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Utilization of Cellulosic Wastes in Textile and Garment Industries. I. Synthesis and Grafting Characterization of Carboxymethyl Cellulose from Knitted Rag

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ABSTRACT: Carboxymethyl cellulose (CMC) was synthesized from knitted rags, a cellulosic wastes of textile and garment industries, in aqueous ethanolic sodium hydroxide and subsequently mono-chloroacetic acid reaction medium. A low substituted to high substituted products was obtained from single step up to seven steps carboxymethylation of cellulose. In this way, it was possible to produce low cost and different grades or substituted carboxymethylation derivatives of cellulose. Solubility, degree of substitution (DS), CMC content, and molecular weight of CMC were increased gradually with the increase of the number of reaction steps, although fourth step attained optimal. The DS of CMC from one to seven reaction steps was 0.91–2.84, respectively. Similarly, CMC content and molecular weight of CMC were 72.60%–85.00% and 153,886–252,231, respectively. Grafting of CMC film with methyl methacrylate (MMA) improved strength properties, but it decreased moisture content due to the incorporation of hydrophobic MMA monomer. The prepared CMC, when applied as sizing agent to cotton fabric, was deposited on the fabric surface through bond formation making it smooth without any crack or rupture. © 2012 Wiley Periodicals, Inc. J. Appl. Polym. Sci. 000: 000–000, 2012

KEYWORDS: carboxymethyl cellulose; cellulose and other wood products; knitted rag; textiles; grafting; fibers; copolymers

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INTRODUCTION

Carboxymethyl cellulose (CMC) is one of the most versatile cellulosic derivatives of the present day world and tremendously used in textiles and many other industrial fields including food products, paper, cosmetics, pharmaceuticals, and adhesives.[1] Hence it is most important in the textile manufacturing and in everyday life. It is true for present day that Bangladesh is a textile industry-based country and large amount of CMC is being imported to meet its demand for textile sector in every year and the importance of CMC is increasing day by day.[2]

It has been reported that CMC was prepared from different cellulosic sources by different researchers following many different methods.[3–8] Many researchers have investigated the synthesis of CMC from different cellulosic sources, such as paper sludge,[3] hyacinth,[4] wood residue,[7] cotton linters,[8,9] bagasse,[10] etc. No worth mentioning work has been performed on the synthesis of CMC from knitted rag. Knitted rag contains high amount of good quality α-cellulose which is 95–98%,[11] and huge amount of knitted rags are deposited as textile wastes in different garment industries that have virtually no use. The wastes contain both colored and non-colored rags. These are normally dumped in nearby area, and degraded naturally. Hence these created environmental pollution. As the rags contains high amount of good quality α-cellulose, it can easily be collected with almost free of cost and it will be a great source for the manufacture of CMC and other derivatives. The purpose of the present work is to explore the use of these knitted rags by synthesizing different grades of CMC by applying multiple steps carboxymethylation techniques. In this report, a multiple step carboxymethylation techniques will be adopted for producing lucrative CMC, with good solubility and fair degree of substitution (DS). The unique feature of this report is the reuse of the recovered ethanol used in carboxymethylation, the synthesis of CMC with high DS, the synthesis of grafted CMC, and the application of CMC to textiles.

MATERIALS AND METHODS

Materials

The knitted rags (simply rags), a cellulosic wastes of textile and garment industries, was collected from Mozart Knit Ltd, Ashulia, Saver, Dhaka. Ethyl alcohol (Merck, Germany), sodium hydroxide (Merck, India), mono-chloroacetic acid (BDH, England), sodium carbonate (BDH, England), standard CMC (BDH, England), etc.

were purchased as analytical grade and used without further purification.

Preparation of Sample

The collected knitted rag contains both non-colored and colored rags. The separated non-colored knitted rag was washed with 1% Na_2CO_3 solution at 60°C.[9] The colored knitted rag was immersed in bleaching liquor containing 4% (wt/wt) of 35% (wt/wt) hydrogen peroxide, 2.5% sodium silicate, and 1% sodium hydroxide on the basis of rag at 40°C for 1 h in the rag–liquor ratio of 1 : 50. Then the temperature was raised to 85°C and continued for 3 h with occasionally stirring.[12] Finally, the bleached or non-colored knitted rag was washed with sufficient fresh water and then dried in air.

Carboxymethylation

The CMC was synthesized by the conversion of cellulosic rag to alkali cellulose swollen in aqueous NaOH and a surplus of 95% ethanol as solvent with mono-chloroacetic acid. The production of CMC was carried out by the following two steps:

(a) **Basification**

The first step provides the formation of a reaction mixture comprising of 1 g knitted rag and 18% aqueous ethanolic NaOH in the rag–liquor ratio of 1 : 14, and this was carefully controlled to 30°C for 3 h with occasionally stirring. This step is known as steeping.

$$\left[C_6H_7O_2(OH)_3\right]_n + n\text{NaOH} = \left[C_6H_7O_2(OH)_2\text{ONa}\right]_n + nH_2O$$

(b) **Etherification**

In the second step, an 80% (wt/vol) aqueous monochloroacetic acid ($ClCH_2COOH$) was added drop by drop to the previous mixture in the rag–liquor ratio of 1 : 4 and this was carefully controlled to temperature 50°C for 6 h. This step is known as carboxymethylation step.

$$n\text{ClCH}_2\text{COOH} + n\text{NaOH} = n\text{ClCH}_2\text{COONa} + nH_2O$$
$$\left[C_6H_7O_2(OH)_2\text{ONa}\right]_n + n\text{ClCH}_2\text{COONa} = \left[C_6H_7O_2(OH)_2\text{OCH}_2\text{COONa}\right]_n + n\text{NaCl}$$

Single-step carboxymethylation gave only low substituted product. Highly substituted CMC can be prepared by performing multi-steps carboxymethylation of the cellulose. In each step, prepared CMC of immediate previous step was purified with 80% aqueous ethanol solution and then carboxymethylation of this low substituted CMC was done with fresh addition of NaOH and $ClCH_2COOH$. Finally, the synthesized crude Na-CMC was purified with 80% ethanol solution, and then dried in a desiccator over P_2O_5.

Determination of Degree of Substitution

One gram of dried CMC was added to 100 mL of distilled water and 12.50 mL of 1*N* NaOH. After dissolving the mixture was titrated by 1*N* HCl. The DS of CMC was determined by following equation[4,13] as,

$$\text{DS} = 0.162A/(1 - 0.058A)$$

where, *A* is the milliequivalents of sodium hydroxide required per gram of sample.

Determination of Molecular Weight

The Na-CMC was dissolved in aqueous 0.8 *M* NaOH solution. From the intrinsic viscosity, the molecular weight of Na-CMC was determined by using Ostwald viscometer at 35°C and calculated by the following "Mark-Houwink–Sakurada equation[14] as,

$$[\eta] = KM^a$$

where, $[\eta]$ is the intrinsic viscosity, *M* is the molecular weight of Na-CMC, *K* is the constant, and "*a*" is the polymer shape factor.

Determination of CMC Content

Exactly 1.5 g of CMC was added to 100 mL of 80% aqueous methanol solution, stirred, kept for 10 min and filtered. The cake was washed by the fresh 100 mL of 80% aqueous methanol and dried to obtain pure Na-CMC.[6] The CMC content was calculated as,

$$\text{CMC content, \%} = 100M_2/M_1$$

where, M_1 (g) is the weight of sample before wash and M_2 (g) is the weight of washed sample.

Determination of NaCl in Na-CMC

Two grams of CMC were added to 250 mL of 65% aqueous methanol and kept for 5 h. One hundred milliliters of liquid phase were neutralized by dilute 0.1*N* HNO_3 and titrated with 0.1*N* $AgNO_3$ solution.[6] The NaCl content was calculated as,

$$\text{NaCl, \%} = 1.461V/M$$

where, *V* (mL) is the amount of $AgNO_3$ solution and *M* (g) is the weight of dried sample.

Method of Grafting

Graft polymerization of CMC was carried out with 80% methyl methacrylate (MMA), 5% $K_2S_2O_8$, 5% Fe_2SO_4, at 60°C for 90 min in the CMC-liquor ratio of 1 : 30. Graft yield and grafting efficiency was calculated according to the following formula[15,16] as,

$$\text{Grafting yield, \%} = [(W_1 - W_0)/W_0] \times 100$$
$$\text{Grafting efficiency, \%} = [(W_1 - W_0)/W_2] \times 100$$

where, W_0 is the weight of ungrafted CMC film, W_1 is the weight of the grafted CMC film and W_2 is the weight of total monomer used.

Moisture Content

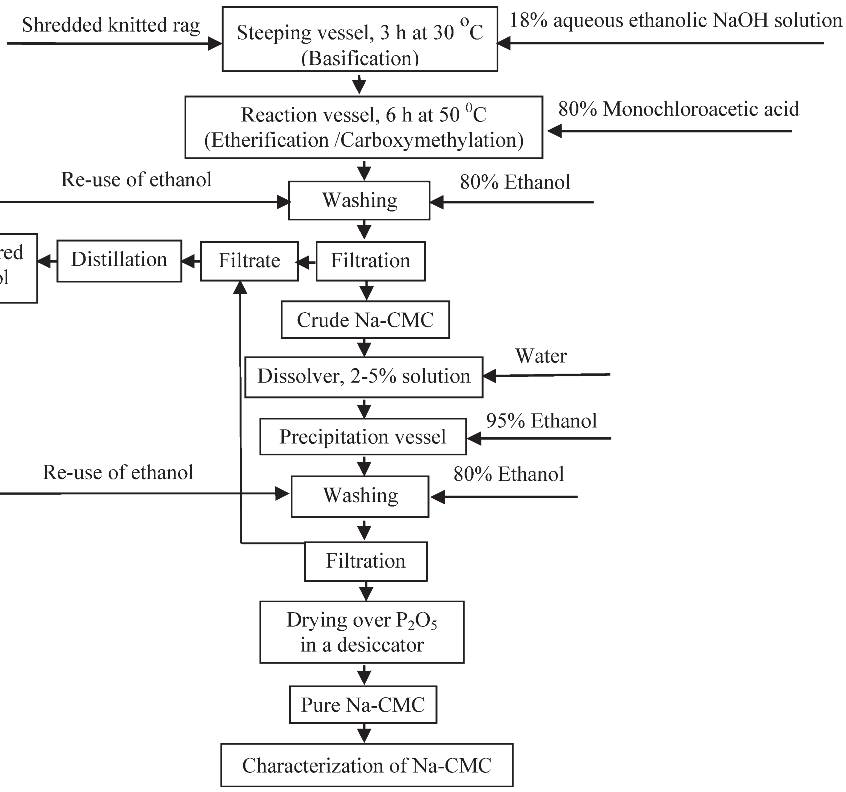
The grafted and ungrafted 2% CMC films were weighed accurately and dried it at 105°C for 2 h in a Forced convection oven (FC-610, Toyo Seisakusho Co. Ltd., Tokyo, Japan). Then these were cooled in a desiccator for about 30 min and weighted again. Moisture content was determined using the equation below as,

$$\text{Moisture content, \%} = [(W_0 - W_1)/W_0] \times 100$$

where, W_0 is the weight of air dry film and *W* is the weight of oven dry film.

Water Absorption

The grafted and ungrafted 2% CMC films (6 cm × 4 cm) were weighed accurately and immersed in distilled water for 15 h at room temperature 30°C. The films were wiped using tissue paper



Scheme 1. Flow chart for the preparation of CMC from knitted rag.

and weighted again. Water absorption was determined using the equation below[17] as,

$$\text{Water absorption, \%} = [(W_1 - W_0)/W_0] \times 100$$

where, W_0 is the weight before immersion and W_1 is the weight after immersion.

Gel Content

Gel content of the grafted and ungrafted 2% CMC films was determined by Soxhlet extraction using hot toluene as solvent. The films (6 cm × 4 cm) were weighed and extraction was carried out with 250 mL toluene for 6 h. After that, the samples were taken out followed by vacuum dried and re-weighed until it approached a constant weight. Gel content of the grafted and ungrafted CMC films was calculated according to the following equation[17] as,

$$\text{Gel content, \%} = (W_1/W_0) \times 100$$

where, W_0 is the weight before extraction and W_1 is the weight after extraction.

Tensile Strength

To determine tensile strength of CMC, modified CMC films of size 2 cm × 1 cm were measured by using a Tensile strength tester (Harada Co. Ltd. Nagano-ken, Japan). The distance between the jaws of the film was 1 cm.

FTIR Measurement

The washed knitted rag and prepared CMC was dried at 50°C and was made it fine powder with the help of molar-pastle. This powder sample was mixed with KBr in the ratio of 1 : 125 and a pellet was prepared. Then, IR-spectra of this KBr pellet was recorded with a FTIR spectrophotometer (Model: FTIR-8900, Shimadju, Japan) in the spectral region of 4000–400 cm^{-1}.

Application of CMC to Cotton Fabric

The cotton fabric was washed with 1% Na_2CO_3 solution at 60°C for 2 h. After drying, this fabric was immersed in 3% CMC solution as sizing agent at 60°C for 2 h. Then the CMC sized fabrics were dried and calendared.

Method of Scanning Electron Microscopy

The surface morphology of washed and CMC-sized cotton fabrics were examined by scanning electron micrograph (SEM, Hitachi, model-S 3400 N VPSEM, Japan). The micrographs were taken at magnification of 500 and 1000 times using (20 kV) accelerating voltage and scale 100 μm, 50 μm, respectively.

RESULTS AND DISCUSSION

The synthesis of Na-CMC from knitted rag (1 g) was carried out according to Scheme 1. The potentiality of the process is the use of the recovered ethanol through distillation after washing of crude Na-CMC. The recovered ethanol was more than 90% in each step and this ethanol was used for the successive steps. The effect of solvent system, alkali concentration for basification, concentration of monochloroacetic acid for etherification, time and temperature of the reaction for the DS was studied. The reaction was optimized with respect to the DS by varying each of the parameters.

Table I. Preparation of Na-CMC by Multi-Steps Carboxymethylation of Knitted Rag (NaOH = 18% in the Rag–Liquor Ratio = 1 : 14; $ClCH_2COOH$ 80% in the Rag–Liquor Ratio 1 : 4)

No. of reaction steps	Yield of Na-CMC (%)	Remarks
1	360	Partially soluble in water
2	751	Soluble in water
3	1052	Highly soluble in water
4	1231	Highly soluble in water
5	1304	Highly soluble in water
6	1404	Highly soluble in water
7	1494	Highly soluble in water

The yield of Na-CMC obtained from carboxymethylation process at one to seven steps is shown in Table I. The optimized conditions for the synthesis of Na-CMC was established as ethanol to cellulose ratio, 12 : 1 (vol/wt); water to cellulose ratio, 2 : 1 (vol/wt); overall concentration of NaOH, 18% (wt/vol); steeping time, 3 h; steeping temperature, 30°C; concentration of monochloroacetic acid, 80% (wt/vol); time of carboxymethylation, 6 h; temperature of carboxymethylation, 50°C. It can be seen from Table I that the yield of CMC increased with the increase of the number of reaction steps in the optimized condition of carboxymethylation. The yield of Na-CMC in first step of carboxymethylation in aqueous ethanolic solution was 360%, but for the second to seven successive steps these were 751–1494%, respectively. Solubility of the prepared Na-CMC samples was tested in water. The Na-CMC obtained from multiple steps carboxymethylation showed a high solubility in water than the product of single step. The role of the solvent in the carboxymethylation reaction is to provide accessibility of etherifying reagent to the reaction centre of the cellulose chain.[18]

The carboxymethylation reaction was optimized with respect to DS by varying each of the parameters. The DS of the prepared Na-CMC was determined[4,13] and the DS data of each step are listed in Table II. It can be seen from Table II that DS obtained at the initial step is 0.91 and with the increase of the number of reaction step, the amount of DS is decreased. The reason of this phenomenon was due to the fast substitution reaction for initial steps under alkaline conditions. The hydroxyl group of cellulose in rag fiber was very active and could be replaced by carboxymethyl group, which decreased the number of OH groups very fast. It can also be seen from Table II that from the 4th step to 7th step reaction, the increasing rate of DS is very slow. The DS value obtained at the 4th step is 2.72 and that obtained at the 7th step is 2.84 which is maximum. The DS of CMC obtained until now by 10th step carboxymethylation[19] is 2.63.

Table II. Determination of DS in Na-CMC Prepared From Knitted Rag at Different Steps

No. of reaction steps	Milliequivalents of NaOH required per gram of sample, *A*	Degree of substitution
1	4.21	0.91
2	6.07	1.64
3	7.93	2.46
4	8.49	2.72
5	8.63	2.80
6	8.68	2.82
7	8.70	2.84

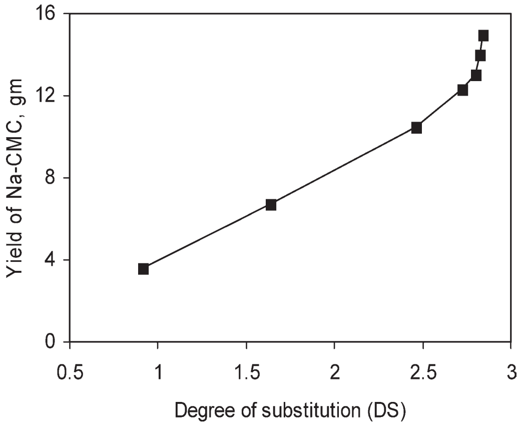


Figure 1. The relation between yield of Na-CMC (g) and the DSs.

Table III. Determination of Molecular Weight of Na-CMC in Aqueous 0.78*M* NaOH Aqueous Medium at the One to Seven Steps Product (Value of $k = 37 \times 10^{-5}$ dL/g and $a = 0.61$ at 35°C)[13]

No. of reaction steps	Degree of substitution	Intrinsic viscosity	Molecular weight
1	0.91	0.54	153,886
2	1.64	0.61	187,888
3	2.46	0.66	213,796
4	2.72	0.68	224,543
5	2.80	0.71	241,046
6	2.82	0.72	246,603
7	2.84	0.73	252,231

Table IV. Determination of CMC, NaCl, and Others in Synthesized Na-CMC After Several Reaction Steps

No. of reaction steps	Degree of substitution	CMC (%)	NaCl (%)	Others (%)
1	0.91	72.60	3.07	24.33
2	1.64	76.80	3.29	19.91
3	2.46	79.00	3.44	17.56
4	2.72	81.40	3.51	15.09
5	2.80	82.80	3.58	13.62
6	2.82	83.80	3.65	12.55
7	2.84	85.00	3.65	11.35

Table V. Effect of Monomer Concentration on Modification of CMC Film With Methylmethacrylate (MMA)

No. of experiments	Monomer concentration (%)	Grafting yield (%)	Grafting efficiency (%)
1	40	10.29	25.74
2	60	12.76	19.60
3	*80*	*20.58*	19.40
4	90	13.51	15.15
5	100	12.35	12.35

Figure 1 shows that the yield of CMC was increased gradually from initial step with the increase of DS. Complete carboxymethylation of cellulose, i.e., theoretically highest DS, which is three but practically it is not possible. That is why near the end of the reaction steps DS increasing was slowing down as well as yield of Na-CMC. This might occur due to successive treatment with NaOH generated "activated" hydroxyl groups for substitution. The carboxymethylation depends upon the accessibility of reagent and the availability of the activated hydroxyl groups. Increased substitution by successive treatment with alkali clearly showed activation of the secondary hydroxyl groups.

The molecular weight of Na-CMC obtained from each step was determined by viscosity measurement using Ostwald viscometer. The viscosity measurement of each Na-CMC sample was calculated by using "Mark-Houwink–Sakurada" equation,[14] $[\eta] = KM^a$. From Table III it can be seen that the molecular weight of the prepared Na-CMC increased gradually with the increase of reaction steps from one to seven. The reason is that as the DS increased with successive steps, number of OH groups attached to cellulose molecule was replaced by carboxymethyl groups. As the molecular weight of carboxymethyl group is higher than the OH group, the molecular weight of final product Na-CMC also increased with the reaction steps. Table III shows that increase of DS and molecular weight was strung down with the number of successive steps indicating the DS of Na-CMC went on to the completeness of the substitution reaction.

The amount of CMC, NaCl, and others contents in Na-CMC are listed in Table IV. From Table IV it can be observed that percentage of CMC and NaCl increased gradually with the increase of DS as well as number of reaction steps and at the same time percentage of other materials decreased. This result confirms the increase of CMC with the increase of reaction steps.

From Table V it can be seen that maximum graft yield was formed when CMC film was grafted with 80% MMA and then graft yield decreased with further increase of MMA concentration, as homopolymer was formed at the higher concentration of MMA.

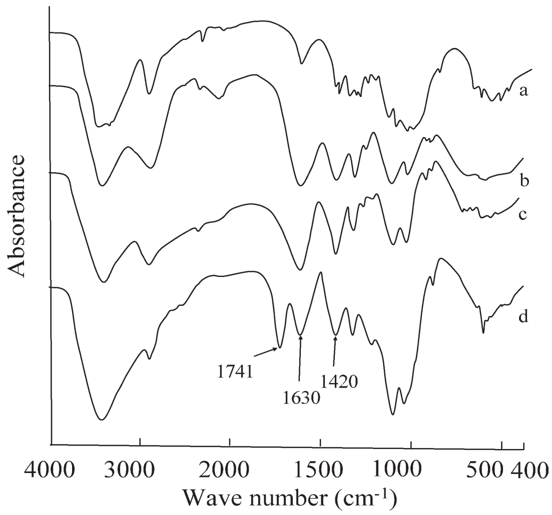


Figure 2. FTIR Spectra of (a) knitted rag (cellulose), (b) standard CMC film (DS = 0.8), (c) prepared CMC film (DS = 2.72), and (d) grafted CMC film.

The results of physical properties, such as moisture content, water absorption, gel content, tensile strength and elongation at break, of CMC and grafted CMC are listed in Table VI. From the Table VI, it can be seen that moisture content and water absorption value of CMC are 2.86% and 7.69%, respectively. These values are much higher than the value of grafted CMC with MMA monomer. This indicates that MMA had been successfully grafted or incorporated with the prepared CMC film and due to the incorporation of hydrophobic MMA monomer, the grafted film lost its moisture absorption capacity. The gel content of grafted film is 97.03% which is lower than CMC film. The high gel content means high reactivity.[17] It is monomer, proved that MMA had been successfully grafted on to prepared CMC film. From Table VI, it can also be observed that tensile strength and elongation at break of CMC film is 0.72 MPa and 2.6%, respectively. Whereas grafted CMC film is 1.34 MPa and 1.7%, respectively. This result shows that the tensile strength of grafted CMC film is higher than CMC film and elongation at break is lower. The tensile strength of grafted CMC film is higher due to the incorporation of MMA monomer onto CMC film and this reasonably increased the rigidity of the grafted film and hence lower elongation at break.

FTIR Analysis of Prepared CMC

The FTIR spectra of standard CMC film (DS = 0.8), prepared CMC film (DS = 2.71), and MMA-grafted CMC film are shown in Figure 2. From Figure 2, it can be seen that the peaks assigned

Table VI. Determination of Grafting Properties of 3% CMC film (DS = 2.72)

Samples name	Moisture content (%)	Water absorption (%)	Gel content (%)	Tensile strength (MPa)	Elongation at break (%)
CMC film	2.86	7.69	99.47	0.72	2.6
Grafted CMC film	1.08	1.78	97.03	1.34	1.7

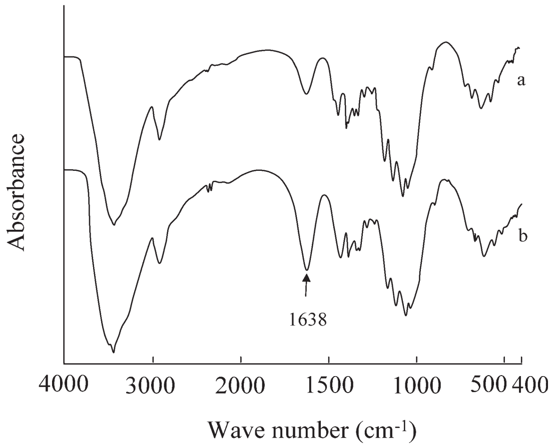


Figure 3. FTIR spectra of (a) washed cotton fabric, (b) 3% CMC modified (DS = 2.46) cotton fabric.

at 1630 cm^{-1} and 1420 cm^{-1} indicate the presence of carboxymethyl substituent in both standard CMC and prepared CMC films [Figure 2(b, c)]. The carboxylate ion[20] gives rises to two bands: a strong asymmetrical stretching band near 1650–1550 cm^{-1} and a weaker symmetrical stretching band near 1400 cm^{-1}. Two weak peaks near 1630 cm^{-1} and 1420 cm^{-1} in cellulose was observed which are due to absorbed water and CH_2, respectively. Hence the occurrence of strong peaks in CMC films confirmed the formation of CMC by the process of etherification between knitted rag and mono-chloroacetic acid in strong alkaline alcoholic medium. An extra absorption band at 1741 cm^{-1} was observed in grafted CMC film indicating the stretching of C=O group, which was the introduction of ester carbonyl group of the grafted MMA.[21,22] So the appearance of new peak in the spectrum of CMC film-*g*-MMA gave supporting evidence for the grafted product and suggests that MMA had been successfully grafted onto prepared CMC film.

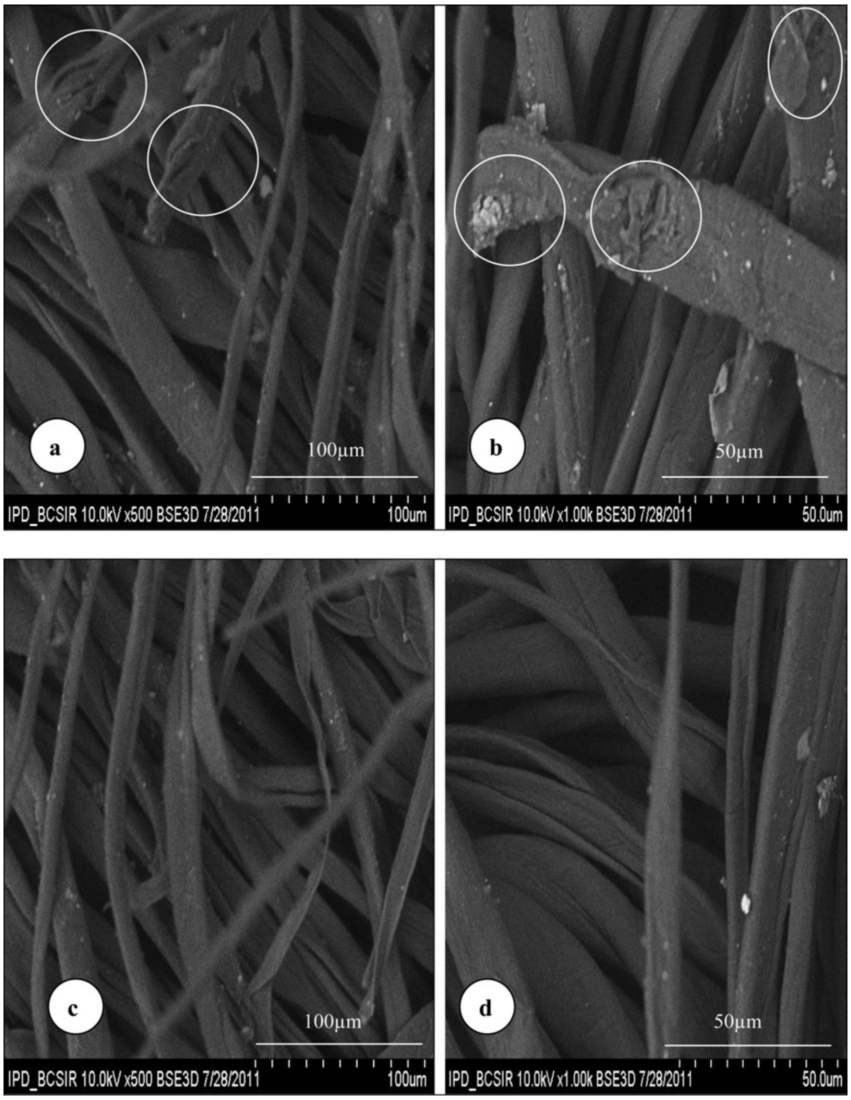


Figure 4. Scanning electron micrograph of (a) washed cotton fabric (magnified 500 times), (b) washed cotton fabric (magnified 1000 times), (c) washed cotton fabric after 3% CMC treatment (magnified 500 times), (d) washed cotton fabric after 3% CMC treatment (magnified 1000 times).

Application of CMC to Textiles

CMC is extensively used as protective coating solution for textiles and many other fields. The prepared CMC was applied as sizing agent to develop the physico-chemical characteristics of both fabric and yarn which was investigated by FTIR and SEM test.

FTIR Spectra of Washed and CMC Modified Cotton Fabric

FTIR spectra of washed and CMC-modified cotton fabrics are shown in Figure 3. From the Figure 3(b) it can be observed that an absorption band at 1638 cm^{-1} was assigned which was done to stretching vibration of C=O in CMC modified cotton fabric. The spectra suggest that CMC had been successfully deposited onto modified cotton fabric.

Scanning Electron Micrograph

Washing with soda ash was done to remove impurities and adventitious dirt. The surface morphology of washed and CMC-modified cotton fabrics was examined by SEM and is shown in Figure 4. Figure 4(a, b) show SEM image of washed cotton fabric and its magnified image, respectively. From the Figure 4(a, b) it can be seen that the image of cotton fiber surface was loosened, ruptured, and cracked which were due to washing with soda ash. Figure 4(c, d) show SEM images of 3% CMC-modified cotton fabric and its magnified image, respectively. It can be seen from the Figure 4(c, d) that the surface of the fiber is very smooth and no rupture is visible in the images, because surface of yarns were coated with CMC as sizing materials and projecting fibers are not visible on surface. This indicates the CMC deposition onto the fabric fiber occurred. This deposited CMC might form chemical bond by abstracting a hydrogen atom from the fabric fiber backbone that was observed in FTIR spectra.

CONCLUSION

High performance CMC (high DS, purity and MW) was successfully produced from the high α-cellulose content knitted rag. The prepared CMC will be a great source of textile finishing agents and other uses. Production of different grade CMC using cellulosic wastes of textile and garment industries can be considered a feasible alternative way for generating value-added product and contributing to solving environmental problems resulting from textile wastes damping by nearby industries.

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REFERENCES

1. Bokias, G.; Mylonas, Y.; Bumbu, G. G.; Slaikos, G.; Vasile, C. *Macromolecules* **2001**, *34*, 4958.
2. Parith, D. V.; Sachiurata, N. D.; Calamari, T. A.; Negulescu, I. *Am. Assoc. Text. Chem. Color.* **2003**, *3*, 15.
3. He, X.; Wu, S.; Fu, D.; Ni, J. *J. Chem. Technol. Biotechnol.* **2009**, *84*, 427.
4. Barai, B. K.; Singhal, R. S.; Kulkarni, P. R. *Carbohydr. Polym.* **1997**, *32*, 229.
5. Whistler, R. L. Methods in Carbohydrate Chemistry; Academic press: London, **1963**; Vol. III, p. 322.
6. Togrul, H.; Arslan, N. *Carbohydr. Polym.* **2003**, *54*, 73.
7. Egbuchunam, O.; Okicimen, E. E. *Indian J. Chem. Technol.* **2003**, *10*, 619.
8. Nagieb, Z.; Sakhawy, M. E.; Samir, K. *Int. J. Polym. Mater.* **2001**, *50*, 163.
9. Jahan, I. A.; Sultana, F.; Islam, M. N.; Hossain, M. A.; Abedin, J. *Bangladesh J. Sci. Ind. Res.* **2007**, *42*, 29.
10. Hussain, G.; Ali, M. M.; Chowdhury, M. F. K. N.; Jahan, M. S.; Rahman, M. L.; Khaleque, M. A.; Jalil, M. A. *Bangladesh J. Sci. Ind. Res.* **1997**, *32*, 207.
11. Herman, F.; Conrad, J.; Perlman, J. M., Encyclopedia of Polymer Science and Technology. Plastics, Resins, Rubbers, Fibers, 1st ed. Vol 3. Interscience Publishers: New York, **1965**.
12. Trotman, E. R. Dyeing and Chemical Technology of Textile Fibres, 6th ed.; Edward Arnold: London, **1984**; p 193.
13. ASTM: D1439-61T: Tentative methods of testing sodium carboxymethyl cellulose, ASTM International, West Conshohocken, PA, USA, 1961.
14. Brandrup, J.; Immergut, E. H.; Grulke, E. A. Polymer Hand-Book, 4th ed.; Wiley: New York, **1998**; p VII/46.
15. Mondal, Md. I. H.; Uraki, Y.; Ubukata, M.; Iloyama, K. *Cellulose*, **2008**, *15*, 581.
16. Sikdar, B.; Basak, R. K.; Mitra, B. C. *J. Appl. Polym. Sci.* **1995**, *55*, 1673.
17. Lin, O. H.; Kumar, R. N.; Rozman, H. D.; Noor, M. A. M. *Carbohydr. Polym.* **2005**, *59*, 57.
18. Savage, A. B.; Young, A. E.; Maasberg, A. T. In Cellulose and Cellulose Derivatives (Part 2), Eds. Ott, E.; Spurlin, H. M., Interscience Publishers, Inc.: New York, **1954**; p 882.
19. Heinze, T.; Koschella, A. *Macromol. Symp.* **2005**, *223*, 13.
20. Silverstein, R. M.; Basster, G. C.; Morril, T. C. Spectrophotomtric Identification of Organic Compounds, 5th ed.; Wiley: New York, **1991**; p 102.
21. O'Connor, R. T.; DuPr'e E. F.; McCall, E. R. *Anal. Chem.* **1957**, *29*, 998.
22. Adinugraha, M. P.; Marseno, D. W.; Haryadi, P. Y. *Carbohydr. Polym.* **2005**, *62*, 164.