 13.55kg
OUTPUT:
Weight of bagasse after washing + Amount of filtrate = 11.6kg
LOSS = Input – Output
= 16.6 - 15.27 = 1.33 kg
Acid Hydrolysis
Total input = 10.80 kg
Total output = 8.02 kg
LOSS =INPUT – OUTPUT
=10.8 - 9.30 = 1.5 kg

High Performance Liquid Chromatography (HPLC) is one of the column chromatographies where the sample (mobile phase-HPLC grade water) pumps at high pressure through a stationary phase in the column. In this method the mixture was separated in qualitative and quantitative manner based on retention time. By obtaining a pure compound assigned peak as a chromatogram, the resultant xylose obtained after experiment 4 of acid hydrolysis was analyzed by HPLC and the chromatograms of standard xylose and the reaction mixture after experimentation are provided in fig. 5. and fig. 6.

Conclusion

Sweet sorghum is abundantly available and a renewable biomass and therefore selected for the experimentation. Many hydrolysis experiments were conducted on a pilot scale and the reaction time was optimized to 3 h, whereas acid concentration was optimized to 6% in the presence of steam which is an economically viable energy input.

Xylan of hemicellulose from sweet sorghum biomass was converted into xylose by acid hydrolysis according to the peaks obtained from the HPLC chromatograms where increasing reaction time resulted in a reduction in the quantity of xylose produced. Hence the reaction time was fixed to 3 h, as the maximum xylose could be produced up to 3 h beyond which product yield reduced.

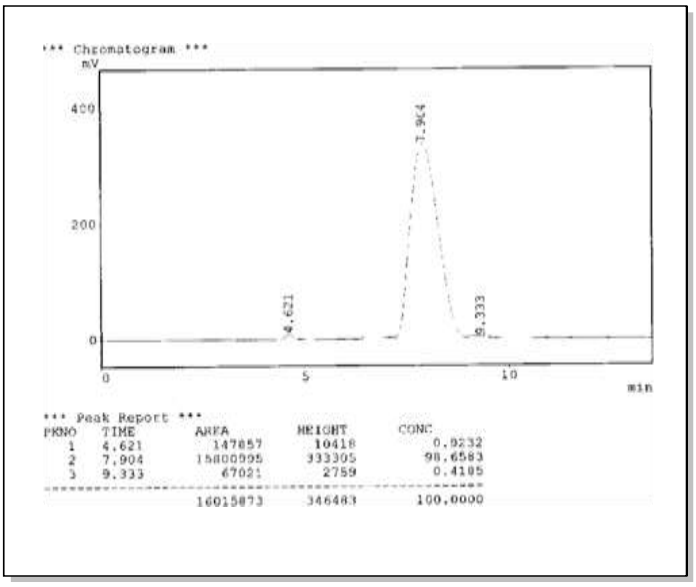

Fig. 5: HPLC chromatogram of xylose

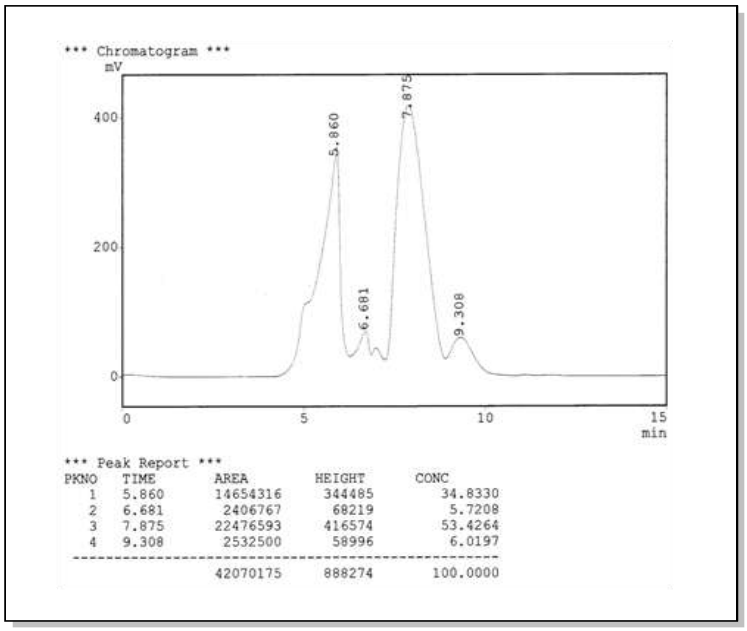

Fig. 6: HPLC chromatogram of the reaction mixture after experimentation

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