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SYNTHESIS OF COMMERCIALLY VALUABLE CELLULOSE ACETATE FROM AGRICULTURE WASTE

B. KANNAMBA. K. GOPINATH, Y. JAHNAI
Department of Chemistry-PG, Andhra Loyola College,
Vijayawada, A.P. India.

Corresponding author: B. Kannamba: kannambanitw@gmail.com

ABSTRACT

Cellulose is the most abundant natural biopolymer. A verity of cellulose derivatives were prepared, and have commercial and industrial applications. The agriculture waste products such as cotton byproducts, banana pith, wheat and rice straw are the rich sources of cellulose. Currently, agriculture waste biomass has been used as raw material for the preparation of verity of cellulose derivatives. Cellulose acetate is one of the soluble cellulose derivatives and it is extensively used in textile and plastic industries for the preparation of sunglasses, linings, home furnishings, draperies, slip covers, cigarette and other filters, ink reservoirs for fiber tip pens. In the present work, agriculture waste biomass is converted into a commercially valuable cellulose acetate. The prepared cellulose acetate is characterized by FTIR spectroscopy.
Key wads: Cellulose; Cellulose acetate; Agriculture waste

1. Introduction

Cellulose is the structural component of plant cell wall and is the most abundant natural biopolymer. Nowadays a number of cellulose derivatives such as methyl cellulose, ethyl cellulose, carboxy methyl cellulose, cellulose acetate have been prepared to change physical and chemical properties of cellulose [1]. Chemically modified cellulose derivatives are tailored for specific industrial applications. For instant cellulose acetate is extensively used in textile industries for preparing blouses, gloves, draperies and plastic industries for the preparation of sunglasses, linings, home furnishings, slip covers, cigarette and other filters, ink reservoirs for fiber tip pens [2].

Generally, cellulose derivatives are prepared from raw cellulose or wood pulp. The wood pulp was obtained from forest, which is the renewable resource. The production of wood pulp from forest trees is one of the main causes for deforestation. Most of the agricultural waste materials such as cotton byproducts, sugar cane baggage, banana byproducts, wheat and rice straw contain cellulose in abundant. Preparation of cellulose derivatives from these agricultural waste materials is an environmental friendly and economically feasible approach. Studies on synthesis of cellulose derivatives from rice husk, cotton byproducts and sugar cane baggage have been reported in the literature [3-6]. Banana fruit-bunch-stem (FBS) is one of the rich sources of cellulose. Studies on synthesis of cellulose acetate from Banana FBS have not been reported. In the present study, an attempt is made to synthesis cellulose acetate from banana FBS, which is an inexpensive and freely available agricultural waste product. The prepared cellulose acetate is characterized by FTIR spectroscopy. The degree of acetylation was determined to estimate the acetyl groups on banana FBS.

2. Experimental

2.1. Materials and chemicals

Banana FBS used in the present study was collected from local market. The material was first washed with distilled water then dried in sunlight. The dried material was cut into small pieces and then powdered with kitchen blender. The powdered material stored at room temperature and used. Acetic anhydride, hydrogen peroxide and all other chemicals of analytical reagent grade were used.

2.2. Preparation of cellulose acetate

Preparation of cellulose acetate is three step processes. First the powdered banana FBS is pretreated with 6 % NaOH solution, heated in a water bath for 45 min, filtered and washed with hot water to remove unwanted materials. This process was repeated to remove hemicelluloses and lignin effectively from the banana FBS. The second step is the bleaching step, the material was suspended in a NaOH solution at pH 12.0 with 3% H_2O_2 in hot water bath at 90^oC. After 60 min the material was filtered, washed with hot water to remove excess NaOH solution and dried. In acetylation of cellulose, 1 g of above product was refluxed with 5 ml of acetic anhydride for 50 min at about 80^oC in presence of iodine as catalyst. The product was then washed with hot water until the product is free from un-reacted reagents and byproducts. The prepared cellulose acetate dried at 50^oC in hot air oven and stored at room temperature.

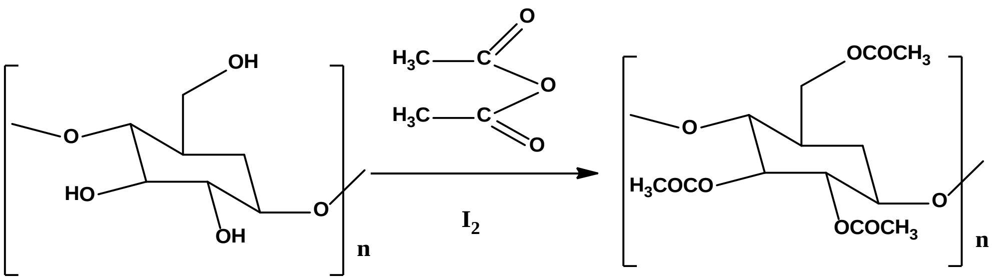
2.3. Characterization of cellulose acetate

The prepared cellulose acetate is characterized by FTIR spectroscopy. The FTIR spectra of cellulose acetate were recorded using Shimadzu 8201PC FTIR spectrometer with a resolution of 4 cm^{-1}. The powdered cellulose acetate was blended with spectroscopic grade KBr in an agate mortar and then pressed into a tablet. The FTIR spectrum of the tablet was scanned within the spectral range of 400−4000 cm^{-1}.

3. Results and discussion

3.1. Optimum reaction conditions

Generally, preparation of cellulose acetate from any material involves three steps [3]. In the first step alkali pretreatment was carried out to remove lignin and the hemicelluloses from agro waste material. The second step is the bleaching step. In this step, the alkali treated material was treated with H_2O_2 for whitening the material. In the last step, the bleached material is acetylated with acetic anhydride. The concentration of alkali, H_2O_2 and acetic anhydride required for effective acetylation is varied from material to material. Therefore, the optimum time, temperature and concentration of reagents required for acetylation of powdered banana FBS was conducted. Acetylation reaction time of 60 min, temperature of 90^oC and 12% NaOH solution for alkali treatment and 3% H_2O_2 for bleaching are the optimum conditions for conversion of cellulose of banana FBS to cellulose acetate.



Scheme 1. Cellulose acetylation reaction

3.2. Degree of acetylation (DA)

The percentage of acetylation of sample was determined quantitatively by titration method [7]. The titration procedure was quantified by the amount of the acetyl groups are abstracted with the treatment of NaOH solution. Exactly, 1 gm of acetylated banana FBS was immersed in 0.1 N of NaOH solution. After 24 h, 5 mL of NaOH solution was titrated with 0.1 N HCl solution, with phenolphthalein as indicator. The amount of the hydroxyl ions participated in the deacetylation reaction of cellulose acetate was calculated from the following equation.

$$\text{Acetylation (\%)} = \frac{m_i - m_f}{m_i} \times 100 \qquad (1)$$

Where, m_i and m_f are the moles of the acetyl groups in the sample material before and after the alkaline treatment. The degree of acetylation (DA) of sample was calculated from the % of acetylation by using the following expression [8].

$$DA = \frac{3.86 \times \text{acetylation}\ \%}{102.4 - \text{acetylation}\ \%} \qquad (2)$$

The acetylation percentage of powdered banana FBS is found to be 98% and the degree of acetylation calculated from acetylation percent is 85.97, which is a significant degree of acetylation. The high acetylation percent and degree of acetylation of powder banana FBS suggest that the adopted procedure is an effective procedure.

3.3. Mass loss of material

After pretreatment, bleaching and acetylation reactions certain quantity of material was lost. The mass loss of sample material influences the % yield of cellulose acetate. The % yield was calculated from mass of material before and after acetylation using the following equation.

$$\text{Mass loss}\ (\%) = \frac{m_i - m_d}{m_i} \times 100 \qquad (3)$$

Where, m_i and m_d are the weight of sample material before and after reaction. The calculated percentage of mass loss is found to be 0.005%. The observed percentage of mass loss is not significant therefore it can be neglected. This result indicates the procedure established for cellulose acetate preparation is an effective and economically feasible.

3.4. Characterization of cellulose acetate

Cellulose acetate prepared from banana FBS was characterized by FTIR spectral studies. The FTIR spectra of sample material before and after acetylation were compared. Before acetylation, the FTIR spectrum shows a broad band at 3396 cm^{-1}, which is attributed to O-H stretching vibration of alcohol group. In addition, a sharp peak observed at 1033 cm^{-1} corresponds to C-O-C pyranose ring skeletal vibration. After acetylation, a broad band at 3396 cm^{-1} corresponds to O-H stretching vibration is reduced. This clearly indicates that some of the hydroxyl groups (-OH) of cellulose was replaced by acetyl (-COCH3) groups [9]. At the same time, a strong and sharp peak at 1022 cm^{-1} corresponds to C-O stretching vibration of acetyl group is appeared in the spectrum. These results further provide the evidence of acetylation of banana FBS powder.

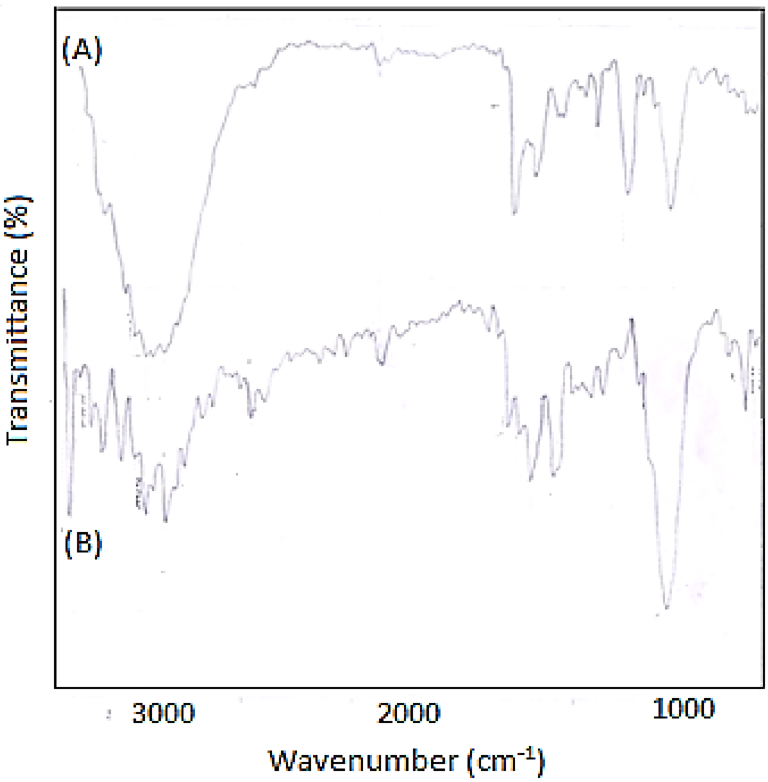


Figure.1. FTIR spectrum of banana FBS powder, (A) before and (B) after acetylation.

4. Conclusions

The optimum reaction conditions required for converting banana FBS powder to cellulose acetate was established. The prepared cellulose acetate is characterized by FTIR spectroscopy. FTIR spectral studies provide the evidence of acetylation of banana FBS. Nearly 98% acetylation of banana FBS powder was achieved under the optimum experimental conditions. This high acetylation percentage infers the production of cellulose acetate from powdered banana FBS is economically promising method. And it provides an opportunity to commercial production of cellulose acetate from banana FBS powder.

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