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Synthesis and characterization of microcrystalline cellulose powder from corn husk fibre using bio-chemical route

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Abstract
In the present study low cost microcrystalline cellulose (MCC) powder was prepared from cornhusk fibres, extracted chemically followed by anaerobic consortium treatment. Cornhusk fibres were treated with 10% alkali at 120 for 60 min followed by anaerobic consortium treatment for 3 days. It was then bleached with hydrogen peroxide and finally washed. Bleached pulp was hydrolysed using 4 N HCl to get the MCC. In the present investigation, we have characterized the MCC prepared from cornhusk fibres thoroughly for its physico-chemical properties and compared with Avicel-PH 101, a commercial grade MCC. Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Powder Diffraction (XRD) and Differential Scanning Calorimetry (DSC) were used for characterization of samples. Similarly the powder and flow properties of the MCC prepared from cornhusk fibres were also investigated and the results were compared with Avicel-PH 101. Our results showed that various properties and the purity of MCC prepared from Cornhusk fibres are comparable to the commercial grade MCC. Since, cornhusk is an agricultural waste product, MCC obtained from cornhusk fibres will be from cheaper raw materials than current market MCC.

Keywords: Cornhusk fibres, Extraction, Anaerobic consortium treatment, microcrystalline cellulose, Physico-chemical and flow properties

1. Introduction

In the present scenario, researchers from all over the world are focusing on utilization of agro wastes generated from food and fibre crops for various industrial applications. Corn (Zea mays) is from Graminacea family referred as Miracle crop and Queen of the Cereals. It is the third most important cereal crop after rice and wheat in the world. The cultivation of maize is continuously increasing due to its diversified usage in sectors like food, feed and ethanol production. In India, on an average 24.5 million tons of cornhusk are produced annually. Cornhusk is mainly used as animal feed and for decorations in natural crafts. It is also used for wrapping different types of seafood, such as fish, prawns and crabs for its easy roasting. Inspite ofall these uses, substantial quantities of cornhusk are being incinerated. Instead of burning, the left over cornhusk can be used for synthesis of MCC, as cornhusk is rich in cellulose content (Reddy and Yang 2005; Kambli *et al*. 2016) [1]. Microcrystalline cellulose (MCC) is partially purified de-polymerized crystalline cellulose having potential industrial applications. Cellulose, a biopolymer is the basic and major constituent of plants. It is the most abundant polymer in nature. The basic method for preparing MCC from purified pulps was first described in Battista and Smith (1962) [17] (US. Pat. No. 2978446) which still represents the basis for many conventional MCC manufacturing processes. For over 50 years MCC is prepared from available cellulosic material or pulp by acid hydrolysis. Commercially available MCC is mostly derived from wood pulp and also cotton based materials by treating with acids like hydrochloric acid (HCl) (Brittain *et al*. 1993) [16]. In wellknown commercial process for making MCC by using partial acid hydrolysis of purified cellulose, at which only the amorphous areas of the polysaccharides are hydrolysed, dissolved and removed. The crystalline cellulose areas are not hydrolysed and are recovered. The quality MCC has high demands in the field of cosmetics, food and pharmaceutical industries because it can be used as good suspension stabilizer and a reinforcing agent for final products such as medical tablets and capsules (Ruan *et al*. 1996; Laka and Chernyavskaya 2007) [3]. Due to its low cost, MCC prepared from cornhusk can be a potential excipient in the preparation of pharmaceutical dosage forms (Vora and Shah 2015). MCC is used as bulking agent and fat substitute in food products such as baked foods, dairy products, desserts, frozen foods etc. It helps in enhancing mouthfeel, texture and consistency of the food product. In pharmaceutical industry, use of MCC is demandableas it exhibits chemical inertness and has no taste and odor. MCC is used an excipient in almost every kind of oral dosage likepellets, tablets, capsules, sachets and others. Commercially available MCC is derived from costly wood pulp and also purified cotton. The need for cheaper sources of MCC has led to the investigation of other lignocellulosic materials based on different agricultural residues (Suesal and Suwanruji 2011). Many R&D reports are also available for the preparation of MCC using other methods such as radiation degradation with deposited electron energy, reactive extrusion, two-step radiation-enzymatic depolymerisation process, etc. (Stupinska *et al*. 2006 [12], 2007; US patent No. US6228213 B1 2001). It is generally understood that the differences in the properties of MCC between different manufacturers are due to the type of pulp used as raw material, and processing conditions. Since cellulose from different sources has different properties, for example

crystallinity, moisture content, surface area and porous structure, molecular weight, and so on. It is expected that MCC obtained from different sources would also have different properties. The combination of anaerobic method with chemical for preparation of MCC will reduce the cost and also help in reducing the toxic effluents released in environment (Shaikh 2000). In the present study, cornhusk fibres were subjected to chemical and anaerobic consortium treatment to prepare MCC. Also, we compared the properties of the prepared MCC with commercial grade MCC.

1.1 Material and Method

For the fibre extraction the required cornhusks (maize cob sheaths) were collected from local market, Mumbai, India. The peeled cornhusks from maize cobs were manually cleaned to remove foreign materials. Both the ends of the cornhusks were removed and air dried. These air dried cornhusks were chemically treated to extract fibres. For chemical extraction of fibres, sodium hydroxide (98%, Merck Specialties Private Limited, India) was used. Avicel01 (a commercial brand of microcrystalline cellulose) was used for the comparison of prepared MCC. Hydrochloric acid (HCl) (35%, Fishers Scientific, India) was used for acid hydrolysis. CIRCOT, India, anaerobic consortium was used for anaerobic consortium treatment in the preparation of MCC.

1.2 Procedure for fibre extraction

The cornhusk fibres were extracted at 120 for 60 min by using 10% w/w concentration of sodium hydroxide. Fifty g of corn husk was taken in one liter of alkali solution with the material to liquor ratio 1:20. The extracted fibres were then neutralized with dilute acetic acid 0.1% (w/v) and air dried. The chemical composition of cornhusk fibres used in the present study is reported in our previous work (Kambli *et al*.Cellulose1232016) [1]. The fibre contain 7.5% lignin, 50–55% cellulose and 39.39% hemicellulose. Procedure for the preparation of microcrystalline cellulose powder The anaerobic consortium was prepared by adding 4% (w/v) papaya pulp, 2.5% (w/v) paste of spinach leaves to 40% (w/v) cow dung slurry in 5 L glass bottle. The anaerobic condition was created by sealing the top of glass bottle with rubber cork and the gas outlet was connected inside water. The extracted corn husk fibres were cut into 2 cm size and 1 kg of these fibres was transferred to anaerobic digester containing 4 L of 30 days old anaerobic consortium. Thus, the material to liquor ratio was 1:4. The digester was kept under anaerobic condition for 3 days. The pulp obtained after 3 days was washed thoroughly. This pulp was bleached at 85 for 60 min using hydrogen peroxide (0.3% w/v), sodium hydroxide (0.1% w/v) and sodium silicate (0.15% w/v) as a stabilizer with material to liquor ratio 1:20. The bleached sample was acid hydrolysed by using 2.5 N HCl (1:10 material to liquor ratio). Hydrolysis was done by passing steam through the material for 20 min, then hydrolysed pulp was cooled and finally washed thoroughly with water till it was free of acid (pH 6.5–7.0). The material was dried in incubator at 37 for 30 h then pulverized (0.053 mm mesh size) to obtain MCC powder.

2. Physico-Chemical Characterization of mcc

The physico-chemical properties of MCC prepared from cornhusk fibres were compared with commercial grade MCC (Avicel-PH 101).

2.1. Determination of moisture content

In a tared weighing bottle, 2 g of sample (A) was taken. The weighing bottles carrying the sample was kept in oven at 105 for drying. After drying, it was cooled in desiccator and weighed accurately until a constant weight (B) was obtained. The following formula was used to calculate moisture content.

2.2. Determination of ash content

By using ASTM (E 1755-01) test method, the ash content in the sample was determined. The sample (5 g) was placed in a crucible and accurately weighed. The sample was carbonized by igniting over the Bunsen flame. After carbonization, the crucible was transferred to a muffle furnace along with the sample. Ashing was done at 575 ± 25 till it attained a constant weight. The ash content was calculated based on dry weight basis.

2.3. Determination of cellulose content

Halliwel method (Halliwell 1959) was used to estimate the cellulose content in the MCC. About 2 g of the samples in duplicates were extracted for 6 h withetherina soxhlet assembly.For1 hthese defatted samples were boiled in 0.5% ammonium oxalate in the ratio of 1:50 (material to liquor ratio) to remove water soluble matter and pectin. To remove lignin, samples were bleached with 1% sodium chlorite solution and 0.05 Naceticacidintheratioof1:50inaboilingwater bath for 1 h. The obtained holocellulose was then treated with 5% potassium hydroxide (KOH) initially for 2 h which was then again treated with 24% KOH for 2 h to remove hemicellulose. The resultant cellulose was stirred with a 20 fold volume of phosphoric acid (90%) for2 hat1 washed with1% solution of sodium carbonate and finally with water. The residue was completely dried in incubator at 37 and weighed to constant weight. Thus the actual weight of pure cellulose was obtained by subtracting the ash content from the residual weight.

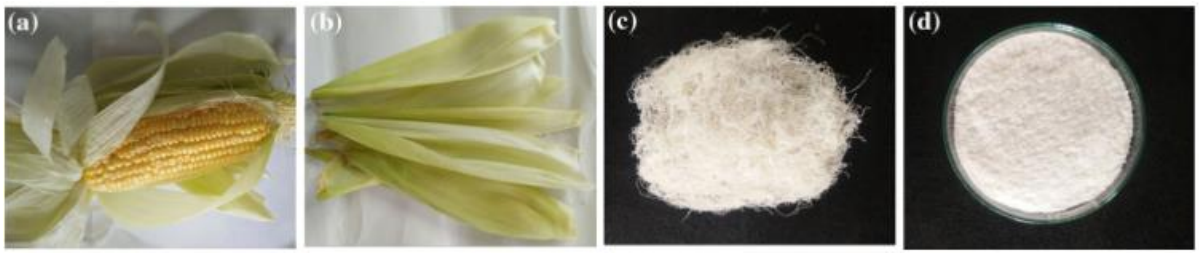

Fig 1: a Corn, b cornhusk, c chemically extracted cornhusk fibres and d MCC from cornhusk fibres

2.4 Sem analysis

The study of SEM micrographs for cornhusk MCC and commercial MCC are presented in Fig. 6a, b respectively. It can be observed from the SEM micrographs
that cornhusk MCC surface is plain and uniform in size as compared to commercial MCC. Whereas commercial MCC shows a mixture of plated shaped, complex and ribbon like structure.

2.5. Thermal property

Both the curves are showing initial endothermic peak at around 60–100 ℃, due to the evaporation of the moisture. Next endothermic peak observed at around 350 ℃ is responsible for the pyrolysis of cellulose. Indeed, this endothermic peak is responsible for the heat absorption by the cellulose polymer, depolymerisation of the polymer and the production of the flammable gases. Only difference is, commercial grade MCC is showing pyrolysis endotherm at 20–25 ℃ lower temperature than the other one. It may be due to the difference of the maturity or variety/grade of the cotton fibre which affect the fineness of the cotton fibre. However, as per conclusion.

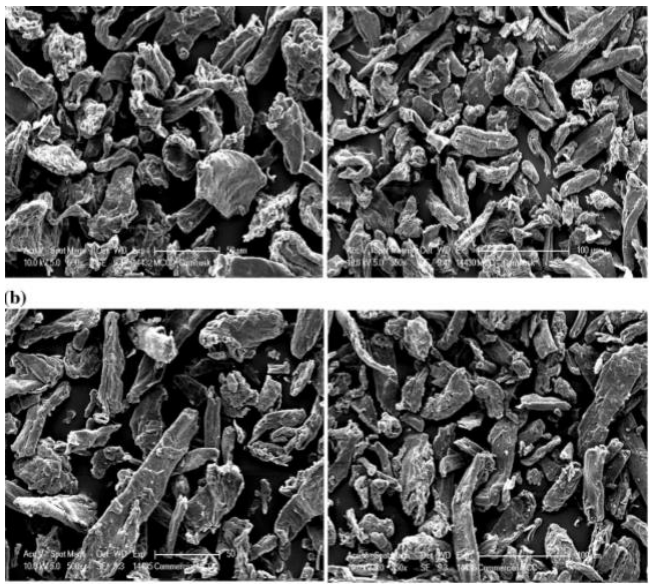

Fig 2: Sem micrographs of a mcc prepared from cornhusk fibres, b commercial mcc

3. Conclusion

Cornhusk fibres are rich in cellulose and annually available agro residue waste and thus can be a potential raw material for MCC production. The present study revealed that MCC prepared by chemical and biological treatment yielded cellulose content of 98.2%. The physico-chemical characterization studies showed that the quality of MCC prepared from cornhusk is at par with commercial grade MCC and also meets the national and international standards. To conclude, the present study reveals a high quality MCC could be prepared from cornhusk by following bio-chemical route and thus the use of environmentally toxic chemicals would be reduced. Further work may be carried out on development of a green technology for MCC preparation with very minimum chemical usage.

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