Synthesis and characterization of carboxymethyl cellulose from office waste paper: A greener approach towards waste management

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In the present study, functionalization of mixed office waste (MOW) paper has been carried out to synthesize carboxymethyl cellulose, a most widely used product for various applications. MOW was pulped and deinked prior to carboxymethylation. The deinked pulp yield was 80.62 ± 2.0% with 72.30 ± 1.50% deinkability factor. The deinked pulp was converted to CMC by alkalization followed by etherification using NaOH and CICH₂COONa respectively, in an alcoholic medium. Maximum degree of substitution (DS) (1.07) of prepared CMC was achieved at 50 °C with 0.094 M and 0.108 M concentrations of NaOH and CICH₂COONa respectively for 3 h reaction time. The rheological characteristics of 1–3% aqueous solution of optimized CMC product showed the non-Newtonian pseudoplastic behavior. Fourier transform infra red (FTIR), nuclear magnetic resonance (NMR) and scanning electron microscope (SEM) study were used to characterize the CMC product.

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1. Introduction

The conversion of waste materials into useful products would alleviate a variety of socioeconomic problems such as providing a greener approach to manufacturing. Recycling of waste has huge environmental and economical benefits as recycling of one ton of waste paper saves 17 trees and 7000 gallons of water (ITC, 2014). Across the world, lots of waste is raised on a daily base and yet the economic systems of these states continue to dwindle unabated. These wastes could be recycled into the products of societal and economic interests. Paper is a significant component of our daily life and likewise one of the most prominent cellulosic biomass wastes produced in an ample amount in several subjects (Gulsoy et al., 2013). The number of times paper can be reprocessed by paper industry is limited due to the shortening of the fiber length and the resulting reduction in tensile strength. Thus, the loss of paper making properties causes it to occupy 30–40% of landfill sites in developed countries (Adhikari et al., 2008). In accession to this, the cellulosic rich fibers, left over from the process of papermaking, are discharged with the wastewater in amounts of several thousand tons a year (Nikolov et al., 2000). Feeble mechanical strength of waste paper has some drawbacks to use in the paper industry alone. However, derivatives of waste paper can find applications in other industrial areas. Thus, chemical adjustment of its cellulosic fibers in the yield of different cellulose derivatives is an additional possible way for its employment and management (Ünlü, 2013).

Cellulose is the most abundant polymer on earth, which fixes it also the most common organic compound. Fair yield of cellulose via photosynthesis is estimated around 830 million metric tons per annum. As 40% of dry-weight of crops is composed of cellulose and its annual output is approximately 200 million tons (Ünlü, 2013). Plants contain approximately 33% cellulose whereas wood contains around 50% and cotton contains 90%. Cellulose is a linear and fairly rigid homopolymer consisting of D-anhydroglucopyranose units (AGU). These units are linked together by β-(1–4) glycosidic bonds formed between C-1 and C-4 of adjacent glucose moieties (Klemm et al., 2001). Most of the cellulose is extensively used as a raw material by the paper industry for the production of paper and cardboard products (Bachheti et al., 2010; Dutt et al., 2011) and a small fraction is used in the production of commodity materials and value added carboxymethyl cellulose and methyl cellulose, etc. Moreover, it can be chemically altered to pay value added cellulose derivatives such as carboxymethyl cellulose, methyl cellulose, ethyl cellulose, hydroxypropyl cellulose, cyano-ethyl cellulose, and so forth (Varsney and Naithani, 2011). Among all these modified cellulosic products, CMC is manufactured in significant amounts due to its wide commercial applications with...
regard to volume demand. It accepts a broad circle of applications within different industries including food ingredients, pharmaceuticals, cosmetics, paper products, adhesives, lithography, ceramics, detergents, and materials (Saputra et al., 2014). Such diverse applications along with its low pricing make CMC as one of the major market shareholder within all the cellulose ether product categories.

Now a day’s cotton and wood pulp are basic raw material for producing modified cellulose products. Competitive demand of these crude materials for many other industries will not be affordable in future for the formation of modified cellulose products. Thus, there is an urgent demand for low cost, non-competitive and sustainable raw materials for the production of modified cellulose products. Recently some new and underutilized raw materials like bamboo shaving (Chen and Lou, 2014), water hyacinth (Saputra et al., 2014), cotton linters (Khullar et al., 2005; Xiquan et al., 1990), textile waste (Bidgoli et al., 2014), recycled news paper (Unlü, 2013), unused paper (Mahkami and Talaieipour, 2011), paper sludge (He et al., 2009) and broad bean hull (Al-Subhi, 2014), were also explored for the CMC synthesis, but none of them has been commercialized so far. The central aim of the present work is to utilize the abundantly available, underutilized raw material paper cellulose rich biomass as an alternative feedstock rather than expensive cotton linters and wood pulp for CMC production, which now, are discouraged due to strict environment conservative regulations. Waste paper cellulose is highly amorphous in nature, hence unlike commercially available cellulose, it is more accessible to chemical change (Adhikari et al., 2008). Further, the mixed office waste paper has high cellulose, low lignin and less ink contents in comparison to waste newsprint or other types of paper waste, hence a high degree of functionalization can be attained.

Previously attempts have also been made for the synthesis of CMC from waste paper (Mahkami and Talaieipour, 2011; Unlü, 2013). However, to date, to the best of our knowledge, no research has described the pre-steps of processing like deinking and pulping etc. truly required for a CMC of higher grade from real waste paper in true sense. Moreover, the reuse of waste paper essentially depends on the process of pulping followed by deinking. Deinking is a sophisticated and essential process for reuse of waste paper. The complete process of deinking has not been performed in these studies while in some other attempts (Mahkami and Talaieipour, 2011) authors has undertaken fresh unprinted paper which does not contain ink or other impurities and thus cannot be classified under “waste”. Prompted by the aforesaid facts, the possibility of using mixed office waste paper as a feedstock for production of CMC was examined and findings are reported herein.

2. Experimental

2.1. Materials

Mixed office waste (MOW) paper was used as a raw material. The samples were collected from the different Research Divisions of Forest Research Institute, Dehradun, Uttarakhand (India). MOW mainly consisted of copier and computer printout papers. The wastepaper was manually sorted to remove non-paper objects such as stickers, staples, rubber bands, and others. The sorted waste paper stock was stored in polyethylene bags at room temperature until needed. All the chemicals used were of analytical grade.

2.2. Methods

2.2.1. Characterization of MOW

Proximate chemical composition of the MOW was studied as per the Technical Association of the Pulp and Paper Industry (TAPPI) standard methods. TAPPI method Nos. T 421 om-02; T 09 m-54; T 19 m-50; T 203 cm-99; T 222 om-02; T 211 om-02 and T 207 cm-99 were used for the determination of moisture content, holocellulose, pentosans, alpha-cellulose, lignin, ash, hot water solubles respectively. The MOW was processed using valley beater (Valley Iron Work Co., Appleton, WI, USA) at room temperature for defiberization and production of pulp for 20 min. Pulp was air dried and powdered in a Willey mill (A. Gallenkamp Co., Ltd., London) to 60 mesh size. The powdered material was used for proximate analysis.

2.2.2. Pulping and deinking

The reuse of waste paper essentially depends on the process of pulping followed by deinking. Deinking is a sophisticated process for reuse of waste paper. In the present study MOW paper was pulped and deinked in the hydrapulper followed by the flotation cell. The process of deinking involves removal of ink particles from the fiber surface and the separation of the dispersed ink particles from fiber suspensions by washing or flotation (Bajpai and Bajpai, 1998; Prasad et al., 1993). The ink particles are physically bonded to the fibers because of high heat, making it difficult to remove from the cellulosic fiber. In the flotation deinking, air bubbles rise through the agitated liquid in the tank containing suspended waste paper pulp and contaminant particles. The rising bubbles collect hydrophobic contaminants and ink agglomerates. The attached particles are then transported to a froth layer, from where they are easily removed. The MOW was manually torn into a size of approximately 1-in. squares, pulped and subsequently subjected to deinking. Prior to pulping of waste paper, two hundred gram of oven dried MOW was soaked in water (500 ml) for overnight at room temperature. Wet MOW was charged in hydrapulper with NaOH (2% w/w), Na2SiO3 (2% w/w), H2O2 (1% w/w) at a 12% pulp consistency for 30 min at 55 ± 2 °C. This pulp was transferred to a flotation cell with Tween-80 (0.1% w/w) at 40 ± 2 °C for 10 min flotation time. The consistency of the pulp was maintained 1% in the flotation cell by addition of water. After the completion of deinking, the pulp was recovered on muslin cloth from the drain valve of the flotation cell. The pulp was then washed at 2% consistency in a plastic container followed by filtration through a pulp screen. The washing process was repeated three times. After washing, the residual water present in the deinked pulp was drained by the laboratory hydro extractor machine. The hydro extracted deinked MOW pulp was shredded in the pulp shredder and stored in airtight plastic containers at 4 °C. The deinking efficiency of the process was evaluated by means of brightness measurements, as indicated in TAPPI method T452 om-08. The effectiveness of deinking depends on the technique used, printing conditions, quality of ink, and kind of printing substrate.

2.2.3. Sample preparation

Hydroextracted deinked MOW pulp was placed in oven at 105 ± 2 °C for overnight drying. The oven-dried samples were then passed through a laboratory mixer in order to avoid the lump forming in the pulp. Then the disconcerted deinked MOW pulp was processed for carboxymethylation.

2.2.4. Carboxymethyl cellulose synthesis

CMC was produced by etherification of the hydroxyl groups with sodium monochloroacetate (SMCA) in the presence of aqueous alkali. The carboxymethylation of MOW paper proceeds through the Williamson’s ether synthesis reaction steps (Tijsen et al., 2001) with an undesired side reaction. In the main reactions the sodium hydroxide reacts first with the hydroxyl groups of the cellulose to give alkoxide. The carboxymethyl groups are then formed in a SN2 reaction between the cellulose alkoxide and SMCA (Eq. (1)).
Cell-OH + ClCH₂COONa + NaOH

\[
\text{Cell-O-CH₂COONa + NaCl + H₂O} \quad (1)
\]

The condensation reaction is the production of sodium glycolate by the reaction of sodium hydroxide with sodium monochloroacetate (Eq. (2)).

\[
\text{NaOH + ClCH₂COONa} \rightarrow \text{HO-CH₂COONa} + \text{NaCl} \quad (2)
\]

The conditions for carboxymethylation of MOW pulp was optimized with respect to DS by varying the process parameters. Each parameter was varied individually by keeping the remaining parameters constant during the course of the reaction. Isopropanol as a solvent was used to provide miscibility and accessibility of the etherifying reagent to the reaction center of the cellulose chain rather than glycolate formation during carboxymethylation.

Synthesis of CMC from MOW was carried out in two steps of alkalization followed by etherification under heterogeneous conditions. Alkalization was done by addition of aqueous NaOH (0.063–0.156 M) in a vigorously stirred slurry of MOW pulp (5 g) in iso-propanol (115 ml) over a period of 30 min at 25 °C. Stirring was continued for another 60 min. Finally, pre-dissolved sodium monochloroacetate (SMCA, 0.075–0.118 M) in 10 ml iso-propanol was added to the alkali treated mass, under continuous stirring. The reaction mixture was heated (40–70 °C) for 1–4 h. After completion of etherification reaction the excess alkali was neutralized with acetic acid (5 M). The reaction mixture was filtered, washed with 70% aqueous methanol, followed by absolute methanol, and dried at 60 °C in a hot air oven.

2.2.5. Determination of degree of substitution (DS)

The degree of substitution (DS) of the MOW derived CMC was determined by the method as described by Bono et al. (2009). Prepared CMC sample (4 g) was stirred in a beaker with 20 M ethanol (75 ml) for 5 min. Nitric acid (16 M, 5 ml) was added to this solution followed by boiling the solution for 7 min. This solution was again stirred for 10 min in hot conditions. The solution was filtered under vacuum and the residue was washed with 5 ml of 18 M ethanol (60 °C) for 5 times. Then the residue was washed with anhydrous methanol (50 ml). Finally, the residue was dried at 60 °C for 3 h and cooled in a desiccator for half an hour. Aqueous solution of CMC (1%) mixed with 25 ml of 0.3 M NaOH solution. This mixture was boiled for 15 min and titrated with 0.3 M HCl after cooling by using phenolphthalein indicator. To estimate the degree of substitution following Eqs. (3) and (4) were used:

\[
A = \frac{BC - DE}{F} \quad (3)
\]

Degree of substitution = \[
\frac{0.162 \times A}{1 - (0.058 \times A)} \quad (4)
\]

where \(A\) = milli-equivalents of consumed HCl per gram of specimen; \(B\) = volume of NaOH added; \(C\) = molarity of NaOH; \(D\) = volume of consumed HCl; \(E\) = molarity of HCl used; \(F\) = CMC in grams; 162 is the molecular weight of the anhydrous glucose unit and 58 is the net increment in the anhydrous glucose unit for every substituted carboxymethyl group.

2.2.6. Determination of apparent viscosity (η)

The apparent viscosity (η) of the optimized CMC product of different concentration (1–3% aqueous solution), was determined at different shear rate using a Brookfield Digital Viscometer model (DV ULTRA-III USA). The apparent viscosity of the optimized product was also determined at different shear rates, ranging from 3.3 to 26.4 s⁻¹. All measurements were made at 17 ± 1 °C.

2.2.7. Fourier transform infrared (FT-IR) spectroscopy

All FTIR spectra were recorded on a JASCO FT-IR 5300 using KBr pellets. Transmission was measured in the wave number range of 800–4000 cm⁻¹.

2.2.8. Nuclear magnetic resonance (NMR) spectroscopy

All NMR spectra were recorded on Bruker AV400 high resolution multinuclear FT-NMR spectrometer (1H frequency of 400.23 MHz, 13C frequency of 100.64 MHz).

2.2.9. Scanning electron microscope (SEM) study

The surface of MOW pulp and CMC was examined utilizing a scanning electron microscope, FEI, Netherland for observing the surface morphology. The samples were supported on the hot carbon grid. The SEM studies were conducted with an electron beam accelerating potential of 15 kV.

2.2.10. Yield measurement

The yield of CMC was calculated on the oven dry weight basis. The net dry weight of CMC was calculated as per the following formula:

\[
\text{Yield} (\%) = \frac{\text{Weight of dried CMC/Oven dried mass of MOW}}{100}
\]

3. Results and discussions

3.1. Characterization of MOW

Characterization of MOW was carried out in order to determine the feasibility of the MOW for carboxymethylation with respect to chemical makeup. The waste paper dust (60 mesh size) was found to contain moisture content (8.28 ± 0.63%), holocellulose (88.12 ± 2.88%), alpha cellulose (78.52 ± 2.19%), pentosans (7.42 ± 0.16%), lignin (2.33 ± 1.06%), ash (3.50 ± 0.20%) and hot water soluble (2.10 ± 0.20%). The presence of 78.52 ± 2.19% of alpha cellulose content makes MOW a prospective raw material for modified cellulose products.

3.2. Pulping and deinking

The yield of deinked pulp after deinking was found to be 80.62 ± 2.0% with 72.20% deinkability factor. The results reported in the present study are in close agreement with the results reported by Pathak et al. (2011), Varshney et al. (2007).

3.3. Optimization of parameters for carboxymethylation

3.3.1. Effect of NaOH concentration

Fig. 1a shows the effect of NaOH concentration (0.063–0.156 M) on the DS of prepared CMC. It was noticed that DS increased with increasing the concentration of aqueous NaOH and thereafter decreased significantly. The concentration of SMCA (0.086 M), reaction time (3 h) and temperature (50 °C) was kept constant throughout the course of the reaction during the optimization of NaOH concentration with respect to DS for carboxymethylation. The DS of the products so formed has the values varied from 0.628 to 0.831. Maximum DS (0.831) was achieved with 0.094 M and 0.086 M concentrations of NaOH and SMCA respectively at 50 °C temperature for 3 h reaction time. The carboxymethylation process involves the two competitive reactions which take place simultaneously as described in Section 2.2.4. The increase in DS (0.831) is due to the predominance of Eq. (1) over its competitive Eq. (2). Above this NaOH concentration the side Eq. (2) between NaOH and SMCA becomes more substantial by consuming NaOH.
to form sodium glycolate lowering thereby the DS (0.628). These findings are in good conformity with those described in literature (Barai et al., 1997; Varshney et al., 2006).

3.3.2. Effect of SMCA concentration
The effect of SMCA concentration on the DS of CMC was evaluated by varying SMCA concentration from 0.075 to 0.119 M (Fig. 1b) at constant reaction parameters, i.e. concentration of NaOH (0.094 M), reaction time (3.0 h) and temperature (50 °C).

Fig. 1b reveals that DS increased from 0.452–1.07 by increasing the SMCA concentration. Maximum DS (1.07) was achieved at 0.108 M SMCA concentration. Increase in concentration of SMCA up to 0.108 M promotes the availability of the acid molecules in the proximity of the cellulose hydroxyls, facilitating thereby the carboxymethylation (Barai et al., 1997). No significant increase in DS occurred on further increase in SMCA concentration. Moreover, further increase in the concentration of SMCA resulted decrease in DS. This may likely be assigned to the non-availability of enough cellulose-alkoxide for reaction with SMCA (Varshney et al., 2006).

3.3.3. Effect of temperature
The effect of temperature on carboxymethylation was also studied with respect to DS (Fig. 1c). All the reactions were performed with optimized conditions for NaOH (0.094 M) and SMCA (0.108 M) at different temperatures (40–70 °C) for 3 h reaction time. Maximum DS (1.07) was achieved at 50 °C. The increase in

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Fig. 1. (a) Effect of NaOH concentration on DS (SMCA 0.108 M; temp. 50 °C; time 3.0 h). (b) Effect of SMCA concentration on DS (NaOH 0.094 M, temp. 50 °C; time 3.0 h). (c) Effect of temperature on DS (0.094 M NaOH, 0.108 M SMCA, 3 h). (d) Effect of reaction time on DS (0.094 M NaOH, 0.108 M SMCA, temp. 50 °C). (e) Effect of shear rates on the apparent viscosities of the aqueous solutions (1–3%) of the optimized CMC.
DS up to 50 °C could be linked with the advantageous effect of temperature on the swelling ability of cellulose fibers as well as diffusion and adsorption of SMCA facilitating thereby the reaction between SMCA and the cellulose molecules. Further increase in temperature resulted in a decrease in DS. Lowering of the DS beyond 50 °C could be ascribed to the degradation of carboxymethylated cellulose under the influence of atmospheric oxygen. Similar observations have been noted for the carboxymethylation of cellulose from different cutting materials (Hebeish et al., 1984; Tijsen et al., 2001; Youssef et al., 1989).

3.3.4. Effect of reaction time

The effect of reaction time on the DS of CMC was also studied (Fig. 1d). The reactions for optimization of time (1–4 h) with respect to DS were performed with 0.094 M NaOH, 0.108 M SMCA concentration at 50 °C. Fig. 1d shows the increase in DS (1.07) with respect to time, which may be due to the fact that there is a better reaction environment created and a prolonged time available for carboxymethylation. This may contribute to the better reaction efficiency and higher DS of the final product (Barai et al., 1997). Moreover, further increase in time up to 4 h resulted in a decrease in DS. The lowering of DS on prolonging the carboxymethylation time may be attributed to the atmospheric oxidative degradation of CMC. Similar behavior of reaction, on the DS of carboxymethylated flax cellulose has been reported by Hebeish et al. (1984).

DS of the products obtained under various reaction conditions was determined and its dependence on each of the variables was investigated. DS is a major factor for CMC solubility in aqueous medium, as approximately below 0.4 DS, the polymer is swellable but insoluble in water; above this the polymer is fully soluble with its hydro-affinity increasing with increasing DS (Waring and Parsons, 2001). The partial derivatization, as is frequently the case for most cellulose derivatives, including CMC, leaves unsubstituted OH groups that can interact by intra- and inter-molecular hydrogen bonding thereby limiting the solubility (Arinaitwe and Pawlik, 2014). The higher DS (1.07) of CMC under optimized parameters in present investigation is highly comparable with CMCs prepared from other underutilized cellulosic sources like Eichornia crassipes (DS, 0.24–0.73), Palm Kernel Cake (DS, 0.67), waste of cotton ginning industry (DS, 0.874) sago waste (DS, 0.33–0.82), sugar beet pulp cellulose (DS, 0.11–0.67), and Lantana weed (DS, 0.20–1.22) (Barai et al., 1997; Bono et al., 2009).

![Fig. 2. FTIR spectra of (a) optimized CMC product (DS 1.07) from MOW and (b) standard CMC product (Sigma Aldrich, USA).](image1)

![Fig. 3. (a) 1H NMR spectrum of CMC (DS 1.07) in D2O and (b) 13C NMR spectrum of CMC (DS 1.07) in D2O.](image2)
Haleem et al., 2014; Pushpamalar et al., 2006; Togrul & Arslam, 2003; Varshney et al., 2006).

3.4. Yield

In the present study the yield of CMC product obtained at the optimized conditions (NaOH-0.094 M; SMCA-0.108 M; temperature-50 °C, reaction time-3 h) was 1.508 g/g or 150.8%. The comparison of CMC product yield from MOW in the present study with the established reporting (Barai et al., 1997; Bono et al., 2009; Pushpamalar et al., 2006; Togrul & Arslam, 2003; Varshney et al., 2006) further supports the suitability of MOW paper as a tool of sustainable source for CMC products.

3.5. Rheological study

To assess the importance and feasibility of any CMC product for the application, viscosity profile is considered as one of the important parameters. Viscosity is due to friction between neighboring parcels of the fluid that are travelling at different speeds. The rheological property of 1–3% aqueous solution of the optimized CMC product (DS 1.07) was examined with regard to shear rate and apparent viscosity (η). Fig. 1e summarizes the result of shear rate of CMC on its apparent viscosity. A plot of η of the aqueous solutions (1–3%) of the optimized CMC versus shear rates (Fig. 1e) shows that η for all the solutions of the optimized CMC product rely upon the shear rate and decrease with increasing shear rate. No time effects were detected and the viscosity obtained with decreasing rate was identical with that obtained with increasing shear rate. Therefore, all the solutions of different concentration of the optimized product exhibit non-Newtonian pseudoplastic behavior. These consequences are in close conformity with the resolutions reported by Khullar et al. (2006). The carboxymethyl cellulose formed from MOW is in the range of viscosity required for commercial grade carboxymethyl cellulose. The commercial grade low viscosity CMC shows a viscosity range of 20–50 cp at 2% concentration (Bono et al., 2009), whereas CMC from MOW show a higher viscosity of 76 cp at the same concentration. The viscosity profile of CMC from MOW reveals the use of waste paper for CMC formation may be a feasible option for technocrats.

3.6. Spectroscopic study

3.6.1. FT-IR analyses

Spectroscopic techniques were further used for the characterization of synthesized CMC. An infrared spectroscopy spectrum of optimized CMC (DS 1.07) and standard CMC (Sigma Aldrich, USA) is shown in Fig. 2a and b respectively. The FTIR spectrum of both the samples was performed for the wave-numbers 800–4000 cm⁻¹. Fig. 2a shows the peaks corresponding to the backbone of the cellulose molecule were observed at 3427 cm⁻¹ (broad absorption band due to stretching of –OH groups and intermolecular and
intramolecular hydrogen bonds), 2930 cm\(^{-1}\) (C-H stretching), 1423 cm\(^{-1}\) (CH\(_3\) scissoring), 1327 cm\(^{-1}\) (OH bending) and 1062 cm\(^{-1}\) (CH-O-CH\(_2\) stretching) (Pushpamalar et al., 2006). The peak at 1614 cm\(^{-1}\) (COO\(^{-}\)) in the FTIR spectrum of CMC from waste paper confirmed the carboxymethylation. According to reported data, the peaks around wavelength 1620 and 1423 cm\(^{-1}\) represent two different functional groups in CMC (Adingragraha and Marseno, 2005). The similar trend of IR spectra in case of CMC from MOW (Fig. 2a) and commercial CMC (Fig. 2b) further supports the successful synthesis of CMC from MOW.

3.6.2. NMR analyses

The confirmation of carboxymethylation in MOW was further confirmed by NMR techniques. Fig. 3a and b shows the \(^1\)H and \(^{13}\)CNMR spectra of prepared CMC (DS 1.07) respectively. The \(^1\)H spectrum of CMC is a combination of various overlapped signals. The major seven peaks are obtained at \(\delta\) 3.17, \(\delta\) 3.31, \(\delta\) 3.62, \(\delta\) 3.76, \(\delta\) 3.90, \(\delta\) 4.06 and \(\delta\) 4.19 ppm. The information about the structure of prepared CMC could not be obtained from the \(^1\)H NMR spectrum, but the behavior and signals range obtained in \(^1\)H NMR of CMC is similar and typical as reported by previous workers for CMC (Capitani et al., 2000; Kono, 2013; Reeves et al., 2010). Due to high solution viscosity, \(^{13}\)C NMR spectrum was not recorded with high clarity, but the signals obtained in the range of \(\delta\) 62–82 ppm, \(\delta\) 102–103 ppm and \(\delta\) 177–178 ppm are the characteristic range of CMC as assigned earlier (Kamide et al., 1985; Kono, 2013). The peaks for the ring carbons at 2–5 position appeared as complex spectral lines in the region of \(\delta\) 62–82 ppm. Whereas the three signals obtained at \(\delta\) 73.50 ppm, \(\delta\) 75.20 ppm and \(\delta\) 76.10 ppm were assigned for methylene carbons (Capitani et al., 2000). The signals originated at \(\delta\) 177 ppm and \(\delta\) 178 ppm were assigned for carbonyl carbon of CMC (Capitani et al., 2000).

3.7. SEM analysis

Scanning electron microscopy was employed to investigate the surface morphology of the native MOW pulp and CMC from MOW (Fig. 4a–d). Fig. 4a and c reveals that the native MOW pulp has distinct, elongated and regular fibrous structure. Fig. 4b and d changed due to the chemical insertion and attachments of reacting species with the pulp. The fiber structure of native pulp has a regular and smooth surface with small alveolate holes and are with no defects (Fig. 4a and c), whereas after carboxymethylation the surface structure was lost completely with the loosening of alveolates holes structure and conversion into a corroded structure. The alkaline environment during the modification process accounts for the structural changes and similar morphological changes have also been reported during carboxymethylation of various polysaccharides (Bi et al., 2008; Gong et al., 2012; Kumar and Ahuja, 2012; Wang et al., 2010).

4. Conclusion

Recycling of waste paper is an important component of waste management. At present, there is growing appreciation for waste paper as a viable renewable resource. The disposal and the apt utilization of the continuously increasing volume of waste paper in a controlled and sustainable manner is a significant challenge. In this way, the present study can play an important role in advancing the technologies, which can exploit this material in an alternative viable way. In the current study, CMC was synthesized successfully from MOW paper. Maximum DS (1.07) of prepared CMC was achieved at 50°C with 0.094 M and 0.108 M concentrations of NaOH and SMCA respectively for 3 h reaction time. FTIR, NMR and SEM study confirmed the synthesis of CMC from MOW. Furthermore, the measurements of viscosity of the optimized CMC (1–3% solutions) reveal the utility of the product for commercial applications.

In this context waste paper therefore seems an attractive potential feedstock for production of water soluble Na-CMC for a variety of applications, paving thereby a way for its management and effective utilization. This clean production concept shows a prompt utilization potential for waste paper. It could achieve a reduction of investment and raw material costs with the decline in the demand of competitive and conventional raw materials like cotton fibers and wood pulp for modified cellulosic products.

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