Scholars Journal of Agriculture and Veterinary Sciences

OPEN ACCESS

Abbreviated Key Title: Sch J Agric Vet Sci
ISSN 2348–8883 (Print) | ISSN 2348–1854 (Online)
Journal homepage: https://saspublishers.com

Preparation and Characterization of Cellulose Derivatives from Agricultural Wastes

Md. Eleas Hossain[1*]

[1]Lecturer of Chemistry, Chitra Model College, Bagharpara- Jashore, Bangladesh

DOI: 10.36347/sjavs.2024.v11i02.001 | **Received:** 10.02.2024 | **Accepted:** 18.03.2024 | **Published:** 09.04.2024

***Corresponding author:** Md. Eleas Hossain
Lecturer of Chemistry, Chitra Model College, Bagharpara- Jashore, Bangladesh.

Abstract | **Original Research Article**

Background: Rice straw was pretreated with dilute alkaline and acid solutions to extract cellulose, which was then converted into cellulose acetate with acetic anhydride and phosphotungstic acid (H3PW12O40·6H2O). The concentration of KOH and the duration of immersion in acetic acid solution affected the removal of hemicellulose and lignin, and after pretreatment with 4% KOH and immersion in acetic acid for 5 hours, the treated rice straw contained 83wt.% cellulose. Phosphotungstic acid has been shown to be an excellent catalyst for the acetylation of rice straw cellulose. The degree of substitution (DS) values had a substantial effect on the solubility of cellulose acetate, and acetone-soluble cellulose acetate with DS values about 2.2 can be generated by varying the amount of phosphotungstic acid and acetylation duration. ***Objectives:*** The aim of the study was to evaluate the preparation and characterization of cellulose derivatives from agricultural wastes. ***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:*** Trans-esterification allows for the fast synthesis of cellulose acetate from rice and wheat straw. Agricultural wastes like rice straw and wheat straw can be transformed into value added materials like cellulose acetate using a trans esterification procedure.

Keywords: α-cellulose, trans-esterification, DS, Rice straw and Wheat straw.

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INTRODUCTION

Chemical modification of cellulose is the most common way for modifying the characteristics of natural biopolymers [1,2]. This renewable resource derived from numerous biomasses is utilized in the framework of sustainable development. Carboxymethylcellulose is used in nontoxic, biodegradable materials with a growing number of applications [3]. One of the most essential qualities of carboxymethylation processes is their capacity to create cellulose derivatives that are water soluble [4]. As a result, CMC has a wide range of industrial applications, including dissolved and dispersed adhesives, water binders, thickeners, film formers and suspension aids. CMC is also used to improve the quality of paper. Additionally, CMC is used as a color thickener in the textile sector [5]. The purified product contributes significantly to food items as a preservative for fresh fruit coating, and it is also employed as a thickening in the pharmaceutical and cosmetic industries.

Overexploitation of fossil fuels has led to resource scarcity and global climate disruption. Because of the world's growing population and increased demand for energy, there is a need to research sustainable energy. Biomass-based renewable energy, such as cellulosic ethanol, is seen as a potential solution to the energy crisis and global warming [6]. Major cellulosic ethanol sources include switchgrass, crop wastes, and forestry biomass [7]. Cellulose is the primary component of plant biomass, which is widely available and does not compete with food supplies.

Although various lignocellulosic agricultural residues such as corn fiber, corn stover, wheat straw, rice straw, and rice hull contain about 45-70 wt.% complex carbohydrates, including cellulose and hemicellulose, and thus can serve as low-cost feedstocks for the production of fuel ethanol, rice is the world's largest cereal crop, with rice straw accounting for approximately 45% of rice production volume [8]. As a result, rice straw is the most readily available cellulose source derived from agricultural crop leftovers worldwide. Rice straw is mostly made of cellulose (38.3%), hemicellulose (31.6 wt.%), lignin (11.8 wt.%), and ash (18.3 wt.%), hence efficient separation is required before converting it into usable chemicals [9, 10].

Cellulose is frequently modified through chemical esterification, and cellulose acetate is one of the most economically important cellulose derivatives, utilized in a variety of applications including fiber, film, paint, filter, and dialyzer [11]. The global production of cellulose acetates is projected to be 1.5 billion pounds per year. Wood and cotton are the primary supplies for industrial acetylation [12]. In recent years, low-cost lignocellulosic biomass has gained popularity as a renewable resource due to its abundance and widespread cultivation. So far, few studies on the production of cellulose acetate from rice straw have been proposed. Furthermore, strong inorganic acid-catalyzed operations frequently cause corrosion and generate a substantial amount of waste. As a result, there is a need to investigate greener methods for preparing partly substituted and acetone-soluble cellulose acetates with DS values ranging from 2.2 to 2.7 [13].

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).

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.

DISCUSSION

Cellulosic and non-cellulosic compositions in rice straws

Rice straws were chemically treated to separate cellulose and α-cellulose using 3 wt% nitric acid, 1.5 wt% NaOH solution, sodium chlorite bleaching, and 17.5 wt% NaOH solution. Rice straw's cellulose and α-cellulose percentages were determined using recorded data. It was discovered that rice straw had around 36.0 wt% cellulosic materials. Noncellulosic elements in rice straw were also calculated, with a percentage of around 64.0 wt%. The rice straw's composition, notably its α-cellulose content, is consistent with previous research [14].

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

Microparticles and microfibres may be seen in both pictures. Microparticles were most likely produced by the aggregation of cellulose microfibres or cellulose fibres. Alternatively, the cellulose microparticles in rice straw may not be adequately separated during the bleaching operation. However, α-cellulose had fewer agglomerated particles than bleached rice straw or cellulose. Optical microscopic investigation shows that the isolated α-cellulose exhibits particle and fiber shapes.

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

This study produced cellulose acetate from α-cellulose extracted from rice straw using a trans-esterification procedure. Traditionally, cellulose acetate is made by reacting anhydride groups of acetic anhydrides with hydroxyl groups of cellulose in the presence of sulfuric acid as a catalyst. In this study, cellulose acetate was synthesized using vinyl acetate rather than acetic anhydride. The acetyl group of vinyl acetate combines with the hydroxyl groups of cellulose, forming an ester bond via the trans-esterification reaction process. After the reaction was complete, the produced cellulose acetate was dissolved in DMSO. When water was added to the viscous dark liquid, a light-yellow solid substance formed. The solid was then dried with a fan at ambient temperature and then in an oven at 60 °C.

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

FTIR analysis was conducted to study the functional groups of cellulose and α cellulose isolated from rice straw. The peaks at 2943-2856 cm-1 correspond to the C H stretching vibration from the -CH2 group of α-cellulose. The brightest bands in the spectra at 1103 and 1051 cm-1 correspond to -CO stretching for secondary alcohols [15].

When comparing the spectra of synthetic cellulose acetate to that of α-cellulose produced from rice straw, the following modifications can be observed:

- The peak depth of the band 3600-3200 cm−1 centered at 3400 cm−1 decreases due to stretching of cellulose's OH groups caused by free OH group replacement for acetyl groups [16, 17].
- A new signal at 1732 cm−1 indicates the stretching of the C=O bond of the ester carbonyl, which is present in the structure of the acetyl groups [18].
- The new peak at 1362 cm−1 corresponds to the vibration of the C H bond in the structure of the acetyl groups [19].
- A new peak at 1224 cm−1 corresponds to the vibration of the C-O bond, which connects cellulose to the acetyl group [19].

Degree of acetylation of the synthesized cellulose acetate from the rice straw based α-cellulose

A saponification procedure was used to evaluate the acetylation percentage of the produced cellulose acetate. The acetate content of the produced cellulose acetate was determined following the procedure described in section 3.7.3. The acetylation percentage in the synthesized cellulose acetate was approximately 39.05%. The acetylation % of the produced cellulose revealed that the cellulose acetate is a triacetate.

Degree of substitution of the synthesized cellulose acetate from α-cellulose obtained from the rice straw

The degree of substitution of the synthesized cellulose acetate was also determined by the saponification reaction. At first, the acetate content of the synthesized cellulose acetate was determined using the procedure as reported under section 3.7.3. Thereafter, based on the acetate content of the synthesized cellulose acetate the degree of substitution was calculated. The degree of substitution of the synthesized cellulose acetate was 2.413. The value of degree of substitution (2.413) of the synthesized cellulose acetate indicated that the synthesized cellulose acetate was a tri-acetate cellulose.

Cellulosic and non-cellulosic compositions in the wheat straw

Wheat straw was treated with weak nitric acid, NaOH, sodium chlorite, and 17.5 wt% NaOH solution to extract cellulose and α-cellulose, identical to the methods used in Part-II rice straw. The weight loss data for each chemical treatment were recorded. We determined the percentages of cellulose and α-cellulose in wheat straw using recorded data. Wheat straw comprised around 44% cellulose and 36.7 % α-cellulose. The wheat straw included around 56.0 weight percent noncellulosic components. Wheat straw's composition, notably its α-cellulose content, is consistent with existing literature [19].

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

Microparticles are most likely created by the aggregation of cellulose microfibres or cellulose fibres. Alternatively, cellulose microparticles in wheat straw may not be adequately separated during the bleaching operation. The α-cellulose image showed less agglomerated particles than the bleached wheat straw or cellulose. Optical microscopic investigation shows that the isolated α-cellulose exhibits particle and fiber shapes.

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

After the reaction was complete, the produced cellulose acetate was dissolved in DMSO. The liquid was then filtered to eliminate the undissolved solid portion. The filtrate, or when water was added to the thick brown liquid, a light-yellow solid substance formed. The solid was separated via filtration. The solid was then dried with a fan at ambient temperature and then in an oven at 60 °C.

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

FTIR analysis was conducted to examine the functional groups of cellulose and α cellulose extracted from wheat straw. The spectra of cellulose and α-cellulose show -OH, stretching vibration, and hydrogen bonds of the hydroxyl groups. The peaks at 2943 - 2856 cm-1 correspond to the C-H stretching vibration of the -CH2 group in α cellulose. The brightest bands in the spectra at 1103 and 1051 cm-1 correspond to -CO stretching for secondary alcohols [16].

When comparing the spectrum of produced cellulose acetate with that of α-cellulose, we can see the following alterations, similar to the spectra analyzed for rice straw:

The peak depth of the band 3600-3200 cm−1 decreased, centered at 3400 cm−1, indicating stretching of cellulose's OH groups due to free OH group replacement for acetyl groups [17, 18].

The new signal at 1733 cm−1 corresponds to the stretching of the C=O bond of ester carbonyl, which is found in the structure of acetyl groups [17].

The new peak at 1362 cm−1 corresponds to the vibration of the C H bond in the structure of the acetyl groups [15].

A new peak at 1224 cm−1 corresponds to the vibration of the C-O bond that connects cellulose to the acetyl group [15].

Degree of acetylation of the synthesized cellulose acetate from the wheat straw based α-cellulose

The percentage of acetylation and degree of substitution of the generated cellulose acetate from wheat straw-based cellulose were measured using a saponification procedure similar to that used to synthesis cellulose acetate from rice straw cellulose. The acetate content of the produced cellulose acetate was determined following the procedure described in section 3.7.3. The acetylation percentage in the synthesized cellulose acetate was approximately 40.79%. The acetate content percentage in the produced cellulose acetate suggests that it was a triacetate.

Degree of substitution of the synthesized cellulose acetate from the wheat straw based α-cellulose

The saponification reaction measured the degree of substitution in the produced cellulose acetate. First, the acetate content of the produced cellulose acetate was determined using the procedure described in section 3.7.3. The degree of substitution was then estimated using the acetate content of the produced cellulose acetate. The degree of substitution for the produced cellulose acetate was 2.59. The degree of substitution (2.59) of the produced cellulose acetate suggested that it was triacetate cellulose.

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

The FTIR spectra of the produced cellulose acetates from rice and wheat straws are compared. Both spectra show nearly identical peaks, particularly the most significant peak at 1732 cm-1 corresponding to the carbonyl (CO) group, which provides solid proof of the esterification reaction. The trans-esterification reaction is independent of the α-cellulose supply, as confirmed by FTIR spectra. However, if the two spectra are closely examined, a small variation in the range of 3000-2800 cm-1 can be noted. The peaks in this region represent stretching vibrations of the CH, CH2, and CH3 groups. In the spectrum of the produced cellulose acetate from wheat straw, the peak associated with the CH3 group is sharper than that of 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.

CONCLUSION

In this paper, cellulose acetate was manufactured from two agricultural residues, rice straw and wheat straw, using a trans-esterification method. Cellulose acetate was synthesized using α cellulose fibers from rice and wheat straw through trans-esterification with vinyl acetate monomer. Wheat straw yields slightly higher values than rice straw. Wheat straw has higher α-cellulose concentration, acetylation, and substitution values compared to rice straw, making it a better resource for cellulose and cellulose acetate production.

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

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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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