Conversion of Cotton to Glucose by Base Hydrolysis Using Various Hydrolytic Conditions

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ABSTRACT

Cotton is a natural polymer which contains cellulose. It can be hydrolyzed by different methods to convert it to useful products. The process of hydrolysis may greatly affected by different factors i.e. concentration of solvent, heating source, and the time duration. In the present work, cotton was hydrolyzed by base under different heating sources for hydrolysis including hotplate, sonication and autoclave. The hydrolysis process was done for 60 min using 3M and 6M NaOH solutions. The hydrolysis process resulted in 21 to 39 % conversion of cotton to glucose. The obtained glucose may be used for different purposes such as bioethanol production, bacterial cellulose production and other purposes while the residue may be utilized for adsorption.

Key words: Cotton, base hydrolysis, glucose

Introduction

Cotton is a soft, fluffy staple fiber that grows in a protective capsule, around the seeds of cotton plants of the genus Gossypium. The fiber of cotton is pure cellulose. The use of cotton for fabric is known to date to prehistoric times. Cotton is used to make a number of textile products. In addition to the textile industry, cotton is used in fishing nets, coffee filters, tents, explosives manufacture (nitrocellulose), cotton paper, and in book binding. The first Chinese paper was made of cotton fiber.

Waste Cotton products have no value; hence to make it useful recycling or conversion of cotton to useful products is necessary. For this purpose hydrolysis of cotton is done. Hydrolysis usually means the cleavage of chemical bonds by the addition of water. Generally, hydrolysis is a step in the degradation of a substance. After hydrolysis cotton is converted into Glucose and cotton residue.

Different researchers have reported different methods for cotton hydrolysis. Yang *et al* [1] reported the systemic study on thermal dilute-acid hydrolysis of cotton straw. The process has been established in two-step reaction, which can greatly improve the utilization efficiency of lignocellulosic biomass.

Haykir *et al* [2] subjected cotton stalk to ionic liquid pretreatment via several ionic liquids. Ionic liquids freely provided conversion of cotton stalk into fermentable sugars upon enzymatic hydrolysis.

Vani *et al* [3] attemted Alkali assisted microwave pretreatment (AAMP) of cotton plant residue (CPR) with high pressure reactor pretreatment was compared. The yield of sugar was 0.495 g/g.

Binod *et al* [4] evaluated cotton stalk as feedstock for bioethanol production. Different pretreatment strategy were tried using sodium hydroxide(NaOH) in a high pressure reactor equipped with a pitch blade turbine stirrer, followed by enzymatic hydrolysis using celluloses, the process optimization was carried out using Taguchi experimental design.

Chu *et al* [5] reported the kinetics of cotton cellulose hydrolysis using concentrated sulfuric acid (H_2SO_4) and the performance of fermentative hydrogen production from the hydro lysate in the batch system was carried out. Effects of sulfuric acid concentration, cotton cellulose concentration and operating temperature on the cotton cellulose hydrolysis were investigated.

Samaniuk *et al* [6] examined enzymatic hydrolysis of lingo cellulosic biomass in a high shear environment. The conversion of cellulose to glucose in sample mixed in a torque remoter producing shear flows similar to those found in twin screw extruders was greater than that of unmixed sample. In addition, there is a synergistic effect of mixing and enzymatic hydrolysis; mixing increases the rates of cellulose conversion while the increased conversion facilitaties mixing. The synergy appears to results in part from reduction in particle size, which is more important when hydrolysis occurs during strong mixing.

Ha *et al* [7] reported for increasing cellulose accessibilities to the enzymatic attack, the pretreatment is a essential stage to change some structural characteristics of cellulosic materials. As a new method, microwave irradiation on cellulose dissolution pretreatment with ionic liquids (ILs) was investigated in this study. The rate of reaction in enzymatic hydrolysis of cotton cellulose was increased by at least 12-fold after IL dissolution pretreatment at 110 °C

and by 50-fold after IL dissolution pretreatment with microwave irradiation.

Fukuoka and Dhepe [8] studied the conversion of cellulose into sugar alcohol by using Pt or Ru catalysts supported on inorganic oxides in water under hydrogen pressure.

Satyamurthy *et al* [9] attempted the cellulolytic fungus Trichoderma reesei was used to prepare cellulose Nano whiskers (CNW) by controlled hydrolysis of microcrystalline cellulose (MCC). Lin *et al* [10] treated cotton cellulose with acid either in water or in ethanol, containing 1.39% HCl, at 45 and 65 °C for 1–5 h. The morphology and molecular weight distribution of celluloses before and after acid treatments were observed, and the differences in the structure of celluloses treated at different situations were compared. The soluble sugar contents of cellulose during acid treatment were lower than 6%.

Jeihanipour and Taherzada [11] investigated ethanol production from cotton linter and waste of blue jeans materials. Alkali pretreatment monitored by enzymatic hydrolysis resulted in almost whole conversion of the cotton and jeans to glucose, which was then fermented by Saccharomyces cerevisiae and converted to ethanol.

Nevel and Upton [12] attempted the partial hydrolysis of cotton cellulose by hydrogen chloride in benzene containing various proportions of water by measurements of variability, loss of weight, and copper number.

2.0 Materials and Methods

2.1. Hydrolysis of cotton using hotplate

Materials

The pure cotton was obtained from the pharmacy shop near to Shankar campus Mardan.

Apparatus

The apparatus which were used for the hydrolysis experiments are 250 ml flask and stirrer. The Hotplate of ROMMELSBACHAR (made in Germany) was used for heating and stirring purpose. Electrical balance of SHIMADZU was used for weighing of cotton.

Procedure

10 g cotton was taken in watch glass and weighted by electrical balance. Then the cotton was taken into 250 ml flask. 200 ml of 3M of NaOH solution was added into that flask. The flask was putted on hotplate and kept the Temp at 84˚C .The solution was heated for 60 min and stirred the solution by glass rod. After 1 hour heating, the flask was

removed from hotplate. Then the extract was separated from cotton residue. The extract was stored in bottle. Benedict test was performed for the quantitative determination of glucose. Solid residue was weighted by electrical balance. The same procedure was repeated for cotton hydrolysis with 6 M NaOH solution.

2.2. Hydrolysis of cotton using Sonicator

Materials

The pure cotton was obtained from the pharmacy shop near to Shankar campus Mardan.

Apparatus

The apparatus which were used for the hydrolysis experiments are 250 ml flask and stirrer. The Sonicator instrument of (model power sonic 405) made by HWASHIN Technology SEOUL KOREA was used for heating. Electrical balance of SHIMADZU was used for weighing of cotton.

Procedure

10 g of cotton was taken in watch glass and weighted by electrical balance. Then the cotton was taken into 250 ml flask. 200 ml of 3M of NaOH solution was putted into that flask. The flask was put in the ultrasonic bath and the Temp was kept at 50 ˚C. The net solution was sonicated for 1 h at 50 ˚C. After 1 hour heating the flask was removed from sonicator. After cooling the extract was separated from the cotton residue. The extract was stored in bottle. Benedict test was performed for the quantitative determination of glucose. Solid residue was weighted by electrical balance. The same procedure was repeated for cotton hydrolysis with 6 M NaOH solution.

2.3. Hydrolysis of cotton using Autoclave

Materials

The pure cotton was obtained from the pharmacy shop near to Shankar campus Mardan.

Apparatus

The apparatus which were used for the hydrolysis experiments are 250 ml flask and stirrer. Autoclave WagTech international model no 25X-2. Electrical balance of SHIMADZU was used for weighing of cotton.

Procedure

10 g of cotton was taken in watch glass and weighed by electrical balance. Then the cotton was taken into 250 ml flask. 200 ml of 3M NaOH solution was added into that flask. The flask was putted in Autoclave and

kept the Temp at 121 ˚C and 1.5 atm pressure. After 1 hour heating the flask was removed from Autoclave. After cooling the extract was separated from cotton residue. The extract was stored in bottle. Benedict test was performed for the quantitative determination of glucose. Solid residue was weighted by electrical balance. The same procedure was repeated for cotton hydrolysis with 6 M NaOH solution.

2.4. Determination of lambda max of the extracted solution after hydrolysis

Materials

Covets**,** UV 1100 spectrophotometer Robus technology S No; 5041106094, flasks etc.

Chemicals

The extracted solution from cotton via NaOH as sample, distilled water as reference

Procedure

The spectrometer was switched on about 20 min before the analysis. The sample and reference were taken in the covets. The spectrometer was first blanked with reference solution before every reading. The wavelength was started from 200 nm and up to 680 nm. The lambda max was determined.

3.5. Determination of glucose concentration by using Benedict solution through spectrophotometer

Materials

Test tubes, test tube holders, watman filter paper, funnel, UV 1100 spectrophotometer Robus technology S No; 5041106094 and Numerical show constant temperature water bathing boiler (NUOWAI).

Chemicals

Benedict solution, distilled water and 1% glucose solution.

Procedure

In this study 1% (w/v) glucose standard solution was prepared. From 1% glucose solution 1 ml, 2 ml, 3 ml, 4 ml and 5 ml samples were kept in test tubes respectively.1 ml of Benedict solution was added to each sample. In contrast to each sample 5 ml, 4 ml, 3 ml, 2 ml and 1 ml of Distilled water was also added to test tube as shown in Table 1. 5 ml of each sample was added into another three test tube and then mixed with one ml of benedict solution and one ml of distilled water as indicated in Table 2. These test tubes were fitted in test tube holder and kept in water bath for 20 min at 80 C. After 20 min heating test tube

were removed from water bath and allowed for cooling then filtered determine the absorbance of each of the samples using a spectrophotometer set to read at 735 nm. Distilled water was used to set the 100%. A graph of the standard solutions plotted the concentration of each unknown glucose solutions was determined.

Table 1. Preparation of standard sample for determination of glucose by benedict solution method.

Tube No.	**ml of glucose soln**	**ml of H_2O**	**ml of Benedict's soln**
1	1.0	5.0	1.0
2	2.0	4.0	1.0
3	3.0	3.0	1.0
4	4.0	2.0	1.0
5	5.0	1.0	1.0

Table 2. Preparation of samples taken after hydrolysis of 10g cotton treated with 3M and 6M NaOH solution for 60min on different hydrolysis methods for determination of glucose by Benedict test.

Tube No. (sample)	**ml of glucose soln**	**ml of H_2O**	**ml of Benedict's soln**
6 (Hot plate)	5.0	1.0	1.0
7 (Sonication)	5.0	1.0	1.0
8 (Autoclave)	5.0	1.0	1.0

3. Results and Discussion

3.1. Determination of Lambda max of extracted solution by hydrolysis

Cotton contains cellulose which was hydrolyzed by NaOH at different conditions. The extract of hydrolyzed cotton used for analysis. The lambda max of extract was determined i.e. 320 nm shown in Fig 1.

3.2. Hydrolysis of 10 g cotton by 3M NaOH solution for 60 min at different conditions

First the 3M NaOH solution was used for hydrolysis of cotton at different hydrolyses methods (Hotplate, sonication and autoclave) for 60 min to convert cotton to glucose. Hotplate, sonication, and autoclave were used as heating penetrating sources for hydrolysis. The extract which obtained by

the hydrolysis of 3M NaOH solution on Autoclave heated for 60 min had greater amount of glucose content as compared to Hotplate and sonication as shown in Table 5. The reason was that, the autoclave have high temperature 121 ˚C and high pressure 1.5 atm pressure due to which the solvent entered to the cotton mass and destroyed its structure as a result more extraction of glucose occurred. The amount of glucose obtained was estimated as 21.19%, 28.05% and 34.00% for hotplate, sonication and autoclave conditions respectively. The percentage of glucose content obtained after the dry weight of residue was less than that of actual i.e. 20.60%, 27.50 and 33.60 by Hotplate, sonication, autoclave respectively. This might be due the fact that, the cotton sample also contains some volatile component which was evaporated during heating. Due to which the residue weight is less than the expected shown in Table 6.

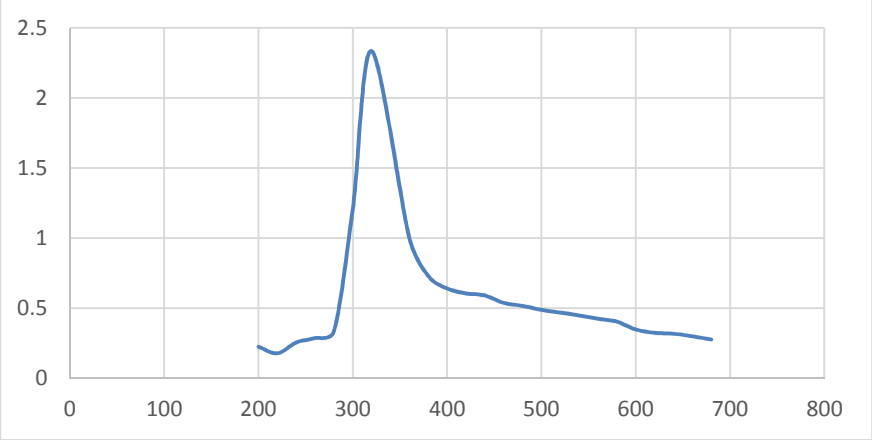

Fig 1. Lambda Max of cotton extract

Table 3. Absorbance of standard solution of glucose for making standard curve for Determination of glucose in hydrolysate obtained with 3M NaOH for 60 min.

Conc. (ml)	Absorbance
1	0.100
2	0.124
3	0.156
4	0.212
5	0.296

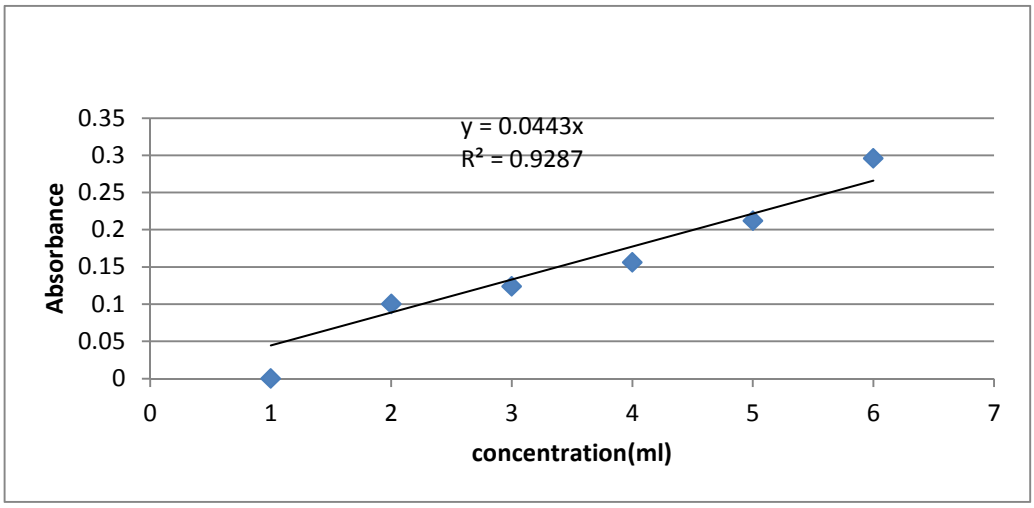

Fig 2. Standard curve for glucose determination in cotton extract treated with 3M NaOH solution for 60 min.

Table 4. Absorbance of samples of extract obtained after hydrolysis of cotton with 3M NaOH solution for 60 min.

S. No.	Condition/Source	Absorbance
1.	Hot plate	0.288
2.	Sonication	0.369
3.	Autoclave	0.440

Table 5. Glucose content in 10g of cotton hydrolyzed by 3M NaOH solution for 60 min.

Condition/Source	Initial volume of NaOH solution(ml)	Volume after hydrolysis(ml)	Temperature(C)	Glucose content (%)	
				w/v%	w/w%
Hotplate	200	163	84	1.30	21.19
Sonication	200	169	50	1.70	28.05
Autoclave	200	173	121	1.90	34.00

Table 6. Weight of cotton lost by hydrolysis with 3M NaOH for 60 min.

Condition/Source	Weight of cotton after hydrolysis(g)	Difference=wt of cotton before hydrolysis-wt after hydrolysis	%age of weight loss
Hotplate	7.94	2.06	20.60%
Sonication	7.25	2.75	27.50%
Autoclave	6.74	3.36	33.60%

3.3. Hydrolysis of 10 g cotton by 6M NaOH solution for 60 min at different conditions

Similarly, 6M NaOH solution was also used for hydrolysis of 10 g cotton sample at different hydrolytic methods (Hotplate, sonication and autoclave) for 60 min to convert cotton to Glucose. Hotplate, sonication, and autoclave were used as heating source for hydrolysis. The extract which obtained by the hydrolysis of 10 g cotton treated with 6M NaOH solution heated for 60 min had greater amount of glucose content as compared to extract by 3M NaOH solution for 60 min. This indicates that the concentration of NaoH has greater effect on the hydrolysis of cotton at different hydrolytic conditions as it is clear from the results that by increasing the concentration of NaOH solution incresed the glucose content in the extract. Autoclave has

shown the the best results for hydrolysis of cotton due to high tempreture 121 ˚C and pressure 1.5 atm as shown in Table 9. The amount of glucose which was obtained was 27.50%, 30.30% and 39.00% for hotplate, sonication and autoclave respectively. The percentage of glucose content obtained after the dry weight of residue was less than that of actual i.e. 27.50%, 29.00% and 38.50 by Hotplate, sonication, autoclave respectively. As explained above this might be due the fact that, the cotton sample also contains some volatile component which was evaporated during heating. Due to which the residue weight is less than the expected shown in the Table 10.

Table 7. Absorbance of standard solution of glucose for making standard curve for Determination of glucose in hydrolysate obtained with 6M NaOH for 60 min.

S. No.	Solutions(ml)	Absorbance
1.	1	0.1
2.	2	0.124
3.	3	0.156
4.	4	0.212
5.	5	0.296

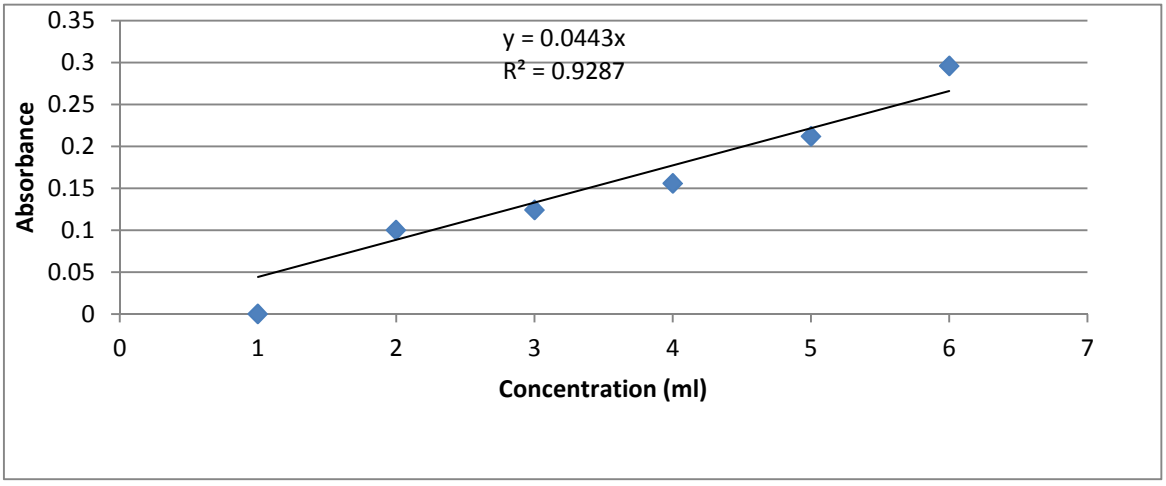

Fig 3. Standard curve for glucose determination in cotton extract treated with 6M NaOH solution for 60 min.

Table 8. Absorbance of samples of extract obtained after hydrolysis of cotton with 6M NaOH solution for 60 min.

S. No.	Condition/Source	Absorbance
1.	Hotplate	0.395
2.	Sonication	0.404
3.	Autoclave	0.498

Table 9. Glucose content in 10g of cotton treated with 6M NaOH solution for 60 min.

Condition/Source	Volume of solution(ml)	Volume of solution after hydrolysis(ml)	Temperature (C)	Glucose content (%)	
				w/v%	w/w%
Hotplate	200	155	84	1.70	27.50
Sonication	200	167	50	1.80	30.30
Autoclave	200	174	121	2.20	39.00

Table 10. Loss in weight of cotton after hydrolysis with 6M NaOH with 6M NaOH for 60 min.

Condition/Source	Weight (g) of cotton after hydrolysis	Difference=wt of cotton before hydrolysis-wt after hydrolysis	%age of weight loss
Hotplate	7.35	2.75	27.50%
sonication	7.10	2.90	29.00%
autoclave	6.15	3.85	38.50%

4. Conclusion

The cotton contains about 90 % of cellulose. It can be hydrolyzed by different methods. In the present work the cotton was hydrolyzed by NaOH solution having different concentrations (3M and 6M) using different hydrolyzing methods (Hotplate, Sonication and Autoclave) to convert cotton to glucose. The conditions for hydrolysis were optimized and it was concluded that autoclave resulted in best hydrolysis of cotton for both 3 M and 6 M NaOH solutions. The hydrolysis of cotton by Autoclave gives best result due to high pressure and temperature which help in conversion of cotton to glucose. This work may provide easy method for hydrolysis at very low cost. The basic theme of this work was to develop a method for converting the cotton waste materials to useful product. The cotton waste residues which are obtained after hydrolysis may also be used for adsorption.

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