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Cellulose Ether From Chambal Wood

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Abstract

Saw dust of Chambal Wood *(artocarpus chaplasha)*, a cellulosic waste, was successfully utilized for the preparation of sodium carboxymethylcellulose (Na-CMC), a water-soluble cellulose ether. Saw dust was analyzed for its moisture, ash, alcohol-benzene soluble substances, lignin and alpha-cellulose contents. Alpha-cellulose fraction was isolated and subsequently carboxymethylated to Na-CMC. Water-soluble product was purified and degree of substitution value was determined. Multistage carboxymethylation was performed to get highly substituted Na-CMC.

INTRODUCTION

The demand of sodium carboxymethylcellulose is increasing day by day in both cellulosic and non-cellulosic industries due to its outstanding characteristics. The basic steps involved in the preparation of Na-CMC remained unaltered since its original study by Jansen[1], Chowdhury[2] and Hoppler[3]. It was used mainly to substitute naturally occuring gums and as a synthetic detergent aid[4]. Its other uses include its application in textiles, paints, foods, drilling muds, paper, as a thickening agent, tablet binder, protective colloid, coagulating agent, film former etc.

Na-CMC was usually prepared in aqueous medium. In this laboratory an excellent method [5] was developed by simplifying the entire process of carboxymethylation, where water-ethanol was used as reaction medium.

Now a days, cellulose waste materials are used as a source of cellulose. Saw dust might be considered as a cheap locally available cellulosic raw material. In the present work, saw dust of Chambal wood (artocarpus chaplasha) was used as the source of cellulose from which Na-CMC was prepared by carboxymethylation in both aqueous and non-aqueous medium. For higher substitution, multistage carboxymethylation[6] was carried out.

EXPERIMENTAL

Saw dust of artocarpus chaplasha (Chambal wood) was collected from a local saw mill. This wood sample (40-60 mesh) was analyzed for its moisture, ash[7], alcohol-benzene soluble substances[4], lignin[9] and alpha-cellulose contents[10]. Alpha-cellulose was isolated, dried and kept in a desiccator. Analytical grade of crystalline monochloroacetic acid and sodium hydroxide of E. Merck (West Germany), and absolute alcohol, prepared by distillation over calcium oxide, were used in the experiments.

Carboxymethylation in aqueous medium:

5g of alpha-cellulose was steeped in 25ml. of 70% aqueous NaOH solution and the whole mass was thoughly mixed, covered and kept in a thermostat at 28-30°C. Steeping was continued for two hours with occasional stirring. Then 10ml. of 80% aqueous monochloroacetic acid was added dropwise to the alkali cellulose with vigorous stirring. Carboxymethylation was then continued for 6 hours; first 3 hours at 30-35°C. and the remaining 3 hours at 40-45°C. At the end of reaction period, crude Na-CMC was washed three times with 80% ethanol.

Purification: Crude Na-CMC was first dissolved in water to make a 2% aqueous solution and then 95% ethanol was added into it with thorough stirring to precipitate out fibrous Na-CMC. Precipitated Na-CMC was filtered, washed with 95% ethanol and dried in vacuum. The degree of substitution (D.S.) of Na-CMC was determined by Copper precipitation method[11]. Results are given in Table 1.

Carboxymethylation in aqueous-ethnolic medium: To prepare Na-CMC in aqueous-ethanolic medium, 5g. of alpha-cellulose of saw dust was steeped in 18% aqueous-ethanolic NaOH (1:6 by volume) solution at 28-30°C. for 2 hours. 10ml. of 80% ethanolic monochloroacetic acid solution was then added dropwise into the alkali cellulose and carboxymethylation was carried out for 6 hours; first 3 hours at 30-35°C. and the remaining 3 hours at 40-45°C. Crude Na-CMC was then washed thoroughly with 80% ethanol and dissolved in water to make a 2% solution. Pure fibrous Na-CMC was precipitated out by using 95% ethanol, filtered, dried in vacuum and kept in a desiccator. D.S. value was determined by Copper precipitation method. Results are given in Table-2.

Multistage carboxymethylation:

Single stage carboxymethylation always gives a low or partially substituted product. For higher substitution, multistage carboxymethylation was essential. 5g. of alpha-cellulose was steeped with 18% aqueous-ethanolic NaOH (1:6 by volume) solution for 2 hours at 28-30°C. Carboxymethylation was carried out by using 80% ethanolic monochloroacetic acid under the same conditions of time and temperature. The product (Na-CMC) was washed, purified and analyzed accordingly. To perform second stage Carboxymethylation, Na-CMC of first stage was again treated with 18% NaOH and subsequently carboxymethylated to second stage Na-CMC. Similarly third, fourth and fifth stages of carboxymethylation were carried out. D.S. values of all the products were determined accordingly and results were given in Table-3 and plotted in Fig. 1.

DISCUSSIONS

Results of the analysis of Chambal wood was given in Table 1. Ash, alcohol-benzene soluble substances and lignin contents were 1.58%, 4.78% and 31.59% respectively: Alpha-cellulose content was found to be 53.92%

Table 2 shows that alpha-cellulose of the saw dust of Chambal wood could successfully be utilized for carboxymethylation both in aqueous and aqueous-ethanolic medium. In both the cases crude Na-CMC contained some water- insoluble fibres. Considering the heterogeneous nature of the reactions, these water-insoluble fibres might be considered as unreacted or very low-substituted fibres. However, in both the cases, purified Na-CMC samples were soluble in water and insoluble in 80% ethanol. The values of D.S. of Na-CMC prepared in aqueous and aqueous-ethanolic medium were found to be 0.91 and 1.04 respectively. Better substitution was obtained in aqueous-ethanolic medium.

Highly substituted Na-CMC could not be prepared by single stage carboxymethylation. Higher substitution could only be achieved by performing multistage carboxymethylation. For multistage carboxymethylation, we preferred water-ethanol as the reaction medium, as it was observed earlier that better D.S. values could be obtained by single stage carboxymethylation only in aqueous medium. Moreover, low NaOH concentration (18%) could be used in aqueous-ethanolic medium.

Results of multistage carboxymethylation are given in Table 3. Multistage carboxymethylation was performed upto fifth stage. D.S. values of Na-CMC increased with the number of stages of carboxymethylation. All the products were soluble in water. Crude products could easily be purified by using 95% ethanol. Washing and purification operations were also done with 95% ethanol.

In fig. 1 the variation of degree of substitution of Na-CMC with the number of stages of carboxymethylation was plotted. Values of the D.S. of Na-CMC were found to increase with the number of stages of carboxymethylation and in the fifth stage Na-CMC was highly substituted (2.74). At higher stages, however, rate of increase of D.S. of Na-CMC decreased slightly. Yields of products were in good agreement with the theoretical value. A flow sheet for the manufacturing process of Na-CMC was also given.

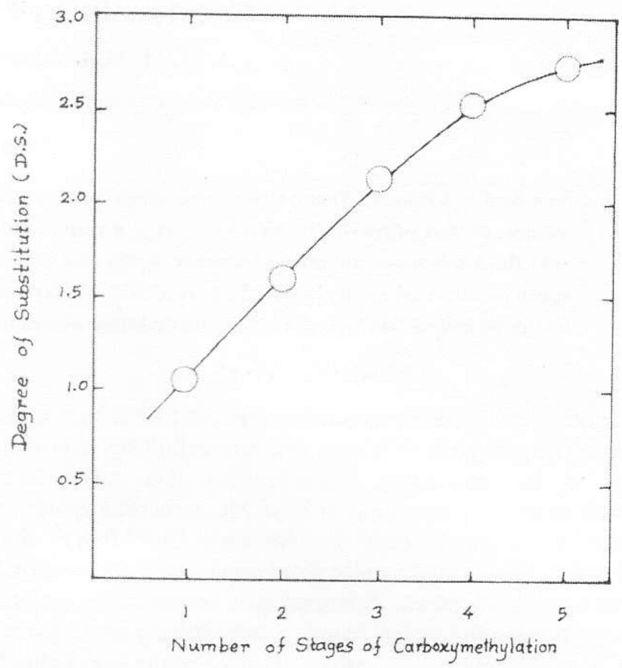

Fig. 1: Multistage Carboxymethylation of Sow dust.

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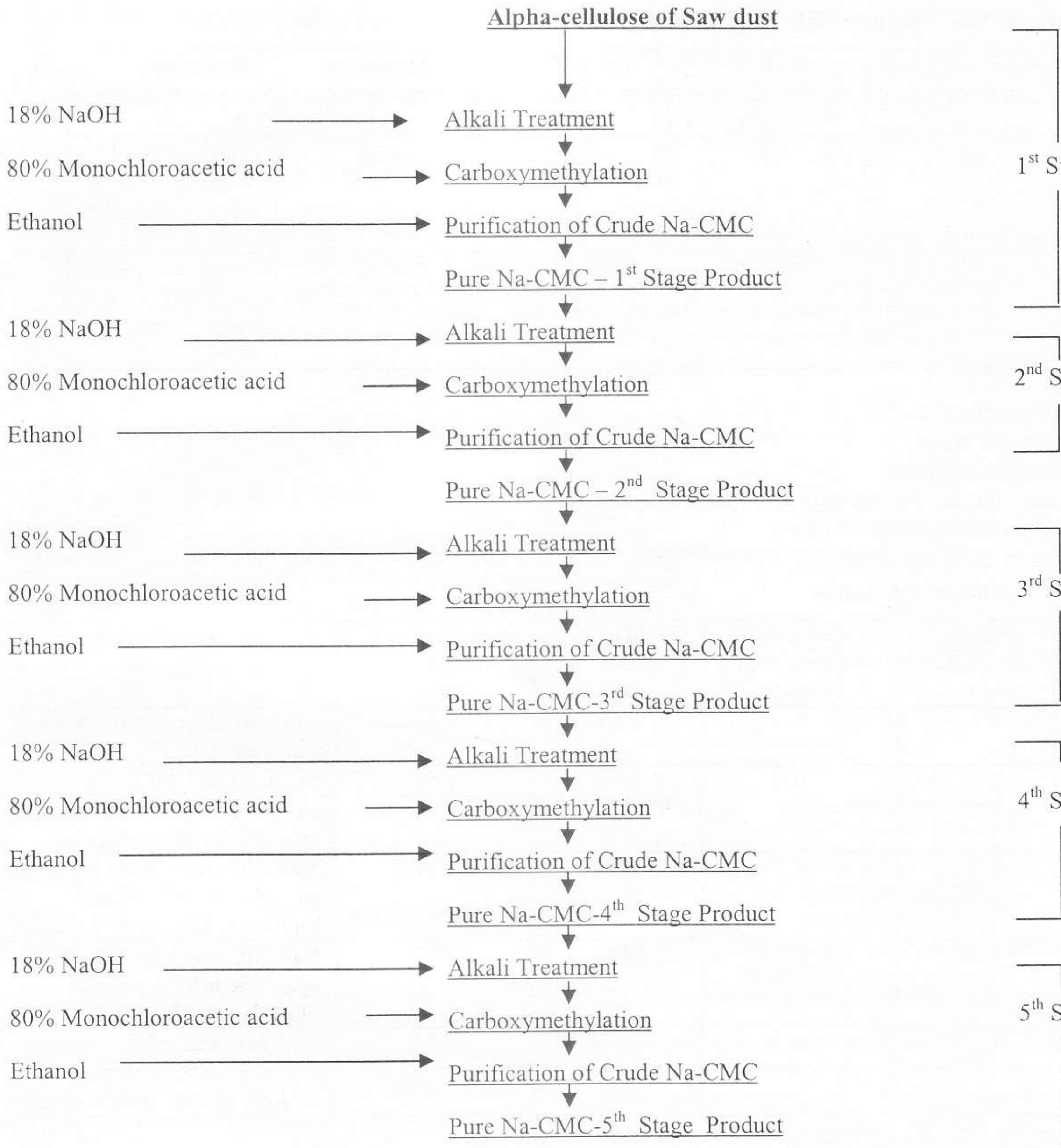

Flow Sheet for the manufacture of Na-CMC

Results

Table 1: Analysis of saw dust

Ash Content	1.58%
Alcohol-Benzene soluble substances content	4.78%
Lignin Content	31.59%
Alpha cellulose content	53.92%

Table 2: Preparation of Na-CMC by Single Stage Carboxymethylation.

Reaction Medium	Water –cellulose ratio (v/w)	Ethanol-cellulose ratio (v/w)	Yield of Na-CMC g.	Degree of substitution (D.S.)	Observation
Aqueous	1	6	7.79	0.91	Crude product contained some water insoluble fibers.
Aqueous - Ethanolic	1	6	8.17	1.04	Crude product contained some water insoluble fibers.

Cellulose take = 5g.
Conditions for alkali treatment:
Steeping time = 2 hours.
Conditions for Carboxymethylation:
Concentration of monochloroacetic acid= 80% ethanolic solution.
Time of carboxymethylation : 6 hours.
Temperature of carboxymethylation = 30-35°C for the first three hours and 40-45°C for the remaining three hours.

Table 3: Multistage Carboxymethylation

Number of stages	Water – cellulose ratio (v/w)	Ethanol-cellulose ratio (v/w)	Yield of Na-CMC g.	Degree of substitution (D.S.)	Observation
1	1	6	8.17	1.04	Crude product contained some water insoluble fibers.
2	1	6	9.96	1.58	Product (Na-CMC) was completely soluble in water and insoluble in 80% ethanol
3	1	6	11.02	2.11	Na-CMC was soluble in water, ; insoluble in 95% ethanol; Slightly soluble in 80% ethanol
4	1	6	11.65	2.52	Na-CMC was soluble in water, ; insoluble in 95% ethanol; Slightly soluble in 80% ethanol
5	1	6	12.74	2.74	Na-CMC was soluble in water, ; insoluble in 95% ethanol; Slightly soluble in 80% ethanol

Cellulose take = 5g
Conditions for alkali treatment:
Concentration of NaOH = 18% aqueous-ethanolic solution.
Steeping time = 2 hours

Conditions for Carboxymethylation:
Concentration of monochloroacetic acid= 80% ethanolic solution.
Time of carboxymethylation : 6 hours
Temperature of carboxymethylation = 30-35°C for the first three hours and 40-45°C for the remaining three hours.