Preparation and Characterization of Cellulose from Corncob

Phyo Nandar Cho

Abstract

In this research, cellulose was synthesized from corncob. Corncob was collected during the corn grain harvest. Cellulose was prepared from corncob using a soda process. The three different concentrations of sodium hydroxide 1 %, 2 %, and 3 % were used. Corncob cellulose was characterized by Fourier Transformed Infrared (FT-IR) spectroscopy, Scanning Electron Microscope (SEM), and X-Ray Diffraction (XRD) method. The result of the FT-IR spectrum shows that the functional groups of cellulose pulp were confirmed. SEM data shows the fibrous morphology of corncob cellulose. The crystal size of the obtained cellulose analyzed by XRD was 29.3 nm. This corncob cellulose can be used as a superabsorbent for the water retention test.

Keywords: Corncob, Cellulose, FT-IR, SEM, XRD

Introduction

Corn is the most significant cereal crop after rice in Myanmar and it is farmed all around the country. Corn is consumed in domestic and also exported abroad. Corn is grown on over 400000 ha in our country. Therefore corncob (CC) is an abundant agricultural waste. Agricultural wastes such as wheat straw, rice straw, corncob, corn straw, cassava dregs, and others can be used as a more attractive alternative to get cellulose. (Helmiyati, 2017).

Corncobs contain 36 % cellulose, 26 % hemicellulose, and 17 % lignin,(Helmiyati,2014). This research aims to extract cellulose from corncob and will be used as a superabsorbent.

Cellulose, an organic compound that has the formula $(C_6H_{10}O_5)_n$ a polysaccharide containing a linear chain of several hundred to many thousands of β (1-4) linked D – glucose units. Some species of bacteria secrete to form biofilms. Cellulose is the most abundant organic polymer on earth..

Cellulose is made up of most of the plant's cell walls. Cellulose is probably the most abundant organic compound on earth because it is made by all plants. Besides being a primary building material for plants, cellulose is used in many different ways. According to how it is treated, cellulose can be used to make paper, film, and plastics, in addition to having many other industrial uses. The paper in this book contains cellulose, as do some of the clothes you are wearing. Cellulose is also a major source of needed fiber in our diet for human beings. Chemists and biologists describe cellulose as a complex carbohydrate. Carbohydrates are organic compounds that are made up of carbon, hydrogen, and oxygen and function as sources of energy for living things. Plants can make their own carbohydrates which are used for energy and to build their cell walls (Htun Min Latt, 2014).

Cellulose, which is one of the most widely used natural substances, has become one of the most important commercial raw materials. The major sources of cellulose are plant fibers and of course wood. Cellulose cannot be soluble in water, so, is easily separated from the other constituents of a plant. Cellulose is mainly used in the textile industry, as chemical fibers, chemical filters, and fiber-reinforcement composites, due to their similar properties to engineered fibers, being another option for biocomposites and polymer composites. Despite being an important natural resource, many of the products that were made from cellulose are being produced

¹ Daw, Demonstrator, Department of Chemistry, Mandalar University

easier and cheaper using other materials. This research work focuses on the use of corncob waste as a substrate for the preparation of low-cost superabsorbent copolymers for economic and environmental purposes (Swantomo,2013).

Materials and Methods

Sample Collection and Preparation of Cellulose

Corncob was collected during corn grain harvest. Corncob was used as raw material. Sodium hydroxide (NaOH) was used to extract cellulose from corncob. Potassium hydroxide (KOH) and hydrogen peroxide (H₂O₂) were used to remove impurities from cellulose. Sodium hypochlorite (NaOCl) was used to remove the color.

Procedure

The corncob was washed by distilled water to remove dust and impurities and sundried. The dried corncob was cut into the small pieces and grinded with grinder. The corncob powder was sieved by using 100 mesh size sieves. The cellulose pulp was prepared employing three different concentration of sodium hydroxide 1 %, 2 % and 3 %. 30 g of sifted raw corncob powder was mixed with 300 mL of 1 % sodium hydroxide and refluxed in water bath at 70°C for 1hour and then filtered. The resulting residue was mixed with 500 mL of 5 % potassium hydroxide and 10 % hydrogen peroxide and shaken for two hours and then filtered. The resulting cellulose pulp was bleached with sodium hypochlorite and filtered and washed until neutral. Then bleached pulp was oven dried. Raw materials of cellulose pulp are shown in the following Figure 1.



Raw Corncob



Corncob Powder



Corncob Pulp

Figure 1. Raw corncob cellulose

10.00 g of the bleached pulp was treated with 50 mL of 17.8 % sodium hydroxide solution for about 25 minutes. The material was filtered and washed with a litre of water. The residue was rinsed with 100 mL of 10 % acetic acid. Finally, the acid was completely removed by washing the pulp with a litre of hot water. The pulp obtained was dried at room temperature for 24 hours followed by oven dry at 100 °C for 2 hours (Swantomo, 2013). Similarly, cellulose pulp was also prepared by using 2 % and 3 % sodium hydroxide solution. Dried corncob cellulose powders are shown in the following Figure 2.



Figure 2. 1 %,2 % and 3 % Dried corncob cellulose

Scanning Electron Microscope (SEM) Analysis of Cellulose

Scanning electron microscopy was used to study the morphological structure of cellulose. Original corncob cellulose was qualitatively analysed by SEM method. The morphological structure of cellulose was measured at Department of Chemistry in Yadanabon University.

FT-IR Analysis

The Fourier Transformed Infrared (FT-IR) Spectroscopy was used to identify the functional group of the active compounds based on the peak value in the region of infrared radiation. The FT-IR spectra of the original corncob cellulose was recorded by FT-IR spectrophotometer (PerkinElmer, UK, L1600400) in the wave number range 400-4000cm⁻¹. The FT-IR spectrum of corncob cellulose was measured at Department of Chemistry in Monywa University.

Crystallization Analyzed by XRD

The crystallization of corncob cellulose was examined by XRD measurement performed on a multiplex 2 kW (Rigaku, Japan) using Cu K-alpha ($\Lambda = 1.54056 \text{ A}^\circ$) at 40 kV and 50 mA. Scherrer's equation was used for calculating crystallite size in plane

$$D = \frac{k\lambda}{\beta \cos\theta}$$

Where λ is wavelength of X-ray tube, $\lambda = 1.54056 \text{ A}^\circ$, β is the pure integral of width of the reflection at half maximum height (FWHM, full width at half maximum) of 113 peak at 2θ about 22° and 0.89 is the Scherrer constant. XRD analysis was measured at University Research Centre (URC) Yangon.

Results and Discussion

Effect of Alkali Concentration on Yield of Corncob Pulp

The three different concentrations of alkali were used for corncob powder 30 g and the results are shown in the following table 1.

Sample	Concentration	of	Cellulose	Cellulose
	NaOH $(g_{mL} 1)$		Yield	Yield (%)
			(g)	
1	1		9.90	33.0
2	2		8.90	30.0
3	3		8.10	27.0

Table 1. The Different	Concentration on	Yield of (Corncob Pulp
------------------------	-------------------------	------------	--------------

According to the results, when 1 % and 2 % concentrations of alkali were used, the resulting pulp was obtained a high yield (33 %) and (30 %). But it was hard and colored. The concentration of alkali (3 %) corncob, and the yield (27.0 %) of pulp was considerably low but the quality of pulp was found to be better and could be bleached easily. Therefore, the alkali (3 %) corncob cellulose pulp concentration was chosen for superabsorbent.

Characterization of Cellulose Using FT-IR

In Figure 2, the absorption at 3332.42 cm^{-1} is assigned to stretching of OH groups. The absorption at 2895 cm^{-1} is assigned to stretching vibration of CH groups. The absorption at 1314.72 cm^{-1} is assigned to plane bending of OH groups. The peak at 1026.44 cm^{-1} indicates the C–O–C stretching of cellulose (Silverstein, 2005).

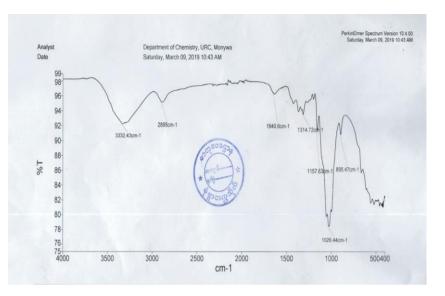


Figure 3. FTIR spectrum of corncob cellulose

Table 2. Band Assignment	t of Corncob Cellulose
--------------------------	------------------------

No.	Wave number of	*Assignment
	absorption band (cm ⁻¹)	
1	3332.42	Strong hydrogen-bonded O-H Stretching vibration
2	2895	C-H Stretching vibration of sp ³ hydrocarbon
3	1314.72	C-H in Plane bending
4	1026.44	C-O-C Stretching vibration of pyranose ring

* Silverstein (2005)

Scanning Electron Microscopy of corncob Pulp

The morphological structure including the average breadth of fibers in corncob cellulose pulp was determined using Scanning Electron Microscope (SEM). The SEM analysis of the pulps using an alkali concentration of 3% is shown in Figure 4. According to the SEM results, it was found that average breadths of fiber varied between $3.7\mu m$ to $9.4\mu m$. This indicated that the average breadth of the fiber obtained from the soda pulping process showed satisfactory conditions to be used as a cellulose backbone for the graft copolymerization process.

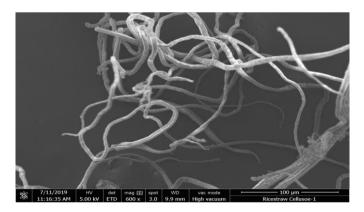


Figure 4. SEM image of corncob cellulose pulp (3% NaOH)

Characterization of Corncob Cellulose Using XRD

X-ray diffractogram of corncob cellulose was presented in Figure 5. Shows in Figure 5, it can be seen the diffraction pattern of corncob cellulose with a sharp peak at 22.3° as the intensity of crystalline. The crystalline size of cellulose calculated by Scherrer's law was 29.3 nm, which indicated nanocrystal size.

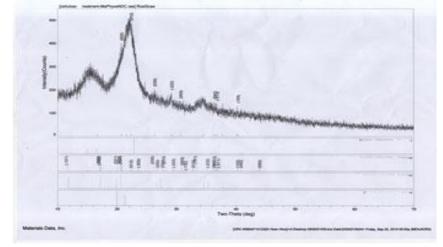


Figure 5. XRD diffractogram of corncob cellulose

Conclusion

In this research work, cellulose was produced from corncob and characterized the corncob cellulose. Corncob cellulose was prepared by soda process using different alkali concentrations (1 %, 2 % and 3 %). The experiment carried out using 3% alkali concentration at 70°C was found to be a satisfactory condition for the quality of the pulp. According to the SEM analysis of fiber in the corncob pulp sample, it was found that there was enough quality for the required properties of fiber such as paper making and graft copolymerization as a biopolymer. From FT-IR analysis, the functional groups of cellulose pulp were confirmed. The diffraction pattern of XRD indicated that cellulose was a nanocrystal with a size of 29.3nm.

Acknowledgements

The authors sincere and greatly thanks to thank Rector Dr Thar Tun Maung, Dagon University, for their kind permission to report this research paper. We would like to sincere and greatly thanks to Professor Dr Thida Kyaw (Head of Department, Department of Chemistry, Mandalar University), Professor Dr Aye Thant Zin (Department of Chemistry, Mandalar University) and Dr Hnin Hnin Than, (Professor and Head, Department of Chemistry, Dagon University, for allowing us to present this paper and we would like to thank everyone for their kind help and other facilities throughout the course of this research work.

References

- Helmiyati, AFiyriyani and f Meyanti, (2017). "The copolymerization synthesis And Swelling Capacity of Cellulose – Poly Superabsorbent (acrylic acid-Co-acrylamide) based on rice straw", IOP Publishing.
- Helmiyati, AsepSaefumillah and WinaYulianti, (2014)."Synthesis and Swelling Kinetics of Superabsorbent Rice straw Cellulose Graft Copolymer", Asian Journal of Chemistry.
- Htun Min Latt, (2014)." Preparation, Characterization and Application of Modified Polyvinyl Alcohol Hydrogel and Corn Starch Blended Films", PhD dissertation.
- Silverstein, R.M., Website, F.X and Kiemle, D.J (2005)."Spectrometric Identification of Organic Compound" New York.7th Edition, John Wiley & Sons, Inc.
- D Swantomo, Rochmadi, Basuki K.T, Sudiyo.R, (2013)."Synthesis and characterization of Graft Copolymer Rice Straw Cellulose Acrylamide Hydrogels using Gamma Radiation", Atom Indonesia.**39**, No.2, (57-64).