Cellulose Derivatives Part III Na-C.M.C. From Waste Cellulosic Materials*

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Further investigations on the preparation of Na-C. M. C. from waste cellulosic materials were continued. Cellulose from five different sources viz. nitric pulp from fir saw dust 2. rags 3. white writing paper 4. cotton waste and 5. used cyclostyled waste paper was tried. The materials after suitable treatment were converted into Na-C. M. C. by the method reported earlier. The characteristics of the Na-C.M.C. obtained from these sources were studied. The maximum active content of 76.6% was obtained from fir sawdust nitric pulp and the minimum 54.0% from used cyclostyled waste paper having 2% aqueous solution viscosities of 49 cps and 35 cps respectively. The degree of substitution of the Na-C. M. C. from these sources vary from 0.59-0.72. The relationship of active content and the viscosity is shown in the figure. The Na-C. M. C. prepared from these sources have satisfactory gum power.

Introduction

In our earlier communication, we have reported the results of our investigations on the preparation of cellulose derivatives viz. Na-C.M.C. from waste paper. The paper reported the modification and treatments imparted to different wastes prior to the reaction with monochloroacetic acid. The study also iucluded the effect of different reaction timings on the degree of substitution. Continuing the work futher, some more wastes for making Na-C.M.C. derivatives were studied. The present study deals with materials such as saw dust pulp, rags, used cyclostyled waste paper, cotton waste (avilable during the manufacture of absorbent cotton wool I. P.) and white writing paper. One of our earlier communications also reported the results of the studies carried out for graft co-polymerisation with MMA using high alpha pulp. Grafting to the extent of 74.0% was obtained under nitrogen atmosphere and using ceric ion redox system.

Materials

The starting materials used for the present studies were (1) mildly bleached nitric pulp from fir saw dust (ii) Rags (iii) White writing paper (iv) cotton waste (avilable during the manufacture of absorbent cotton wool (I. P.) & (v) used cyclostyled waste paper.

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Pretreatment of Waste

I. **Fir Saw dust :** This is a waste material available in abundance. The pulp was obtained by nitric acid pulping process by treating the saw dust with 30% nitric acid (W/W) and allowing it to reflux for 3 hours. After this nitric acid was decanted, water added and then 2% sodium hydroxide was added to adjust the pH at about 9. Th's was refluxed again for one hour after which the material was washed till alkali free. The pulp thus obtained was given a mild bleaching treatment with $NaClO_2/CH_3COOH$ followed by extensive washing with water. The pulp material was dried and used as another starting material for Na-C. M. C. preparation.

II. **Rags :** These were cut into small pieces followed by shredding and then treated with 2% sodium hydroxide solution (material) liquor ratio 1:10) and kept overnight. Thereafter it was thoroughly washed with water over 40–60 mesh sieve to remove the alkali. It was then given a bleaching treatment with 1% sodium hypochlorite at pH of about 8.5 for one hour at 3% consistency. The material afterwards was washed extensively with water till free from hypochlorite and alkali and then dried and yield noted. This formed the starting material for the preparation of sodium-carboxymethyl cellulose.

III. **Ordinary white writing paper :** These were torn into short pieces and were immersed in a 2% sodium hydroxide solution overnight. Next day, it was shredded in wet form to get pulp, washed extensively with water over 40–60 mesh sieves. To

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this pulp, a bleaching treatment with 1% sodium hypochlorite solution was given at an adjusted pH of about 9 for three hours. The pulp afterwards was washed thoroughly with water till free from alkali and hpyochlorite. It was then dried and used for the preparation of Na-C. M. C.

IV. **Cotton waste :** These were simply boiled for three hours in excess of water, then dried, weighed and used as the starting material for the preparation of Na-C. M. C.

V. **Used Cyclostyled waste papers :** These were shredded and boiled with 2% alkali in a beaker for 2½ hours over water bath. The material was then cooled, filtered, and washed extensively with water till alkali free. The pulp was dried and used as the starting material in the present paper, for the preparation of Na-C. M. C.

2. 40% sodium hydroxide treatment of the above materials

The method followed was exactly similar to the one described in Part I of our earlier communication.

3. Preparation of sodium carboxymethyl cellulose

The procedure followed is exactly similar to the one described in our Patr I communication.

4. Detailed analysis of sodium carboxymethyl celluloses obtained from various waste materials

Various characteristic analytical properties like (i) Moisture content (ii) solubility in water (iii) gum power (iv) Refractive index (v) Specific gravity (vi) Active content (vii) Degree of substitution (viii) Amount of sodium chloride in the ash of active content (ix) Amount of sodium chloride in Na-C. M. C. (x) Viscosity (xi) Intrinsic viscosity and (xii) degree of polymerisation were all determined exactly by the same procedures as described in Part I of our communication, earlier.

Other chemical constituents like pentosans, alphacellulose ash and alcohol-benzene solubles of the materials were estimated according to Tappi-procedures.

Results & Discussion

The result of the pentosan content, alpha, cellulose, extractives and ash content of the materials are given in Table I. As can be seen from the results that the ash content of cyclostyled waste paper, white writing paper and cotton waste ranges between 10.3-12.60, probably due to the presence of fillers. Treatment with 40% NaOH for 45 minutes markedly reduced the ash content, extractives pentosans as is evident from Table II, while the alpha cellulose increa-

TABLE-I

Analysis of waste Materials

S. No.	Chemical constituents	1	2	3	4	5
1.	Moisture %	8.10	7.00	15.20	8.40	8.00
2.	Ash %	—	1.00	10.97	10.33	12.60
3.	Alcohol-Benzene (1:2 by vol.) Extract %	—	0.70	3.60	0.92	3.45
4.	Alphacellulose %	—	97.90	68.37	97.50	70.60
5.	Pentosans %	—	0.15	18.10	0.20	14.85

1. Fir saw dust pulp; 2. Rags; 3. White writing paper;
4. Cotton waste and 5. Used cyclostyled waste paper.

TABLE-II

Analysis of waste after 40% sodium hydroxide solution treatment for 45 minutes

S. No.	Chemical analysis of various constituents	1	2	3	4	5
1.	Yield %	90.00	97.00	85.00	90.00	95.00
2.	Ash %	—	0.89	4.00	6.92	5.00
3.	Alcohol-Benzene (1:2 by vol.) extract %	—	0.60	1.50	0.76	1.30
4.	Alphacellulose %	—	98.02	88.33	98.00	88.33
5.	Pentosans %	—	0.10	5.62	0.15	4.62

1. Fir sawdust pulp; 2. Rags; 3. White writing paper;
4. Cotton waste and 5. Used cyclostyled waste paper

sed. Characteristic properties of Na--C. M. C. prepared under the conditions are reported in Table III. The active content was minimum 54% in the case of waste cyclostyled paper while a maximum of 76.6% was obtained from the fir saw dust nitric pulp. Degree of substitution varied between 0.59–0.72. Viscosity of 2% aqueous solution ranged between 38.0–53.8 which is, comparatively, lower than the commercial grade. Value of 58.6 with D. S. of 0.76. All the grades of Na–C. M. C. prepared from different wastes gave a satisfactory gum power.

The relationship between the viscosity and active content is shown in figure 1. It is evident that the viscosity of 2% aqueous solution increased with the active content. The active content obtained under similar conditions of reaction with different materials are of the order of Fir Saw Dust pulp > rags > white writing paper > cotton waste > used cyclostyled waste paper (Figure 2).

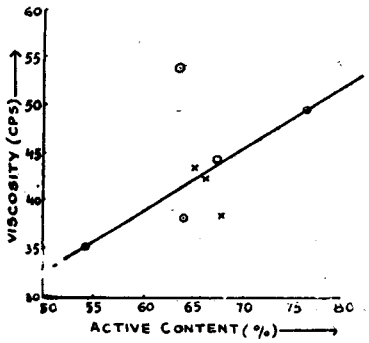

Fig. 1 Relation between active content and viscosity of various Na—C. M. C. Samples.

TABLE–III
Characteristics of sodium carboxymethyl cellulose

S.No.	Detailed analysis of	1	2	3	4	5
1.	Moisture %	9.41	16.66	12.40	11.80	18.84
2.	Ash %	12.60	9.00	18.20	24.60	19.20
3.	Yield %	125.00	130.00	135.00	134.00	140.00
4.	pH (2% aq. sol.)	9.00	7.00–7.50	7.5–8.00	8.00	9.00
5.	Refractive Index (2% aq. sol)	1.333	1.334	1.333	1.335	1.334
6.	Specific gravity (2% aq. sol)	1.040	1.038	1.040	1.041	1.040
7.	Active content %	76.60	67.20	64.10	63.80	54.00
8.	Degree of substitution D.S.	0.72	0.62	0.59	0.70	0.63
9.	NaCl Amount % (in Na-CMC samples)	9.45	7.00	10.81	14.35	12.35
10.	Nacl amount % (in ash of active content)	3.72	2.46	3.36	6.70	3.80
11.	Viscosity (Cps) (2% aq. sol)	49.00	44.10*	38.00	53.80	35.00
12.	Intrinsic viscosity (dl/g)	4.10	3.60	3.50	4.20	3.50
13.	Degree of polymerisation (D P)	410.00	360.00	350.00	420.00	350.00
14.	Colour	Light Brown	White	Light Cream	Light creamish	Light grey
15.	Gum power	V-Good	Good	V-Good	V-Good	V-Good
16.	Solubility	V-Good	Good	Good	Good	Good

1. Fir saw dust pulp; 2. Rags; 3. White writing paper; 4. Cotton waste; 5. Used cyclostyled waste paper.
*1% aqueous solution

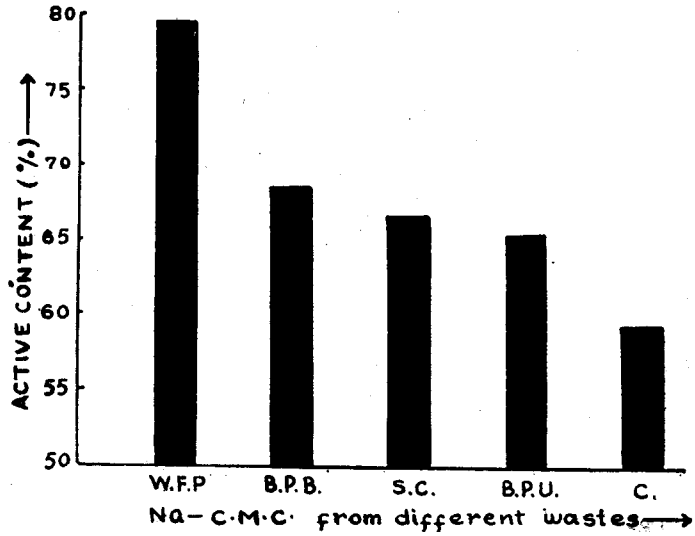

Fig. 2. Active content of Na–C. M. C. from different wastes under identical conditions of preparation.
1— Fir saw dust pulp.
2— Rags.
3— White writing Paper.
4— Cotton waste
5— Used cyclostyled waste paper.

Figure 3–represents the active content obtained from other waste materials already reported in our earlier communication after suitable modifications (Table IV).

TABLE-IV

Active content & viscosity of various Na–C. M. C. samples

Sr. No	Material used	Active content %	Viscosity (CPS) (2% aq. sol.)
1.	Whatmann filter paper	79.00	65.10*
2.	Book paper (bleached)	68.20	38.00
3.	Stationery cuttings	66.20	42.00
4.	Book paper (unbleached)	65.00	43.40
5.	Commercial Na–C.M.C.	59.00	58.60

*1% aq: sol.

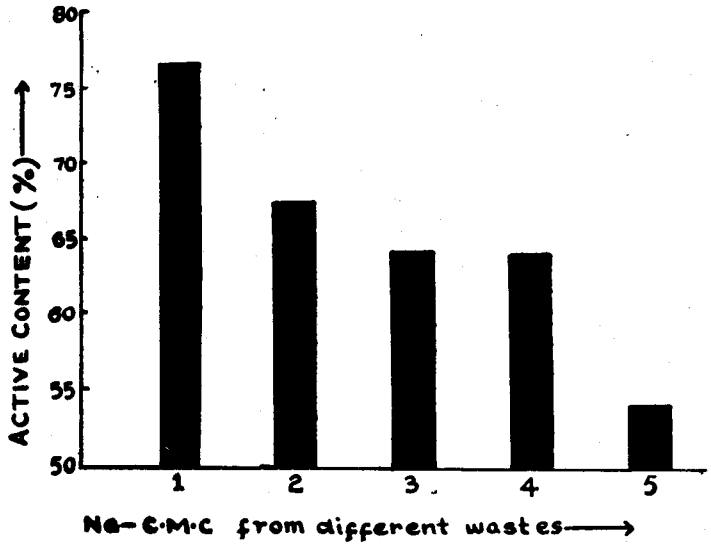

Fig. 3. Active content Na–C. M. C. from different wastes prepared under identical conditions.

W.E.P– Whatmann filter Paper.
B.P.B-- Book Paper (bleached)
S. C.-- Stationery cuttings.
B.P.P.– Book Paper (unbleached)
C.----- Commercial Na–C. M. C.

References

1. Chawla, J. S. & Shanker, G., IPPTA, **9**, 346 (1972).
2. Chawla, J. S. & Shanker, G., IPPTA, **10**, 39 (1973).
3. A. S. T. M. Standards, Part 15, 496–506 (1966).
4. Tappi Standards & suggested methods.