

Notes

Microcrystalline cellulose from bagasse and rice straw

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Microcrystalline cellulose from two different locally available lignocellulosic materials, namely bagasse and rice straw has been prepared by simple process of hydrolysis. The different characteristics of prepared microcrystalline cellulose were determined and are found to be comparable with the characteristics of commercially available microcrystalline cellulose and the specifications given by Indian Standards.

Keywords: Microcrystalline cellulose, Bagasse, Rice straw

Microcrystalline cellulose represents a novel state of commercial cellulose. It is mechanically disintegrated to "level-off DP" cellulose by simple acid hydrolysis of purified alpha-cellulose. Microcrystalline flour is a very pure form of cellulose relatively free from both organic and inorganic contaminants¹.

The process of dislodging colloidal microcrystalline cellulose particles leads to the exposure of tremendous amount of new surface area². This added surface makes available powerful hydrogen bonding forces. By means of these forces microcrystalline cellulose can be utilized as powerful binder such as catalyst, in drugs etc³. Microcrystalline cellulose gels are used as emulsion stabilizers, thickeners and viscosity regulators in pastes, creams, and lotions⁴. Microcrystalline cellulose has many other applications *viz.* in cosmetics as thickener and in food industry as stabilizer, fat replacer and texturing agent. Industrial grades of microcrystalline cellulose that have a high proportion of sub-micron size colloidal microcrystals are excellent stabilizing agents for water based latex paints as well as for Industrial coating and suspensions⁵⁻⁷.

Microcrystalline cellulose could be produced from alpha cellulose obtained from wood pulp or from cotton, by hydrolysis with hydrochloric or sulphuric acid. Microcrystalline cellulose could also be produced in autoclave from ground lignocellulosic

materials by heating with steam at 205-250°C for 15 min followed by bleaching of cellulose with hydrogen peroxide and finally hydrolyzing the cellulose with mineral acid⁸⁻¹¹.

Bagasse and rice straw are easily available fibrous material produced in large quantities every year. At present these materials are utilized either as raw material for papermaking or as potential animal feed sources. In the present investigations these raw materials have been used for the production of microcrystalline cellulose by simple process of hydrolysis with hydrochloric acid. The properties of the prepared microcrystalline cellulose have been examined and compared with the standard microcellulose.

Experimental Procedure

Locally available lignocellulosic materials, such as bagasse and rice straw were collected, sorted and cleaned. Bagasse was depitched manually. Both the raw materials were air dried, grinded and the fraction passing through 40 mesh but retained over 60 mesh was selected for proximate analysis as per Tappi standard¹² and results of proximate analysis are given in Table 1.

The pulp was made from the above mentioned bagasse and rice straw materials by soda process¹³. The pulps obtained were bleached in CEH sequence. These pulps were analyzed as per Tappi standard¹².

Preparation of microcrystalline cellulose (MCC)¹⁴

Fifty grams of oven dried bagasse pulp were added to 1000 mL of 2.5 N hydrochloric acid which was preheated to 85°C. This mixture was heated for 15 min at 85°C and cooled to room temperature. Then it was stirred in a mechanical stirrer for 10 min when all the cellulosic fibers were converted into milky suspension of microcrystalline cellulose. The

Table 1—Proximate analysis of lignocellulosic materials

Parameters	Bagasse	Rice straw
Moisture (%) air dry basis	6.7	8.9
Ash (%) oven dry basis	3.2	12.1
Extractives (%)	1.8	2.2
Hot water solubility (%)	6.1	15.6
Lignin (%)	21.1	18.3

microcrystalline cellulose was filtered through Buchner funnel. It was washed with hot water till it was free from acid. The microcrystalline cellulose thus obtained from bagasse pulp was air dried and disintegrated in mixer grinder, oven dried and weighed (Yield = 90.6%). The alpha cellulose content of bagasse pulp was found to be 92.02%. Actual yield related to alpha cellulose content of bagasse pulp was = $90.6 \times 100 / 92.02 = 98.45\%$.

Following the above procedure microcrystalline cellulose was prepared from rice straw pulp and the yield was found to be 94.6%. The prepared MCCs were analyzed as per Tappi standards¹² and Indian standards¹⁵.

Results and Discussion

The cooking conditions and results of the analysis of the pulps obtained from bagasse and rice straw are given in Tables 2 and 3. A comparison of different parameters of the prepared microcrystalline cellulose and commercially available microcrystalline cellulose is given in Table 4. From Table 4, it was observed that the values of the moisture, ash, ether extractability, cold water solubility, and bulk density of the microcrystalline cellulose obtained from bagasse and rice straw and commercially available microcrystalline cellulose were comparable and were within the range prescribed by Indian Standards.

The pH of the prepared microcrystalline cellulose and the commercially available microcrystalline cellulose varied from 6 to 7 i.e., almost neutral and it was also found that starch was absent in both the prepared microcrystalline cellulose and the commercially available microcrystalline cellulose. Due to this the % assay of all the prepared microcrystalline cellulose and the commercially available microcrystalline cellulose was very high i.e.,

98%.

The brightness of microcrystalline cellulose is a measure of % of blue light reflectance and used for comparing the similar white materials. Viscosity and degree of polymerization of microcrystalline cellulose represent relative hydrolytic degradation of cellulose and it was observed that the values of the viscosity and degree of polymerization of prepared microcrystalline cellulose and the commercially available microcrystalline cellulose were also comparable and within the range prescribed by Indian Standards¹⁵.

Particle size distribution of prepared microcrystalline cellulose and the commercially available microcrystalline cellulose was determined on Fritsch Particle Sizer Analysette. This instrument directly gives % frequency and % cumulative frequency of the corresponding particle size, present in 1 g sample of microcrystalline cellulose powder.

Analysis of 1.0 g of microcrystalline cellulose

Table 2—Cooking conditions and results of pulping

Parameters	Bagasse	Rice straw
Active alkali charge as NaOH (%)	10	10
Bath ratio	1:10	1:10
Cooking time at 160°C (min)	60	45
Pulp yield (%)	56	47
Kappa number	26	24

Table 3—Characteristics of pulps

Parameters	Bagasse	Rice straw
Moisture (%), air dry basis	4.02	4.9
Ash (%) oven dry basis	0.077	1.08
Cellulose content (%)		
Alpha	92.02	91.92
Beta	5.7	4.53
Gamma	2.28	3.55
Viscosity of pulp (c.p.)	22.94	18.204
Degree of polymerization	4591.2	4007.9

Table 4—Comparison of characteristics of prepared MCC, commercially available MCC and the requirement for MCC powder as per Indian standards

Parameters pulp	Requirement for MCC as per Indian stds	Commercially available MCC	MCC obtained from Bagasse pulp	MCC obtained from Rice straw
Moisture (%)	5	4.5	4.49	4.13
Ash (%)	0.5	0.1	0.19	0.21
Ether extract (%)	3-2.09	2.59	2.2	
Cold water sol. (%)	0.2	0.14	0.106	0.18
Bulk density (g/mL)	0.25-0.5	0.26	0.3	0.27
Starch	absent	absent	absent	absent
pH	6-8	6.2	6.45	6.3
Assay (%)	95-99	99.1	98.01	98.3
Brightness (%)	Not known	70.8	72.2	68.9
Viscosity (c.p.)	Minimum 1.8	2.38	1.802	2.38
Degree of polymerization	150-700	465.6	158.4	465.6

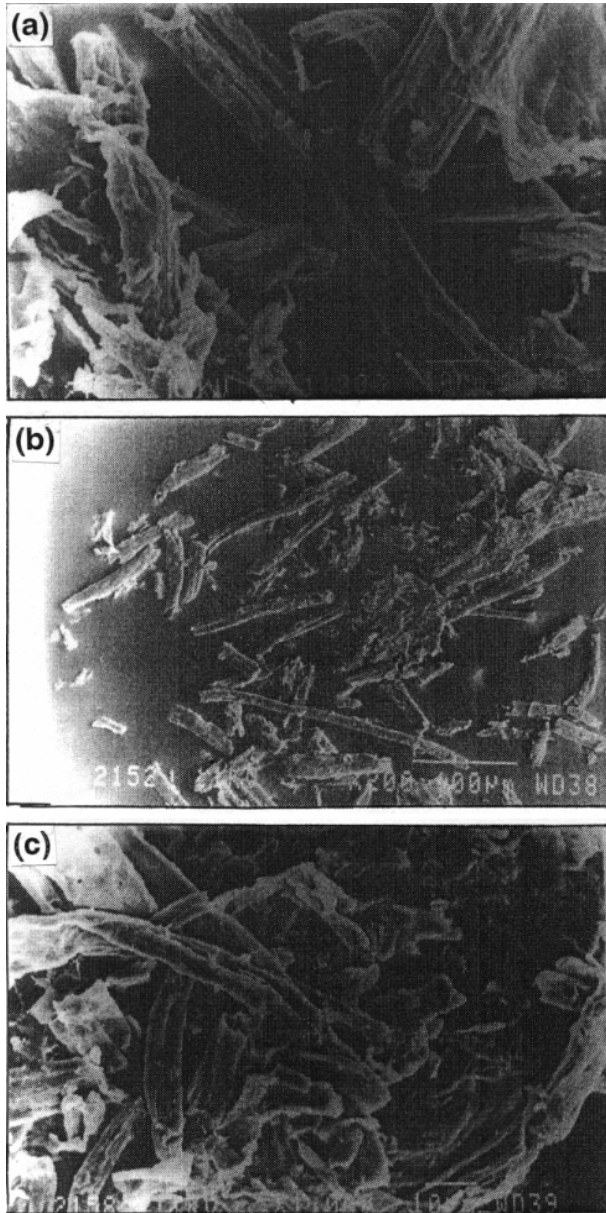


Fig. 1—Scanning Electron Micrograph of MCC obtained from (a) bagasse pulp, (b) rice straw and (c) commercial MCC

powder obtained from bagasse pulp showed that about 45% of microcrystalline cellulose had particle size of 30 microns. Similarly in 1.0 g of microcrystalline cellulose powder obtained from rice straw pulp, about 25% of microcrystalline cellulose had particles size of 33 microns, whereas 1.0 g of commercially available microcrystalline cellulose powder had about 20% microcrystalline cellulose particles of the size of 30

microns.

The scanning electron micrograph (Fig. 1) of various MCCs shows that the diameter of microcrystalline cellulose grains, obtained from bagasse pulp was 10 microns. It was true for rice straw pulp also.

Conclusion

The locally available lignocellulosic waste materials viz. bagasse and rice straw could be converted into value added product like microcrystalline cellulose by simple process of hydrolysis. Hydrolysis with 2.5 N hydrochloric acid was most suitable reaction. The characteristics of the prepared microcrystalline cellulose were comparable with characteristics of commercially available microcrystalline cellulose and the specifications given by the Indian Standards. The simple method of converting these lignocellulosic wastes to value added microcrystalline cellulose offers a possibility to exploit these materials for better use.

References

- 1 Brown R M, in *Proc 3rd Philip Morris Science Symposium, Richmond*, 1 (1978) 15.
- 2 Battista O A & Smith P A, *Ind Eng Chem*, 54 (1962) 20.
- 3 Battista O A, Coppick S, Howsmon J A, Morehead F F & Sisson W A, *Ind Eng Chem*, 48 (1956) 333.
- 4 Marianna L & Svetiana C, *Year Book, Latvian State Institute of Wood Chemistry* (1999), 40.
- 5 Panosyan I, Skurikhin I I, Sviridov A F & Kiseleva T, *Vopr Pitan*, 4 (1994) 45; *Chem Abstr*, 123 (1995) 226191 h.
- 6 Ono S & Keiko K, *Kokai Tokkyo Koho JP Pat.* 07 143 856 (1995); *Chem Abstr*, 123 (1995) 197252 w.
- 7 Janovsky A C, McCredia E J & Janet E, *US Pat.* 5 427 830 (1995); *Chem Abstr*, 123 (1995) 114360 z.
- 8 Gohel M, *Pharm Technol*, 28 (1999) 54; *Chem Abstr*, 132 (2000) 97994 g.
- 9 Mingtai M, *Comprecl*, 1 (2003) 40.
- 10 Ito Y & Msuda A, *Jpn kokai Tokkyo koho JP Pat.* 61 213 201 (1996); *Chem Abstr*, 123 (1996) 37714 r.
- 11 Aranguiz, B Teobaldo, Juan J M Victor H & Fonseca A, *Bol Soc Chil Quim*, 39 (1994) 71.
- 12 McGinnis G D & Shafizadeh F, in *Pulp and Paper Chemistry and Chemical Technology: Cellulose and Hemicellulose*, edited by J D Casey (A Wiley Interscience Pub, New York), 1951, 2.
- 13 Augsburg L & Shangraw R, *University of Nebraska-Industrial Agricultural Product Center, U.S. Pat.* 6 228 213 (2001).
- 14 *TAPPI Standards* (Tappi Press, Atlanta), 1976.
- 15 *Indian Standards*, issued by Indian Standard Institution, New Delhi, IS: 9594 (1980), IS: 9598 (1980) and IS: 6213 part IV (1971).