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Isolation of cellulose from rice straw and its conversion into cellulose acetate catalyzed

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ABSTRACT
Background:*The extraction and characterizations of α-cellulose from rice straw and wheat straw as well as synthesis of cellulose acetate from the extracted α-cellulose by trans-esterification reaction process. Cellulose acetate was synthesized from the extracted α-cellulose following by trans-esterification reaction. The synthesized cellulose acetate was analyzed by Fourier Transform Infrared Spectroscopy (FTIR) and chemical methods. FTIR spectrometry confirmed the synthesis of cellulose acetate i.e. acetylation of cellulose. Degree of acetylation and degree of substitution of the synthesized cellulose acetates were determined by a chemical method.*
Objectives: *The aim of the study was to evaluate the isolation of cellulose from rice straw and its conversion into cellulose acetate catalyzed.*
Methods:*This cross-sectional study was carried out in the Department of Communicable Diseases Control, Director General of Health Services, Dhaka, Bangladesh. For this investigation the rice plant straw was collected from Magura district region.The collected straws were then treated with 3 wt% nitric acid and 1.5 wt% NaOH solution separately. Statistical analyses of the results were be obtained by using window-based Microsoft Excel and Statistical Packages for Social Sciences (SPSS-24).*
Results:*This study observed that the rice straw contained about 35.67% cellulose, 5.50% hemicellulose and 30.18 % α-cellulose. And the wheat straw contained about 43.99% cellulose, 7.30% hemicellulose and 36.70% α-cellulose.The degree of acetylation and degree of substitution of the synthesized cellulose acetate from rice straw were 39.046% and 2.413 respectively. On the other hand, the degree of acetylation and degree of substitution of the synthesized cellulose acetate from wheat straw were 40.79% and 2.59 respectively.*
Conclusion:*The cellulose acetate can be synthesized rapidly from the rice straw and wheat straw by trans-esterification reaction. Agricultural wastes such as rice straw and wheat straw can be converted to value added material such as cellulose acetate by trans esterification reaction.*
Keywords:*α-cellulose, trans-esterification, FTIR, Rice straw and Wheat straw.*

I. INTRODUCTION

Cellulose is used as building materials by the natural world in the construction of plants and trees. Therefore, cellulose is a major component in agricultural residues and wastes. The chemical compositions of agricultural residues and wastes varies depending upon the type of source. The components of these wastes include cellulose, hemicellulose, lignin, pectin, waxes, water soluble substances etc. [1] In the cell wall of lignocellulosic materials, cellulose exists as aggregates of nanosize fibrils. The cellulose nanofibrils act as reinforcements in soft hemicellulose and lignin matrix. [2] Upgrading of such residues and wastes through the development of cellulose derivatives such as cellulose ethers and cellulose esters can be of high environmental, economic and social interest. Cellulose and its derivatives are used to produce a numerous of products such as filters, fibres, films, membranes, food additives, etc. Cellulose is a polymer of anhydro-glucose unit. Each anhydro-glucose unit of cellulose possesses one primary and two secondary hydroxyl groups, linked to C-2, C-3, and C-6 as shown in Figure 1. Hence the properties of cellulose derivatives and their applications depend on the functional group introduced, the degree of substitution (DS), and the average degree of polymerization (DP) etc. [3].

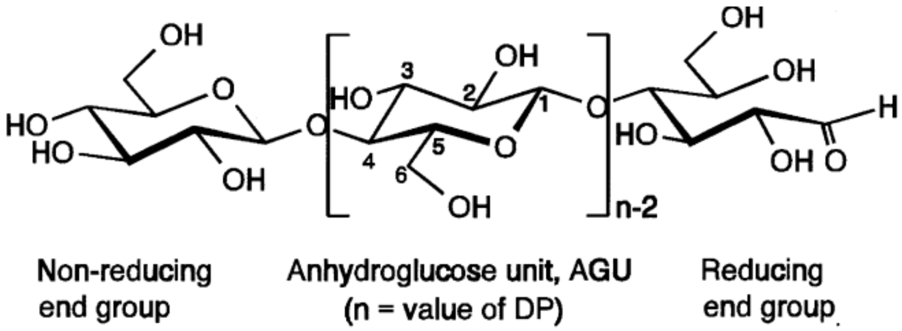

Figure 1: Molecular structure of cellulose.

Bangladesh is the fourth largest rice producing country in the world. About 11.4 million hector land was used for rice cultivation in Bangladesh, [4]. A huge amount of rice straw as agricultural residues and wastes are produced from this land. Some parts of the residues remain in the field and most of these residues are used to feed domestic cattle. Rice straw is a lignocellulosic material. Cellulose, hemicellulose and lignin are the major constituents of rice straw. The chemical compositions of rice straw had also been reported in the literature as cellulose 30-38%, hemicellulose 20–31% and lignin 7–13% [5].

Wheat straw is another major agricultural residue in Bangladesh. About 3.6 lac hectares land were used to cultivate wheat and the total production of wheat crop was about 10 lac tones in Bangladesh. [4] Wheat straw is also a lignocellulosic material like as rice straw. Cellulose, hemicellulose and lignin are the major constituents of wheat straw. The chemical composition of wheat straw is cellulose 38-42%, hemicellulose 34-40%, lignin 15-18% [6]. It can be noticed that the amount of cellulosic material in wheat straw is about 72-82%.

The rice straws and wheat straws which have low value, can be a good source of cellulose and cellulose derivatives. Hence, extraction or isolation of cellulose from rice straw and wheat straw as well as synthesis of chemical derivatives such as cellulose acetate from the isolated cellulose can be a research scope to convert rice straw and wheat straw residues into valuable cellulose ester. Cellulose esters are synthesized by an esterification reaction. Esterification is a process through which the OH group of cellulose is converted into an ester group. [7] The esterification of cellulose is traditionally carried out through the nucleophilic addition of an organic acid anhydride or acid chloride (Figure 2).

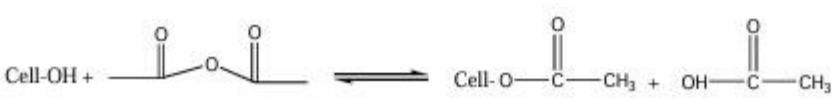

Figure 2: Reaction scheme for the esterification of cellulose with an organic acid.

A variety of anhydrides with different catalysts and solvents are used for esterification of cellulose including acetic anhydride, maleic anhydride, phthalic anhydride, succinic anhydride, and other functionalized anhydrides. [8, 9] However, esterification of cellulose by traditional method needs long time. Isolation of cellulose from rice straw and wheat straw and synthesis of cellulose ester namely cellulose acetate from rice straw cellulose and wheat straw cellulose was planned to be carried out by recently developed rapid trans-esterification method [10]. In this study, vinyl acetate was used in place of acetic anhydride. Figure 3 shows a scheme for trans-esterification reaction for the synthesis of cellulose acetate by vinyl acetate.

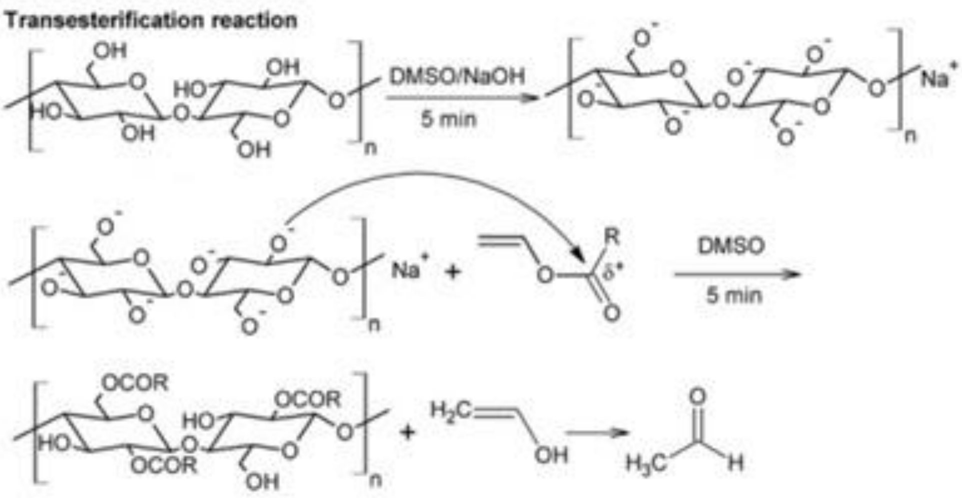

Figure 3:A scheme shows trans-esterification reaction for the synthesis of celluloseAcetate.

II. METHODOLOGY

This cross-sectional study was carried out in the Department of Communicable Diseases Control, Director General of Health Services, Dhaka, Bangladesh. For this investigation the rice plant straw was collected from Magura district region. When rice seed got matured then the seeds were collected from the field and the plant straw were stored as agricultural residues. The straws were dried in air exposing sunlight. These straws were used for the extraction of cellulose. After taking consent and matching eligibility criteria, data were collected from patients on variables of interest using the predesigned structured questionnaire by interview, observation. Statistical analyses of the results were be obtained by using window-based Microsoft Excel and Statistical Packages for Social Sciences (SPSS-24).

III. RESULTS

Table 1: Average compositions of the rice straw

Constituent	Weight (%)
Cellulose	35.67
α-cellulose	30.18
Hemi-cellulose	5.50

Table-1 shows average compositions of the rice straw, it was observed that the rice straw contained about 35.67% cellulose, 5.50% hemicellulose and 30.18 % α-cellulose.

Table 2: Cellulose content in other plant straws

Sources	Cellulose (wt%)
Sugarcane	41-43%
Rye straw	31.8-42.64%
Corn straw	29.80%
Rice straw	32.15%
Wheat straw	34-40%

Table-2 shows Cellulose content in other plant straws, it was observed that the Sugarcane was 41-43%, Rye straw was 31.8-42.64%, Corn straw was 29.80%, Rice straw was 32.15% and Wheat straw 34-40%.

Table 3: Average compositions of wheat straw

Constituent Weight	Weight (%)
Cellulose	43.99
α-cellulose	36.70
Hemi-cellulose	7.30

Table-3 shows average compositions of the wheat straw, it was observed that the wheat straw contained about 43.99% cellulose, 7.30% hemicellulose and 36.70% α-cellulose.

Table 4: The values of α-cellulose content in the straws as well as the values of the degree of acetylation and degree of substitution of the synthesized cellulose acetates

Straw	α- cellulose content (wt%)	Degree of acetylation (%)
Rice	30.0	39.05
Wheat	36.7	40.79

Table-4 shows the values of α-cellulose content in the straws as well as the values of the degree of acetylation and degree of substitution of the synthesized cellulose acetates, it was observed that according to α-cellulose content, 30.0% was rice straw and 36.7% was wheat straw. And according to degree of acetylation 39.05% was rice straw and 40.79% was wheat straw. The values of degree of acetylation and degree of substitution indicated that the synthesized cellulose acetate from both rice straw and wheat straw was a cellulose triacetate.

IV. DISCUSSION

Cellulosic and non-cellulosic compositions in rice straws

Cellulose and α-cellulose were isolated from rice straws by several chemical treatments such as treatment with 3 wt% nitric acid, 1.5 wt% NaOH solution, bleaching with sodium chlorite and finally with 17.5 wt% NaOH solution. Based on the recorded data percentage of cellulose and α-cellulose of rice straw were calculated. It was found that the rice straw contained about 36.0 wt% cellulosic materials. The amount of α-cellulose from bleached rice straw was determined. It was found that the rice straw contained about 30.0 wt % α cellulose. The noncellulosic materials in the rice straw were also calculated and the percentage of noncellulosic materials in the rice straw was about 64.0 wt%. The composition of the rice straw particularly amount of α-cellulose is very similar to the data reported in the literature [5].

Optical microscopic analysis of bleached rice straw/cellulose and α-cellulose

Micro-particles and micro-fibres can be noticed in both images. Micro-particles were probably formed by the agglomeration of cellulose micro-fibres or cellulose fibres. Or cellulose micro-particles in the rice straw might not be separated properly in bleaching operation. However, the number of agglomerated particles were lower in α-cellulose compared with the bleached rice straw or cellulose. According to the optical microscopic analysis, the isolated α-cellulose has particles and fibres shape. It can also be noticed that both length and diameter of the isolated α-cellulose fibres are in micrometer range.

Synthesis of cellulose acetate from the rice straw cellulose and physical appearance of the synthesized cellulose acetate

In this study cellulose acetate was synthesized from α-cellulose isolated from the rice straw by trans-esterification reaction. Traditionally cellulose acetate is synthesized by the reaction of anhydride groups of acetic anhydride and hydroxyl groups of cellulose in presence of sulphuric acid as catalyst. The reaction time is about 12-24 hours. In this study vinyl acetate in place of acetic anhydride was used to synthesize cellulose acetate. Acetyl group of vinyl acetate reacts with the hydroxyl groups of cellulose and form an ester linkage through trans-esterification reaction mechanism. The reaction time is very short about 5 minutes.After complete reaction the synthesized cellulose acetate was dissolved in DMSO. The undissolved solid part was then removed from the liquid by filtration. When water was added into the viscous brown liquid a light-yellow solid material appeared. The solid was separated by filtering. The solid was then dried under a fan at room temperature and finally in an oven at 60 °C.

FTIR analysis of bleached rice straw/cellulose, α-cellulose and cellulose acetate

FTIR analysis was carried out to investigate various functional groups of cellulose and α cellulose isolated from the rice straw The peaks in the region 2943-2856 cm-1 are assigned to the C H stretching vibration from the -CH2 group of α-cellulose. The strongest bands in the spectra at 1103 and 1051 cm-1 are assigned to –CO stretching for secondary alcohol. [11]

If we compare the spectrum of the synthesized cellulose acetate with the spectrum of α-cellulose obtained from the rice straw the following changes can be observed:

-decrease of the peak depth of the band 3600-3200 cm−1centered at 3400 cm−1, corresponding to the stretching of OH groups of cellulose, due to the free OH group replacement for the acetyl groups [12, 13];

-new peak at 1732 cm−1, corresponding to the stretching of C=O bond of ester carbonyl, which is present in the structure of the acetyl groups [12];

-new peak at 1362 cm−1, corresponding to the vibration of C H bond, which is present in the structure of the acetyl groups [10];

-new peak at 1224 cm−1, corresponding to the vibration of C-O bond, which is responsible for the link between cellulose and acetyl group [10]

In the FTIR spectrum of the synthesized cellulose acetate, the carbonyl (CO) group peak at 1732 cm-1, which is not present in the FTIR spectrum of α-cellulose, clearly indicates the acetylation of cellulose. In addition, the depth of the broad stretching band centered at 3400 cm-1 in the FTIR spectrum of α-cellulose is also decreased in the FTIR spectra of synthesized cellulose acetate. This is also an evidence of trans-esterification reaction between OH group of cellulose and vinyl acetate. Hence, FTIR analysis confirmed the acetylation of α-cellulose obtained from the rice straw.

Cellulosic and non-cellulosic compositions in the wheat straw

Cellulose and α-cellulose were obtained from the wheat straw by similar chemical treatments as reported in Part-II Rice straw such as dilute nitric acid, NaOH treatments, bleaching with sodium chlorite and finally with 17.5 wt% NaOH solution. After each chemical treatment the weight loss data of the treated materials were recorded. Based on the recorded data percentage of cellulose and α-cellulose in the wheat straw were calculated. It was found that the wheat straw contained about 44 wt% cellulose and 36.7 wt% α-cellulose. The noncellulosic materials in the wheat straw were also calculated and the percentage of noncellulosic materials in the wheat straw was about 56.0 wt%. The composition of the wheat straw particularly amount of α-cellulose is very similar to the data with the literature. [14]

Optical microscopic analysis of bleached rice straw/cellulose and α-cellulose

Micro-particles and micro-fibres can be noticed in both images. Micro-particles are probably formed by the agglomeration of cellulose micro-fibres or cellulose fibres. Or cellulose micro-particles in the wheat straw might not be separated properly in bleaching operation. However, the number of agglomerated particles were lower in the α-cellulose photograph compared with the bleached wheat straw or cellulose. According to the optical microscopic analysis, the isolated α-cellulose has particles and fibres shape. It is also noticed that both length and diameter of the isolated α-cellulose fibres are in micrometer range as similar to rice straw based α-cellulose.

Physical appearance of the synthesized cellulose acetate from the wheat straw based α-cellulose

After complete reaction the synthesized cellulose acetate was dissolved in DMSO. The undissolved solid part was then removed from the liquid by filtration. The filtrate i.e. When water was added into the viscous brown liquid a light-yellow solid material appeared. The solid was separated by filtering. The solid was then dried under a fan at room temperature and finally in an oven at 60 °C.

FTIR analysis of the bleached wheat straw/cellulose, α-cellulose and cellulose acetate

FTIR analysis was carried out to investigate various functional groups of cellulose and α cellulose obtained from the wheat straw. -OH, stretching vibration and hydrogen bond of the hydroxyl groups of cellulose can be seen in the spectra of both cellulose and α-cellulose. The peaks at 2943 - 2856 cm-1 are assigned to the C-H stretching vibration from the -CH2 group of α cellulose. The strongest bands in the spectra at 1103 and 1051 cm-1 are assigned to –CO stretching for secondary alcohol. [11]

If we compare the spectrum of the synthesized cellulose acetate with the spectrum of α-cellulose then the following changes can be observed like as the spectra analyzed for the rice straw:
-decrease of the peak depth of the band 3600-3200 cm−1centered at 3400 cm−1, corresponding to the stretching of OH groups of cellulose, due to the free OH group replacement for the acetyl groups. [12, 13]
-new peak at 1733 cm−1, corresponding to the stretching of C=O bond of ester carbonyl, which is present in the structure of the acetyl groups [12];
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In the FTIR spectrum of synthesized cellulose acetate, the carbonyl (CO) group peak at 1733 cm-1, which is not present in the FTIR spectrum of cellulose clearly indicates the acetylation of cellulose. In addition, the depth of the broad stretching band centered at 3400 cm-1 in the FTIR spectrum of α-cellulose is also decreased in the FTIR spectra of synthesized cellulose acetate. This is also an evidence of trans-esterification reaction between OH group of cellulose and vinyl acetate. Hence, FTIR analysis confirmed the acetylation of α-cellulose obtained from the wheat straw.

Comparison of the synthesized cellulose acetates from the rice straw and wheat straw

FTIR spectra of the synthesized cellulose acetates from the rice straw and wheat straw are put together in Figure 35 for a comparison. Both spectra exhibit almost same peaks particularly the most significance peak at 1732 cm-1 related to the carbonyl (CO) group, which is strong evidence of the esterification reaction. FTIR spectra confirm that the trans-esterification reaction does not depend on the source of α-cellulose. However, if the two spectra are observed closely then a little change in the region of 3000-2800 cm-1 can be seen. The peaks in this region are related stretching vibration of CH, CH2 and CH3 groups. The peak related to the CH3 group is sharper in the spectrum of the synthesized cellulose acetate from the wheat straw as compared with the rice straw.

Limitations of the study

The present study was conducted in a very short period due to time constraints and funding limitations. The small sample size was also a limitation of the present study.

V. CONCLUSION

In this paper it was possible to synthesize cellulose acetate from two agricultural residues namely rice straw and wheat straw by a trans-esterification reaction. α cellulose fibers isolated from the rice straw and wheat straw were used to synthesis cellulose acetate through trans-esterification reaction using vinyl acetate monomer. The values that obtained for the wheat straw are little bit higher than that of the rice straw. Considering the higher values of α-cellulose content in the wheat straw as well as degree of acetylation and degree of substitution values of the synthesized cellulose acetate from the wheat straw it can be suggested that the wheat straw can be a better resource for the cellulose as well as for the synthesis of cellulose acetate compared with the rice straw.

VI. RECOMMENDATION

This study can serve as a pilot to much larger research involving multiple centers that can provide a nationwide picture, validate regression models proposed in this study for future use and emphasize points to ensure better management and adherence.

ACKNOWLEDGEMENTS

The wide range of disciplines involved in isolation of cellulose from rice straw and its conversion into cellulose acetate catalyzed research means that editors need much assistance from referees in the evaluation of papers submitted for publication. I would also like to be grateful to my colleagues and family who supported me and offered deep insight into the study.

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