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Preparation of Carboxymethyl Cellulose from *Musa paradisiaca* Pseudo Stem Using an Alkaline Treatment

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ABSTRACT

Carboxymethyl cellulose (CMC) extraction from *Musa paradisiaca* (MP) pseudo stem by alkaline treatment and their properties were examined in the current research work. One of the most well-known types of lignin biomass waste that is readily available globally is MP. In many nations, including Taiwan, Sri Lanka, the Philippines, India, and the Philippines, these plants have been used for traditional reasons. Whole plant parts have been used as food, including the pseudo-stem, flower buds, trunk, fruits, and leaves. Sequestration of cellulose was attained by alkaline treatment and bleaching from raw fibers. Cellulose fiber is a biodegradable, naturally occurring, and renewable polymer that is used in a variety of industries, including the food, paper, cosmetic, and pharmaceutical sectors. The cellulose obtained from forest and agricultural residue has numerous advantages such as being environmentally safe, recyclable, and economically feasible respectively. The main process of cellulose extraction from MP pseudo stem are digesting process using a digester, bleaching, and neutralization which shows a zero-waste process. The alkali treatment takes less time to get a final product whereas enzyme treatment, and steam explosion treatment takes high energy and more cost. Hence, cellulose extract from alkaline treatment is economically feasible and environmentally friendly.

INTRODUCTION

In recent years, there is an increasing trend in the application of plant fibers and products. The main advantages of using these products from natural resources are bio-degradability and ease of handling. Deforestation of trees for the production of cellulose can be reduced when cellulose is extracted from agricultural waste. Lignocellulosic resources such as agricultural residues (straw, bark, leaves, and stems), and forest residues (hardwood and softwood) can be reused (Pires et al. 2019). The word 'lignocellulose waste' refers to the part of the crop that can not be harvested (Garcia et al. 2016). Lignocellulosic is the most available and regenerative biomass resource in nature and is underused, approximately 200 billion tonnes of lignocellulosic biomass supply from timber, forestry, industrial, and farm waste worldwide (Kocar et al. 2013). A significant amount of residual biomass (solid, liquid, and gaseous) is generated annually from the worldwide agricultural market, which should be the most available, physical, and renewable natural resource on earth (Santana-Meridas et al. 2014). Many of these residues are considered ecological liabilities unless recycled in an environmentally friendly manner (Hendriks & Zeeman 2009).

The lignocellulose waste, which primarily consists mainly of cellulose, and hemicellulose lined together and provides an enormous amount of energy resources and is an effective and regenerative supply of alternative fuels and useful chemical compounds (WeLi et al. 2015). In the biomass system, cell walls consist mainly of cellulose, hemicellulose, and lignin at a ratio of 4:3:3. This is distinct from sources like hardwood, softwood, and herbs (2014). Natural fibers have many advantages, they have low density, are recyclable, and are biodegradable. Additionally, they are sustainable raw materials and have comparatively immense strength and stiffness (Hendriks & Zeeman 2009). The natural fiber is a heterogeneous polymer network composed of cellulose, hemicellulose, and lignin (WeLi et al. 2015). In tropical and subtropical countries fibrous plants are available abundantly like bananas as an agricultural crop. Banana plants are a member of the *Musaceae* family. The edible banana species belong to the Australimusa and Eumusa groups, a member of *Musa accuminata* and *M. sapientum,* is usually a human convenient eatable banana (Mohapatra et al. 2010). The fiber of bananas is a perfect bast fiber. The chemical composition of banana fiber is cellulose, hemicellulose, and lignin, banana fiber is biodegradable and has no harmful environmental effects and can therefore be graded as eco-friendly fiber.

MP pseudo-stem is a waste product during MP cultivation. Hence it can be collected directly from the MP plants for value-added product-making like CMC (Huber et al. 2012).

Carboxyl methyl cellulose (CMC) is a water-soluble, anionic biopolymer and is one of the cellulose derivatives. Physical and chemical characteristics of CMC include hydrophilicity, pH-sensitivity, non-toxicity, and the ability to gel-forming (Siamak & Ahmad 2019). Because of the CMC's unique features, it has been used in various applications such as lithium-sulfur batteries, elimination of water contaminants, food packaging, etc. (Wang et al. 2018). Various techniques such as enzyme treatment, steam explosion treatment, and alkali treatments have been used to excerpt cellulose fibers from biomass resources (Manilal et al. 2011). The steam explosion behavior requires high pressure, and high temperature and is quite costly similarly enzyme treatment needs a long time and the activity of a low enzyme is hard for industrial production purposes. However, in alkali treatment the cellulose molecule reacts quickly with a strong base, with less energy like reactivity order is LiOH >NaOH >RbOH >CaOH.

Natural fibers are cured using chemicals to expel lignin covering resources like natural oils, waxy stuff, pectin, and so on. NaOH is a quite frequently used chemical for the ablution of the surface of plant fibers. This process is known as mercerization (WeLi et al. 2015). The standard explanation for mercerization recommended by ASTM D1695 is (ASTM 1983) "the process of subjecting a vegetable fiber to the action of a fairly concentrated aqueous solution of a strong base to produce great swelling with resultant changes in the fine structure, dimension, morphology, and mechanical properties". Zeronian proposes another definition of Mercerisation, as Mercerized cellulose is "a sample of cellulose which has been treated with a solution of an alkali metal hydroxide of sufficient strength for complete conversion" (Long Pang et al. 2019).

Cellulose is a homopolymeric, linear syndiotactic (molecular formula $C_6H_{10}O_{5n}$) that is formed by D-anhydrous glucopyranose units (AGU) joined together via □- 1,4-glycosidic bonds commonly called glucose units (Demirbas 2008). Cellulose has a high surface area since it contains well-structured bear hydroxyl groups which lead to great activity and the ability to react with different particular groups (Juntao Tang et al. 2017). Cellulose has the highest degree of polymerization (DP) among biopolymers, consisting of lignocellulosic fibers that can differ the maximum values of 20,000 units and more that depends on the source of cellulose. One of the key properties of cellulose is that it is insoluble in H_2O but absorbs about 14% of water at 60 % relative humidity at a temperature of 20°C.

After harvesting a bunch of *Musa paradisiaca* from trees over a field, vast amounts of pseudo-stem residues are left over because each *Musa paradisiaca* plant cannot be used for the next crop. These pseudo-stems are frequently discarded in soil planting to become organic waste and cause degradation of the ecosystem. Extracting fibers from these pseudo-systems can assist with natural degradation, thereby contributing to ecological equilibrium. Hence, the use of the pseudo-system *Musa paradisiaca*, which was historically called waste, would be greatly beneficial to the ecosystem and would bring additional profits. The reuse of by-products and processes with low environmental impact may be safe and an alternative novel material (Archana Das et al. 2016). Therefore, In the present study, an attempt was made to extract the cellulose from the *Musa paradisiaca* pseudo-stem which is available locally using the alkaline treatment, and to analyze the characteristics.

MATERIALS AND METHODS

Raw Materials

MP pseudo-stem was obtained after harvesting banana trees, the fruits and leaves are used for various purposes, Beater, Digester, Sodium hydroxide solution (NaOH) of 5% concentration, distilled water, and Hydrogen peroxide (H_2O_2) solution, Monochord acetic acid (MCAA) of 10% concentration and Ethanol for neutralization. The cellulose content present in different plants is listed in Table 1. From Table 1 it was inferred that the raw material chosen has approximately 50% cellulose content which is next to cellulose content in wood and cotton waste.

Methodology

The MP pseudo-stem is harvested, firstly the raw material has washed to remove impurities, and then water is sprinkled on the pseudo-stem at a higher velocity so that the impurities get washed away. Later it was chopped into small pieces with help of sharp knives as it increases the surface area

Table 1: Cellulose content present in different plants.

S. No	Raw material	Cellulose Content %
1	Cotton waste	80.95
2	Wood	80.90
3	*Musa paradisiaca* Pseudo-stem	43.0
4	Bagasse	40.0
5	Corn Stalk	35.0
6	Tomato stems	27.0
7	Coconut Shell	19.8
8	Tomato Leaves	10.9

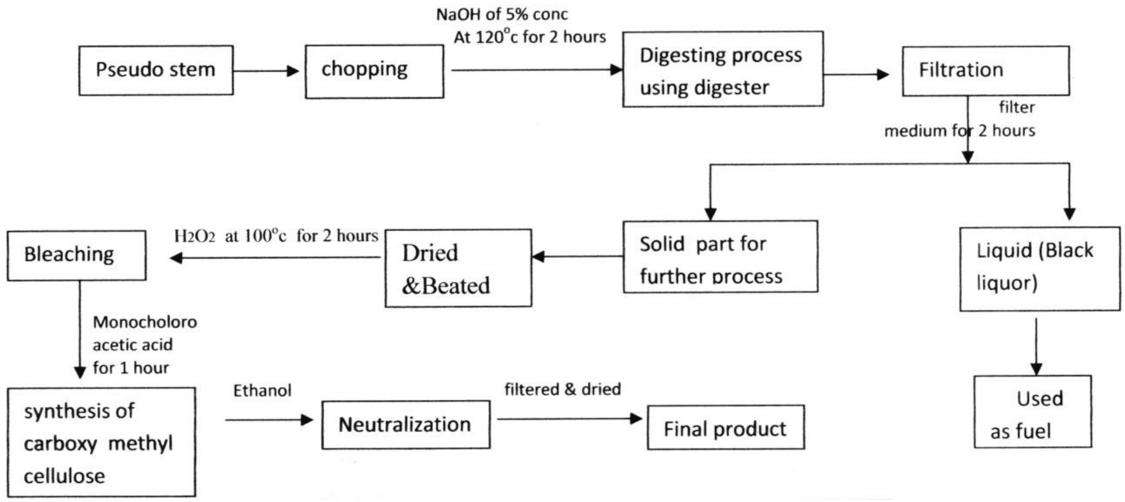

Fig. 1: Extraction of cellulose from *Musa paradisiaca* pseudo-stem.

and easier for further processing of the pseudo-stem. The chopped pieces must be of equal size it is better to use a cutting machine so that the size will be uniform. The detailed process of extracting the cellulose from the MP pseudo-stem was shown in Fig. 1. At that time chopped pieces are given as feed into a digester and 5% NaOH solution was added to eliminate the alkali-based soluble constituents like hemicellulose and other contaminations existing in the outer surface cell walls (Rani et al. 2019) i.e. for 30 g of feed 300 mL of NaOH is required feed to liquor ratio is (1:10), the temperature of the digester is maintained at 120°C and the pressure is maintained at 1.4 bar, and the process takes place for 2 hours (Tadeusz et al. 2020). Once the process is completed the outlet from the digester is taken with a mixture of digested stem and liquid obtained in black color, then the solid and liquid parts were separated by filtration process using a filter medium.

The filtrate, also known as the black liquor, was collected and used as fuel for the digester, and the solid portion kept in the filter medium was taken for additional processing. Then the obtained solid part is washed with water 2 to 3 times with water until they have become alkali-free. The washed fibers are allowed to deplete in free-flowing water. The dried part is added to potassium hydroxide and organic solvent acetone to remove lignin, hemicellulose, and other components without any loss of cellulose (Salam et al. 2007). After that, the product was dried and the solution was filtered. 90% of the lignin and hemicellulose were removed, which is a more effective removal method. The residue biomass underwent a 5-h bleach treatment at room temperature using a 2-percent H_2O_2 solution while being stirred because the residual was black. The chemical changes and fractional delignification of lignocellulosic were attained by oxidizing agents H_2O_2 to improve their properties for make use in various application purposes (Obi Reddy et al. 2017). The substance changed to white color then it was dried for 1 hour, the dried material looks similar to the cellulose obtained from normal wood processing. The bleached material was filtered using a filter medium and then dried for 30 min, later it was processed with Monochloro acetic acid (MCAA). This was the main process for cellulose fiber synthesis, which takes place for nearly one hour so that it forms a small thread-like structure as fiber and it was dried for 30 min. At last, the addition of ethanol for neutralization gives the final product after the filtration and drying process at 90°C.

RESULTS AND DISCUSSION

Chemical Analysis

The chemical composition of *Musa paradisiaca* pseudo-stem was calculated using the standards of Technical Association of the Pulp and Paper Industry (TAPPI) methods for various components, namely: T203 classical method (cm) -99 for cellulose and T 222 official method (om)-06 for lignin (Mwaikambo & Ansell 2001). The cellulose content was found to be 43% and lignin content is 18% and hemicellulose is 28% in the *Musa paradisiaca* pseudo-stem. Following the procedure, cellulose and lignin were chemically analyzed using TAPPI techniques, and for hemicellulose, three liters of ethanol were made for the filtrate produced from the processed biomass following alkaline neutralization. The precipitate produced using a vacuum pump was filtered through a glass funnel. The precipitated substance (i.e. hemicelluloses) was washed with 70 percent ethanol after

Table 2: Comparison of chemical composition in raw material and product.

Content	Cellulose %	Lignin %	Hemi cellulose%
Raw material	43	18	28
Processed	41	2	4

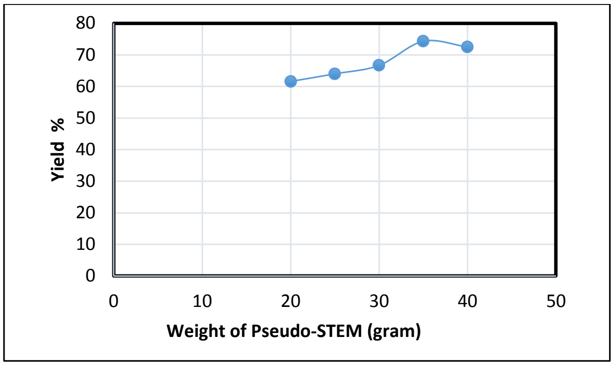

Fig. 2: Percentage yield of cellulose.

filtration and allowed to air dry (Obi Reddy et al. 2017). Table 2 shows the comparison of the chemical composition of raw material and the product.

Cellulose Yield

The cellulose fiber was extracted from the *Musa paradisiaca* pseudo-stem and the yield of cellulose content in the fiber was analyzed. The fresh banana pseudo stems moisture content was estimated to be 96%. Many trials have been carried out with different amounts of feed content, Fig. 2 shows the percentage of yield for different trials, from the graph it was found that the average yield of 60-70%.

Cellulose Confirmation Test

To confirm the presence of cellulose in the extracted fiber two confirmation tests were made - Schulze's reagent test: Schulze's reagent (Chloro-Zinc-Iodide) was added to the final product obtained after the extraction, and it turned purple color which confirms the presence of cellulose. Schulze reagent is an oxidizing mixture composed of a saturated aqueous potassium chlorate solution ($KClO_3$) and varying amounts of concentrated nitric acid (HNO_3) in the ratio of 1:1. Schulze and Tollens believed that Schulze's reagent is insoluble with hemicellulose (Rani et al. 2019). The reagents preparation technique is done by adding $ZnCl_2$ to 8.5 mL water, then dissolving followed by cooling. Lastly, we added $ZnCl_2$ solution dropwise in 20 mL of KI solution until Iodine precipitated, then proceeded to shake.

Determination of Viscosity

First, the cuprammonium reagent is prepared by dissolving solid cupric hydroxide in an ice bath with ammonium hydroxide. This approach makes storage superfluous. Cellulose is then dissolved in the reagent, and cuprous chloride and copper wire are added to prevent oxidation of air (Mwaikambo & Ansell 1999). Mechanical agitation makes the operation easier. In a redwood viscometer, the time of flow of the resulting solution was measured. Dynamic viscosity (also known as absolute viscosity) is the calculation of internal cellulose resistance to movement, whereas kinematic viscosity refers to the dynamic viscosity ratio to cellulose density. From Table 3, it was inferred that the cellulose viscosity is increased with an increase in the solution temperature indicating that the cellulose fiber extracted can withstand high temperatures.

Table 3: Effect of temperature on viscosity of cellulose.

Temperature °C	Density [kg.m^{-3}]	Time taken for 50 mL collection [sec]	Kinematic viscosity [v] m^2.s^{-1}	Dynamic viscosity[μ] Ns.m^{-2}
20	45.24	65	16.055	4.02
30	67.7	78	19.266	6.02
50	72.8	92	22.724	6.47
60	89.7	120	29.64	7.98
70	120.54	180	44.46	10.72

Production Cost

The cost for one piece of Pseudo-stem is Rs 10 of length 20 cm. A banana tree can be made into 10 pieces; hence, the cost of the main raw material is Rs 100. The cost of 1 kg of sodium hydroxide pellet is Rs 80 and the cost of 1 kg of Monocholoro acetic acid (MCAA) is Rs 90. The cost of 500 mL of Liquid ethanol is Rs 135. The overall cost of producing 1 kg of CMC is Rs 300, which is considerably less expensive than the standard method for extracting cellulose. Additionally, there are more raw materials available for CMC manufacturing than there are for wood pulp.

CONCLUSION

In the present work, CMC has been isolated and characterized from *Musa paradisiaca* pseudo-stem fiber waste. Cellulose separation from raw fibers was accomplished with alkaline treatment and bleaching, and there was no wastage in the production of CMC. Highly accessible cellulose was prepared and the deforestation of trees for the cellulose extraction from wood pulp was reduced. Results showed that the yield of cellulose from MP pseudo-stem was 60-70 %. The chemical composition of raw material and the product was analyzed by the standard Technical Association of the Pulp and Paper Associations (TAPPI) methods and the cost for the process was also compared with the general process of cellulose extraction. Alkali treatment can therefore be utilised to extract CMC from biomass resources like MP, which offer viable solutions and are inexpensive.

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