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Isolation and characterization of cellulose fibre from turmeric (*Curcuma longa* L.) leaves

Kalpana Duda, Edukondalu L, Sivala Kumar, Satyanarayana CHVV, Lakshmipathy R and Madava M

Kalpana Duda
Scientist, Department of Agricultural Engineering, Water Technology Center, Prof. Jaya Shankar Telangana State Agricultural University, Hyderabad, Telangana, India

Edukondalu L
Associate Professor, Department of Processing and Food Engineering, Dr NTR College of Agricultural Engineering, Acharya N. G. Ranga Agricultural University, Lam, Guntur, Andhra Pradesh, India

Sivala Kumar
Professor (Rtd.), Department of Processing and Food Engineering, Dr NTR College of Agricultural Engineering, Acharya N. G. Ranga Agricultural University, Lam, Guntur, Andhra Pradesh, India

Satyanarayana CHVV
Professor, Department of Food Engineering, Dr NTR College of Food Science and Technology, Acharya N. G. Ranga Agricultural University, Lam, Guntur, Andhra Pradesh, India

Lakshmipathy R
Scientist, Agricultural Research Station, Amaravathi, Acharya N. G. Ranga Agricultural University, Lam, Guntur, Andhra Pradesh, India

Madava M
Senior Scientist, Regional Agricultural Research Station, Anakapalli, Acharya N. G. Ranga Agricultural University, Lam, Guntur, Andhra Pradesh, India

Correspondence
Kalpana Duda
Scientist, Department of Agricultural Engineering, Water Technology Center, Prof. Jaya Shankar Telangana State Agricultural University, Hyderabad, Telangana, India

Abstract

Being the world's largest manufacturer of turmeric (*Curcuma longa L.)*, India produces tones of turmeric plants every year. The rhizomes are mostly used and leaves are not taken care. This research focuses on mechanical isolation of turmeric leaf cellulose fibre and its quality. The production of cellulose by photosynthesis is estimated to be 10^{11} to10^{12} t/year. Cellulose fibre became a more attractive material for commercial applications. Mechanically isolated (screw press) cellulose fibre produced from turmeric leaves on hydrolysis, delignification and homogenization resulted in a highest degree of fibrillation of 258.34 ± 1.29%. The Kappa number was found to be 15.13 ± 0.01. The chlorine bleached cellulose fibres using DSC when tested at a temperature range of 30 ˚C to 150 ˚C, no sharp melting peaks were observed. It is concluded that cellulose fibers produced from turmeric leaves by mechanical isolation were of bleachable, fibrillent and thermo-stable up to 150 ˚C.

Keywords: Turmeric leaf, cellulose fibre, Kappa number, DSC and fibrillation

1. Introduction

Turmeric (*Curcuma longa L.)*, a rhizomatous herbaceous perennial plant belonging to the ginger family *Zingiberaceae*, is native to tropical South Asia. Individual plants grow to a height of 1 m, and have long, oblong leaves. There are 13 leaves in the life of turmeric with leaf area of maximum 3302.9 cm^2/plant and having LAI of 4.95 (Li *et al.*, 1997) [5]. Being the world's largest manufacturer of turmeric, India produces tones of turmeric plants every year, the rhizomes are mostly used and leaves are not taken care.

Plant cell walls are comprised on three main components cellulose, a glucose polymer; hemi cellulose, a sugar hetropolymer and lignin, a non fermentable phenyl-propene unit. The amount of each of the three principle components can vary significantly between different biomass types and exhibit considerable variation within the same biomass type (Anonymous, 2009) [1]. The production of cellulose by photosynthesis is estimated to be 10^{11} to10^{12} t/year (Klemm *et al.*, 2004) [4]. The use of cellulose in high added value applications is still rare due to its insolubility in water and most organic solvents, hygroscopic character and no melting. Cellulose fibre became a more attractive material for commercial applications.

However, research continues focusing on optimization of the existing techniques and on development of alternative methods, which can benefit the production process (Nechyporchuk *et al.*, 2016) [6]. The crude fibre (CF) in turmeric leaves counts 134 g/kg on dry matter basis (Bhowmik *et al.*, 2008) [2].

The natural fibers can be extracted by mechanical, chemical and semi- chemical processes. Mechanical extraction gives good results in fibre characteristics This research focuses on mechanical isolation of turmeric leaf cellulose fibre and characterization. The cellulose fiber produced was analysed by Kappa number, degree of fibrillation and thermal analysis of isolated fibre.

2. Materials and Methods

2.1 Raw material

Turmeric leaves at mature and harvest stage were procured from local farmers of Jagtiyal, Telangana. The leaves were chopped to 2 cm length and macerated in a screw press, which separated water soluble fractions and cellulose fibre. The cellulose fibre so isolated was washed thrice with tap water, to remove the all adhered water soluble fractions.

Hydrolysis and delignification, was done by cooking cellulose fibres in water at 80 to 120 °C until the fibre got softened and decanted the liquid. The softened fibre was then homogenized in a blender with water and filtered through a cheese cloth, to remove water soluble fractions and the pellet was tray dried at 60 °C until constant weight was obtained.

2.2 Kappa Test

The cellulose fibre so produced was tested for Kappa number to estimate for lignin remained in based on the standard method of TAPPI- T236 om-99. The Kappa number is the volume (in milliliters) of 0.1N Potassium permanganate solution consumed by one gram of moisture-free pulp under the conditions specified in this method. The results were corrected to 50% consumption of the permanganate added (Tasman and Berzins, 1957) [8].

This method applies to the determination of the relative hardness, bleachability, or degree of delignification of pulp.

Calculated Kappa number as follows:

$$p = \frac{(b-a)N}{0.1} \text{ and } K = \frac{p \times f}{w}$$

Where:

K = Kappa number

f = factor for correction to a 50% permanganate consumption, dependent on the value of p (Table 1)

w = Weight of moisture-free pulp in the specimen, g

p = Amount of 0.1N permanganate actually consumed by the test specimen, mL

b = Amount of the thiosulfate consumed in the blank determination, mL

a = Amount of the thiosulfate consumed by the test specimen, mL

N = Normality of the thiosulfate

Factors in Table 1 are based on the equation:

$$\log K = \log p/w + 0.00093\,(p-50).$$

Table 1: Factors *f* to correct for different percentages of permanganate used

P	0	1	2	3	4	5	6	7	8	9
30	0.958	0.960	0.962	0.964	0.966	0.968	0.970	0.973	0.975	0.977
40	0.979	0.981	0.983	0.985	0.987	0.989	0.991	0.994	0.996	0.998
50	1.000	1.002	1.004	1.006	1.009	1.011	1.013	1.015	1.017	1.019
60	1.022	1.024	1.026	1.028	1.030	1.033	1.035	1.037	1.039	1.042
70	1.044									

2.3 Degree of Fibrillation

Water retention value (WRV) is an empirical measure of cellulose's capacity to hold water. During pulp disintegration, the WRV value increases due to an increase of cellulose surface area. Therefore the WRV measurements by TAPPI UM 256 standard can be used as an indication of degree of fibrillation (Nechyporchuk *et al*., 2016) [6]. WRV is a reliable measure to characterize the extent of fibrillation of micro and nanofibrils (Feng Gu *et al*., 2017) [3].

The WRV was calculated as follows;

$$\text{WRV, \%} = \frac{W_w - W_d}{W_d} \times 100$$

Where W_w is weight of wet sediment and W_d is of the dried sediment.

2.4 Differential Scanning Calorimetry (DSC) Thermal Analysis

Differential Scanning Calorimetry is a thermo-analytical technique in which the difference in the amount of heat required to increase the temperature of sample and reference is measured as a function of temperature.

DSC measures the amount of energy absorbed or released by a sample when it is heated or cooled, providing quantitative and qualitative data on endothermic (heat absorption) and exothermic (heat evolution) processes. Temperature can range from -120 °C to 725 °C, an inert atmosphere is required above 600 °C. The temperature is measured with a repeatability of ±0.1°C. DSC/TGA instrument is capable of higher temperature, maximum of 1500 °C. Pans of Aluminum (Al) were chosen to avoid reactions with sample in an atmosphere of nitrogen of 25 mL/min with a sample size from 0.2 mg to 100 mg. Melting behavior of complex organic materials, both temperatures and enthalpies of melting can be used to determine purity of a material. The thermal degradation of natural fibre occurs at three main stages with the evaporation of moisture (35 to 155 ° C). The second with hemicelluloses degradation, this occurs between 250 and 340 ° C. Finally, the third by cellulose degrade between 345 and 400 °C (Omid Nabinejad *et al.*, 2017) [7].

The hydrolysed and delignified cellulose fibre were bleached (Nechyporchuk *et al.*, 2016) [6] using commonly available chlorine bleach. The bleached and dried cellulose fibre was tested for thermal degradation using DSC.

3. Results and Discussion

3.1 Kappa Number

A high Kappa number indicate a high percentage of lignin residues in the fibre. In turmeric leaf fibre isolates, the Kappa number was found to be 15.13 ± 0.01 (Table 2).

Table 2: Turmeric leaf cellulose fibre kappa number

	R1	**R2**	**R3**	**Average**
p	59.4	59.4	59.33	
Correction for 50%	1.019	1.019	1.019	
k	15.13	15.13	15.12	15.13 ± 0.01

3.2 Degree of Fibrillation

After hydrolysis and delignification the water retention value was found to be 258.34 ± 1.29% (Table 3).

Table 3: Turmeric Leaf Cellulose Fibre Water Retention Capacity

	R1	**R2**	**R3**	**Average**
Wet sediment, g	250	250	250	
Dry sediment, g	69.8	69.5	70	
WRV,%	258.17	259.71	257.14	258.34 ± 1.29%

3.3 Differential Scanning Calorimetry (DSC) Thermal Analysis

As discussed earlier, at temperatures 30 to 150 ˚C an endothermic peak was observed and recorded an enthalpy of 1180 mJ/mg at 81.4 ˚C, the process started at 55.5 ˚C and change was observed at 125.7 ˚C (Fig 1). No sharp melting peaks were observed. Thus indicating that, the cellulose fibre was not degraded within study temperature range i.e. temperatures between 30 to 150 ˚C.

Table 4: DSC module testing conditions

Module	DSC
Sample name	leaf fibre
Sample weight, g	0.341
Reference name	Al
Reference weight, g	0.000
Gas1: 25mL/min.	Nitrogen
Pan:	Al
Temperature Program:	
Starting at	30 °C
Closing at	150 ° C
Increment level	5 °C/min.
Increment time	0.5 s

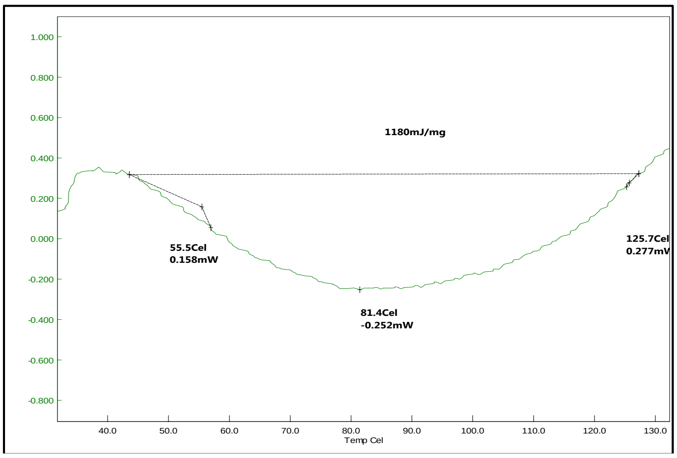

Fig 1: DSC Thermograph for Fibre Isolate from Turmeric Leaves

4. Conclusions

Mechanically isolated (screw press) cellulose fibre produced from turmeric leaves on hydrolysis, delignification and homogenization resulted in a highest degree of fibrillation of 258.34 ± 1.29%, indicating increase in specific surface area. The Kappa number of so produced cellulose fibre was found to be 15.13 ± 0.01. Thus, advocating bleachability of cellulose fibre. The chlorine bleached cellulose fibre when tested for thermal analysis using DSC, resulted in an endothermic peak at 81.4 °C. The transition process started at 55.5 °C and change was observed at 125.7 °C, the highest enthalpy was found to be 1180 mJ/mg at 81.4 °C. No sharp melting peaks were observed. Thus indicating that, the cellulose fibre was not degraded within study temperature range i.e. temperatures between 30 to 150 °C. It is concluded that cellulose fibers produced from turmeric leaves by mechanical isolation were characterized as bleachable, fibrillent and thermo-stable up to 150 °C.

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