Carbohydrate Polymers 113 (2014) 249–255

Contents lists available at ScienceDirect

Carbohydrate Polymers

journal homepage: www.elsevier.com/locate/carbpol

Synthesis of carboxymethyl cellulose from waste of cotton ginning industry

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ARTICLE INFO

Article history:
Received 12 May 2014
Received in revised form 4 July 2014
Accepted 6 July 2014
Available online 17 July 2014

Keywords:
Cellulose
Cotton gin waste
Bleaching
CMC
Biowaste
Etherification

Chemical compounds studied in this article:
Carboxymethyl cellulose (PubChem CID: 6328154)
Monochloroacetic acid (PubChem CID: 300)
Ethanol (PubChem CID: 702)
Methanol (PubChem CID: 887)
Sodium hydroxide (PubChem CID: 14798)
Isopropanol (PubChem CID: 3776)
Hydrochloric acid (PubChem CID: 313)
Nitric acid (PubChem CID: 944)
Sulphuric acid (PubChem CID: 1118)
Glacial acetic acid (PubChem CID: 176)

ABSTRACT

The aim of present work was to isolate cellulose from cotton gin waste (CGW) and synthesis of carboxymethyl cellulose (CMC) from it. Scoured and bleached CGW was used to investigate the effects of temperature, reaction time, acid-base concentration on the physiology of the resultant cellulose polymer. The isolated cellulose from CGW was converted to CMC by etherification using sodium monochloroacetic acid and different sodium hydroxide (NaOH) concentrations (5–40 g/100 mL) were tested to get high quality product. The optimum condition for carboxymethylation was found to be 20 g/100 mL NaOH which provided the highest viscosity and degree of substitution (DS = 0.874). Isolated cellulose and CMC were characterized using Fourier transform infrared spectroscopy (FT-IR) and scanning electron microscopy (SEM). FT-IR analysis revealed that the produced cellulose was of very good quality. Furthermore, X-ray diffraction (XRD) analysis spotlighted crystalline nature of cellulose. SEM images showed rough structure of cellulose while that of the CMC had a smooth surface. This optimized method will be tested at pilot scale in collaboration with local industry.

1. Introduction

On the surface of earth, cellulose is the most abundant renewable organic material. It constitutes about 33% of all plants' matter and the major component of cotton (over 94%) and wood (over 50%) (Klemm, Heublein, Fink, & Bohn, 2005). Cotton fiber is mostly comprised of cellulose with several non-cellulosic components surrounding the cellulose core. These non-cellulosic components are mostly originated in the cuticle layer and the primary wall, which surrounds cotton fiber (Chung, Lee, & Choe, 2004). Generally, cotton fibers are composed of cellulose, hemicellulose, lignin, pectins, proteinaceous matter, waxes and minor soluble polysaccharides (Gumuskaya, Usta, & Kirci, 2003). Cellulose is organized in to fibrils, which are surrounded by a matrix of lignin and hemicellulose (Kaushik & Singh, 2011). Recently, with increased tipping price and strict regulations on particulate emissions, there is rehabilitated interest in recovery of biomaterials (Glew, Stringer, & McQueen-Mason, 2013) and producing valuables such as ethanol from solid organic wastes (Jeoh & Agblevor, 2001).

The composition of cotton gin waste (CGW) in Indo-Pak subcontinent is different from that of developed countries. Cotton in Pakistan is picked by human hands and the harvest is contaminated by leaf fragments during picking and dust during storage. About 40–147 kg of cotton gin waste is produced per bale of cotton (227 kg). Cotton linters from the ginning waste can serve as the raw

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http://dx.doi.org/10.1016/j.carbpol.2014.07.023

material for cellulose isolation and synthesis of cellulose derivatives. Various methods have been developed to isolate cellulose from biomass i.e., steam explosion (Chen & Liu, 2007), Organosolv process (Sun, Sun, Zhao, & Sun, 2004), chlorine free method (Sun, Xu, Geng, Sun, & Sun, 2005), pooled chemical and enzymatic extraction (Reddy & Yang, 2006) and ionic liquids method (Jiang et al., 2011). Air in cotton causes tremendous degradation of the cellulose under hot alkaline conditions by the peeling reaction or hydroxyl carbonyl elimination (Knill & Kennedy, 2003). Alkaline chemical modifications, such as the common mercerization progression used for cotton fibers, have been recognized to improve both cellulose fibers because of the increase in the crystallinity (El-Sakhawy & Hassan, 2007). Alkali treatment also affects the molecular orientation of cellulose crystallites due to the removal of lignin and hemicelluloses (Kim & Netravali, 2013).

In order to increase the cellulose applicability, an unconventional pathway is to convert the cellulose to its derivatives such as carboxymethyl cellulose (CMC). CMC has many applications throughout the food industry, detergents, cosmetics, pharmaceuticals, textiles, paper, adhesives and ceramics (Yang & Zhu, 2007). CMC is a water soluble derivative of cellulose with β-D-glucose and β-D-glucopyranose 2-O-(carboxymethyl)-monosodium salt which are connected via β-1, 4-glycosidic bonds. It also acts as a viscosity modifier, thickener and emulsifier (Ninan et al., 2013).

Many researchers have studied the production of CMC from agricultural waste cellulose sources such as sugar beet pulp (Togrul & Arslan, 2003), cashew tree gum (Silva et al., 2004), sago waste (Pushpamalar, Langford, Ahmad, & Lim, 2006), Cavendish banana pseudo stem (Adinugraha, Marseno, & Haryadi, 2005) and durian rind (Rachtanapun, Luangkamin, Tanprasert, & Suriyatem, 2012). However, to date, to the best of our knowledge, no research has described the isolation of cellulose from cotton gin waste and then conversion of the isolated cellulose to CMC. In this context, the first objective was the isolation of cellulose from cotton gin waste and the second one was to convert it into CMC followed by characterizations of the both.

2. Materials and methods

2.1. Chemicals

Chemicals used in this research i.e., sodium hydroxide (NaOH) 99%, hydrogen peroxide (H_2O_2) 99%, sulfuric acid (H_2SO_4) 98%, hydrochloric acid (HCl) 37%, glacial acetic acid ($C_2H_4O_2$) 99% and monochloroacetic acid ($ClCH_2CO_2H$) 99.0%, all manufactured by Sigma (Sigma-Aldrich Co.), were purchased from local supplier. Nitric acid (HNO_3) (65%) was purchased from Merck, Germany. Isopropyl alcohol (99.8%), ethanol (99.08%) and methanol (99.9%) were taken from Scharlau Chemie (Scharlau Chemie S.A). All chemicals used in this research were of analytical grade.

2.2. Cotton gin waste and cellulose isolation procedures

CGW used in this study was obtained from the cotton ginning factory at Rahim Yar Khan (Southern Punjab—Cotton growing area), Pakistan and isolation of cellulose was carried out adopting the following recipes of reagents summarized in Table 1.

2.2.1. Cel–01: isolation of cellulose from cotton gin waste in 20% (wt) NaOH

The cotton gin waste was treated with 20 wt% NaOH solution at 70 °C for 2 h. Then it was washed with distilled water to remove NaOH. The scoured sample was bleached with 1.5 wt% H_2O_2 in alkaline medium (pH 10) at 70 °C. The pH of the sample was adjusted at 7 by adding dilute HCl. The bleached sample was washed and dried in oven. The dried sample was treated with 10 wt% H_2SO_4 at 70 °C till the long chain of cellulose polymer is converted to oligomers and white suspension is formed. The white suspension of oligomers is filtered and dried. This is the white powder of cellulose in the form of oligomers.

2.2.2. Cel–02: isolation of cellulose from pretreated cotton gin waste with 2 wt% H_2SO_4

The cotton gin waste was treated with 2 wt% H_2SO_4 for 1 h at 70 °C. The treated cotton gin waste was washed several times with distilled water. The sample was treated with 25 wt% NaOH solution at 70 °C for 2 h. Then the sample was treated as reported in cf. 2.2.1.

2.2.3. Cel–03: isolation of cellulose from pretreated cotton gin waste with 1.5 wt% HCl

The sample was heated in 1.5 wt% HCl for 1 h at 75 °C. After washing, the sample was treated with 25 wt% NaOH solution at 70 °C for 2 h. Then the washed cotton gin waste was treated as reported in cf. 2.2.1.

2.2.4. Cel–04: isolation of cellulose from pretreated cotton gin waste with 5 wt% H_2SO_4

The cotton gin waste was treated with 5 wt% H_2SO_4 for 1 h at 80 °C. This sample was treated with 10 wt% detergent solution for 1 h. The cotton gin waste was washed several times with distilled water. The washed cotton gin waste was treated as reported in cf. 2.2.1.

2.2.5. Cel–05: isolation of cellulose from pretreated cotton gin waste with 10 wt% detergent

The cotton gin waste was treated with 10 wt% detergent solution at 70 °C for 1 h. Then it was washed several times with distilled water. The resulting cotton was immersed in 6% NaOH solution for 2 h at 70 °C and washed. Then it was treated as reported in cf.2.2.1.

2.3. Conversion of isolated cellulose into CMC

Isolated cellulose from cotton gin waste was then converted to CMC in two steps: alkalization and etherification of cellulose under heterogeneous conditions. In the first step about 5.0 g of CGWs cellulose powder was weighed and added to 500 mL Schott bottle and followed by 300 mL of water: isopropyl alcohol solvent (1:4) in appropriate ratio. Then, 40 mL of various Concentrations (5, 10, 15, 20, 25 and 30%) w/v sodium hydroxide added drop-wise and stirred for 2 h. The cellulose–NaOH activation reaction is often referred to as mercerization and it is generally performed at approximately room temperature. The carboxymethylation reaction was started by adding varying amounts of sodium monochloroacetic acid (MCA) (1, 2, 3, 4 and 5 g) to the reaction mixture placed on magnetically stirrered hot plate. The reaction mixture was heated up at various temperatures (40–60 °C) with constant stirring for 3 h. The mixture was then filtered and the residue was suspended in 100 mL of methanol for 40 min. The suspended slurry was then neutralized using dilute glacial acetic acid. The residue was filtered again and washed with absolute methanol. The residue from the filtration was dried in hot air oven at 60 °C overnight and the powder obtained was CMC.

2.4. Characterization of isolated cellulose and CMC produced

2.4.1. Fourier transform infrared (FT-IR) spectroscopy

Fourier transform infrared (FT-IR) spectra were collected using spectrophotometer (Spectrum 100, Perkin Elmer FT-IR). Powder samples of cellulose were mixed with KBr (2:100, mg w/w) and forced into transparent disks by hydraulic machine. The spectra were measured in the transmittance mode from an accumulation of 28 scans at a 4 cm^{-1} resolution over 4000–500 cm^{-1} range,

Table 1
Detailed description of the recipes used during the isolation of cellulose.

Name	Pretreatment			Scouring NaOH (%)	Washing temp (°C)	Bleaching H_2O_2:NaOH	Treatment with H_2SO_4 (%)
	HCl	H_2SO_4	Detergent				
Cel-01	Nil	Nil	Nil	20	70	1.5:3	Nil
Cel-02	Nil	2%	Nil	25	70	1.5:3	10
Cel-03	1.5%	Nil	Nil	25	70	1.5:3	10
Cel-04	Nil	5%	10%	20	80	1.5:3	10
Cel-05	Nil	Nil	10%	6	80	1.5:3	10

as reported in literature (Gumuskaya et al., 2003). The *x*-axis represents the wavelength (cm^{-1}) and *y*-axis shows the light transmittance through the sample. The cellulose samples were analyzed by FT-IR to define the functional group of isolated cellulose. In the most basic terms, the infrared spectrum were produced as a result of absorption of electromagnetic radiation at frequencies that correlate to the vibration of particular sets of chemical bonds within a molecule (Elanthikkal, Gopalakrishnapanicker, Varghese, & Guthrie, 2010). The CMC samples were analyzed by FT-IR to define the functional group of synthesized CMC.

2.4.2. Scanning electron microscope (SEM)

The surface morphologies were examined by SEM (Jeol JSM-6490A, Japan Analytical scanning electron microscope). Samples were coated with a thin layer of conducting material (gold) and imaged at ×150 and ×5000 magnifications with 10–15 kV accelerating voltage**.** A focused high energy beam of electron interacted with the surface of sample and generated secondary electron, back scattered electron and characteristic X-rays signals. These signals were perceived by the detector and images were displayed on the cathode ray tube screen.

2.4.3. X-ray diffraction (XRD)

Cellulose samples obtained by treating CGW with different concentration of alkali were analyzed by X-ray diffraction to check the crystalline structure of cellulose. X-Ray diffractometer (Theta/Theta STOE Jeol Germany) was used to obtain X-Ray diffraction patterns of isolated cellulose. Samples were prepared by pressing the powders between two glass slides into a flattened sheet. The X-Ray patterns were taken by using radiation source CuKα by supplying 40 kV and 40 mA to X-ray generator. The patterns were recorded at 2θ from 20° to 80° (Kim, Eom, & Wada, 2010).

2.5. Determination of degree of substitution of CMC

The degree of substitution (DS) of carboxylic group in carboxymethyle cellulose is the average number of hydroxyl group in the cellulose structure which was substituted by carboxymethyl groups at C2, 3 and 6. Absolute values of degree of substitution (DS) were determined by potentiometric titration. One gram of CMC was weighed and added to 250 mL beaker followed by 50 mL of 95% ethanol and stirred. Then, 5 mL of 2 M nitric acid was added and the mixture stirred for 10 min at room temperature. The mixture was then heated to boiling by using magnetic hot plate for 5 min and stirred further for 20 min and left to settle. After the solution had settled, the solution was filtered and the residue was washed with 100 mL 95% ethanol until the acid and salts were removed. The precipitate was washed with methanol and transferred to beaker and heated until the alcohol was removed. The beaker with the precipitate was dried in the oven at 90 °C for 3 h. Carboxylmethyl cellulose (0.5 g) was weighed in 250 mL Erlenmeyer flask and 100 mL distilled water added and stirred. Twenty-five milliliter 0.5 M sodium hydroxide was added and boiled for about 20 min. Then the heated solution was titrated with 0.3 M HCl by using phenophthalein as an indicator to observe the color change from Mexican pink (dark pink) to colorless. The DS of CMC can be calculated using the equations below (Jiang et al., 2011).

$$A = \frac{BC - DE}{F}$$

$$\text{Degree of Substitution} = \frac{0.162 \times A}{1 - (0.058 \times A)}$$

where
A = milli-equivalents of consumed acid per gram of specimen;
B = volume of NaOH added;
C = concentration of NaOH added;
D = volume of consumed HCl;
E = concentration of HCl used;
F = specimen grams used;

162 are the molecular weight of the anhydrous glucose unit and 58 is the net increase in the anhydrous glucose unit for each substituted carboxymethyl group.

3. Results and discussion

3.1. Isolation of cellulose

3.1.1. Effect of scouring on cellulose isolation

NaOH was the key chemical reagent implicated for lignin removal. Among various concentrations (6, 10, 15, 20, 25 and 30% w/v) of NaOH tested at 70–80 °C under normal pressure, 25% of NaOH was observed to be ideal for removing maximum lignin (Table 1). Cel-02 and Cel-04 represented superior results during the entire process. The higher percentage of cellulose was yielded by utilization of 25% NaOH, while higher concentration beyond this treatment led to cellulose degradation. Alkali pretreatment is regarded as an efficient pretreatment method for removing lignin, pectin, waxes and hydrophobic impurities from cotton gin waste and concentrations used in this experiment are in agreement with other research outputs (Binod et al., 2012). Alkali treatment also affects the molecular orientation of cellulose crystallites due to the removal of lignin and hemicellulose. It also increases surface roughness, resulting in better mechanical interlocking and the amount of cellulose exposed on the fiber surface (Mohanty, Misra, & Drzal, 2001). Scouring of cotton gin waste increased strength to give better absorptive properties, and generally, a high degree of luster (Aydin, Tozlu, Kemaloglu, Aytac, & Ozkoc, 2011).

3.1.2. Effect of bleaching on cellulose extraction

Hydrogen peroxide (H_2O_2) was utilized in the bleaching process due to its least hazardous and oxidative damaging potential. As Cel-01 was not pre-treated, the white pulp produced was not entirely free from impurities during the isolation process. Therefore, the extraction of pure cellulose was not up to standard, whereas, dishwashing detergent was used for pretreatment in Cel-04 and Cel-05. Hence, Cel-04 sample (scoured with 20% NaOH at 70 °C) yielded superior quality product, whereas, Cel-05 resulted in insufficient quality because scouring sample contained only 6%

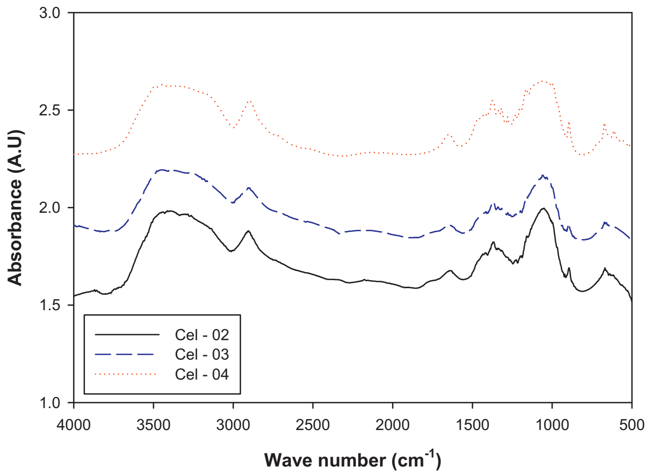

Fig. 1. FT-IR spectra of isolated cellulose from different experiments. Cel-02, Cel-03 and Cel-04 represent different set of conditions.

NaOH. Moreover, the normal white color of the pulp was also lost, which was not the case with other samples. The results thus propagate that bleaching effect could be enhanced by alkali application in the process, which has also been reported earlier (Sette, Boullart, & Kiekens, 1995). The optimum condition for bleaching of cellulose sample was 1.5% hydrogen per oxide (H_2O_2) and 3% NaOH. These optimum conditions escort to completion of the bleaching process in sensible duration of 2 h without evolution of harmful gas. Lower concentrations of the H_2O_2 were found to result into unsatisfactory bleaching while on the other hand, addition of high peroxide could result into high cost. The precipitous increase of the hydrogen per oxide (H_2O_2) can attribute to higher degradation of cellulose than to removal of lignin.

3.1.3. *Effect of temperature and acid on cellulose extraction*

Samples treated with different concentrations (2, 5 and 10%) of H_2SO_4 revealed that concentration of 10% H_2SO_4 was ideal for this procedure. It was observed that yield of cellulose was inversely proportional to temperature and treatment time at all alkali concentrations. We observed that there were some significant effects on amorphous cellulose, forming spatially localized acid hydrolysis during lignin and hemicellulose removal. This spatially localized degradation manifests as a change in cellulose crystallinity. As the concentration of acid increased up to 10% H_2SO_4 for pretreated bleached samples, there was increase in white powder formation. Other researchers (Foston, Hubbell, & Ragauskas, 2011) have observed similar effects with acid treatment time for hemicellulose removal. At high concentration of acid (>10%), the degradation of cellulose is reported by (Adel, El-Wahab, Ibrahim, & Al-Shemy, 2010).

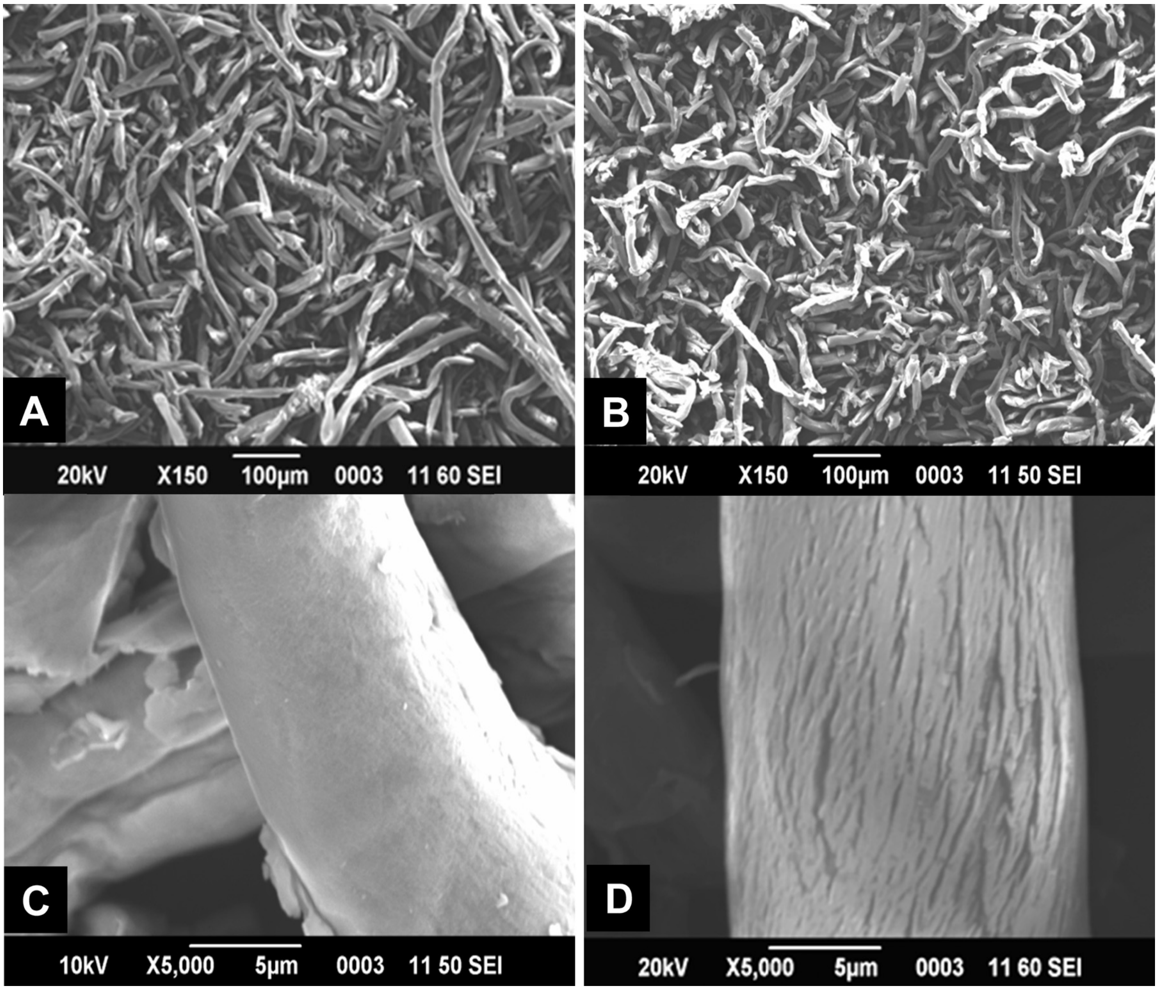

Fig. 2. Morphological analysis of cellulose by scanning electron microscope (SEM). A—Cel-04 and B—Cel-02 are at ×150 magnification. C and D are at higher magnifications (×5000) of Cel-04 and Cel-02. D—Cel-02 show degradation of fibers due to high NaOH (25%) as compared to C—Cel-04 where NaOH concentration was 20% and the surface is comparatively smooth.

Table 2
Assignment of the main absorption wavelengths for FT-IR spectra of isolated cellulose.

Positions			Assignment
Cel-02	Cel-03	Cel-04	
3438	3440	3411	O—H stretching (hydrogen bond)
3284	3263	3283	O—H bending
2903	2903	2903	C—H stretching of alkane
1638	1643	1630	Bending mode of absorb water
1414	1415	1420	C—C—C bending
1365	1372	1365	C—O—H bending
1050	1001	1050	C—O pyranose ring skeletal vibration
894	893	894	O—H bending
669	671	669	C—H rocking

3.2. Characterization of isolated cellulose

3.2.1. Spectroscopic characterization by FT-IR

Fig. 1 demonstrates FT-IR spectra of Cel-02–04 isolated from CGW. The spectra spotlighted the extent of pure cellulose. The extraction procedures removed most of the non-cellulosic materials as the disappearance of the lignin took place at 1600 cm^{-1} and 1510 cm^{-1} absorbance. Likewise, O—H stretching (hydrogen Bond) materialized at 3438 cm^{-1}, 3440 cm^{-1}and 3411 cm^{-1} absorbance in Cel-02, Cel-03 and Cel-04, respectively. The band at 2903 cm^{-1} was due to C—H stretching and is at 1417 cm^{-1} absorbance, reflecting the bending of C—C—C. The Cel-02 and Cel-04 showed C—O—H bending at 1365 cm^{-1} absorbance and comparable bending was observed at 1372 cm^{-1} absorbance in Cel-03. Furthermore, complete description of the absorption of isolated cellulose is summarized in Table 2. The FT-IR spectrum obtained during this study was analogous to the spectrum reported previously (Sun et al., 2004).

3.2.2. Morphological analysis by SEM

The SEM micrographs of the isolated cellulose are shown in Fig. 2 at different magnifications and fragments of various lengths were characterized (Fig. 2A—Cel-04 and B—Cel-02). At higher magnifications (Fig. 2C—Cel-04 and D—Cel-02), completely round and fibrils of various thickness were revealed. However, thickness of the fibrils was reduced in Cel-04 as compared to Cel-02. This observation is in agreement that fiber diameter reduces as the concentration of alkali solution is increased from 25% to 30% (Aydin et al., 2011). This is because during this treatment the intermolecular bond of cellulose are broken and polyhydroxyl group of polysaccharide result into swelling of the fiber which decreases fiber length. SEM analysis of the samples showed that the packed structure of the fibrils was distorted by the removal of non-cellulosic materials. The fibrils were separated from each other and the surface roughness of the alkali treated fibers was improved (Aydin et al., 2011).

3.2.3. X-ray diffraction (XRD) analysis

X-ray scattering techniques are a family of non-destructive analytical techniques which reveal information about the crystallographic structure, chemical composition, and physical properties of materials and thin films. Fig. 3 illustrates two sharp peaks of cellulose in Cel-02 method at $2\theta = 20^\circ$ and 22°, while identical peaks were observed for Cel-03 and Cel-04. This pattern is characteristic of cellulose and is typical of cellulose isolated from other fiber sources (Shogren, Peterson, Evans, & Kenar, 2011).

3.3. Conversion of cellulose to CMC

The yield of CMC product in this work was 1.437 g/g. Product yield was calculated based on dry weight foundation. The lattice dry weight of carboxymethyl cellulose was divided by 5 g of cellulose to obtain the yield value.

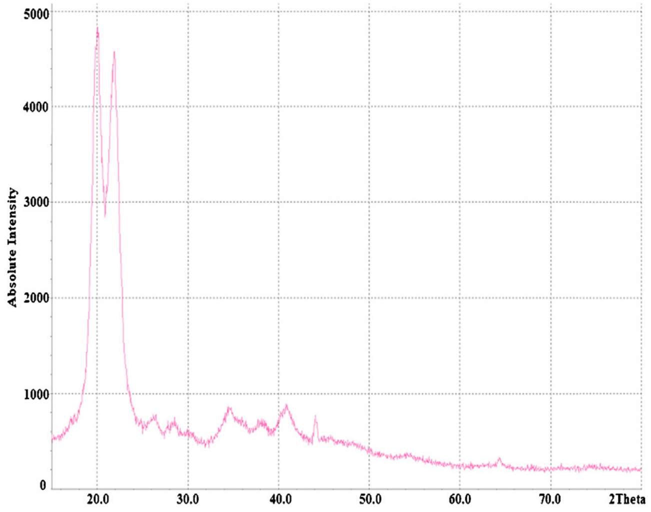

Fig. 3. Crystalline structure of cellulose by X-ray diffraction. The peaks appeared are of typical cellulose as confirmed from by the literature.

$$\text{Product yield} = \frac{\text{Weight of dried CMC}}{\text{Dry weight of cellulose}}$$

3.3.1. FT-IR characterization of CMC

The FT-IR Spectra of CMC (Fig. 4) are similar to the spectra reported previously (Kaushik & Singh, 2011). The FT-IR spectrum of the sample showed that the carboxymethyl and hydroxyl functional groups are found at wavelength of 1620, 1423 and 1328 cm^{-1} respectively. It is obvious that the broad absorption band at 3423 cm^{-1} is due to stretching frequency of hydroxyl group (—OH). The band at 2920 cm^{-1} is due to carbon–hydrogen (C—H) stretching vibration. The presence of strong absorption band at 1620 cm^{-1}, confirmed the presence of COO$^-$. The bands around 1423 and 1328 cm^{-1} are assigned to —CH$_2$ scissoring and hydroxyl group (—OH) bending vibration, respectively. The band at 1054 cm^{-1} is due to >CH—O—CH$_2$ stretching.

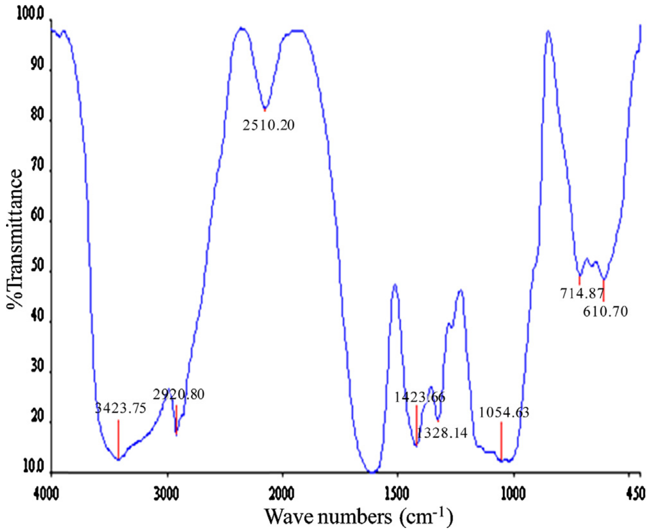

Fig. 4. FT-IR spectrum of carboxymethyl cellulose.

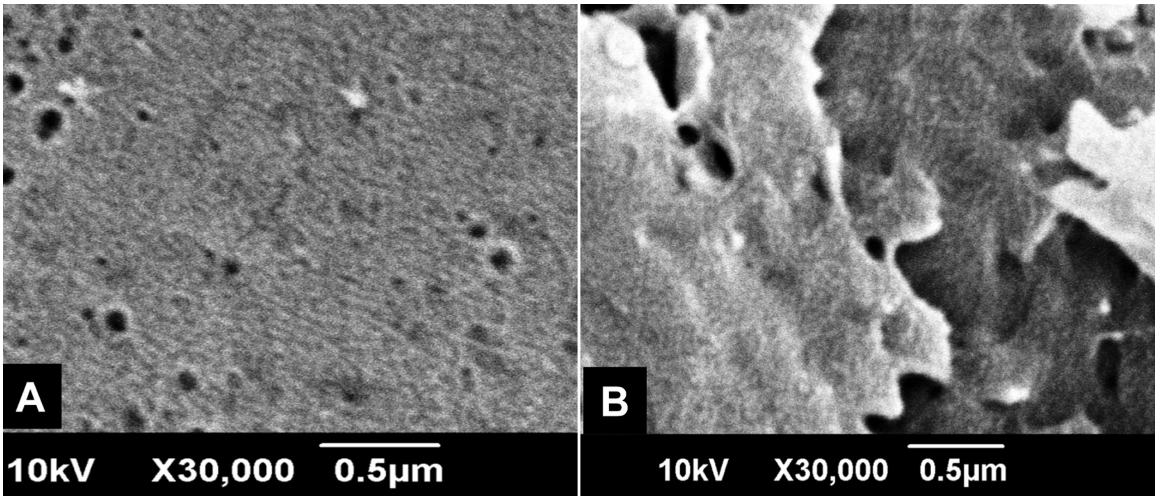

Fig. 5. Morphological analysis of carboxymethyl cellulose by SEM. A—CMC image at ×30,000 with 20% NaOH representing smooth structure as compared to B where NaOH concentration was 30% and the structure was rough.

3.3.2. Morphological analysis of CMC by SEM

Fig. 5 illustrated the SEM micrograph of various CMC powdered samples. The micrographs were prepared from the CMC samples with 10%, 20% and 30% NaOH concentration. The optimum NaOH solution for uniform product of CMC was 20 percent NaOH. The external surface of the CMC samples revealed that the cellulose fibers were methylated intensely. In the next step of the reaction, etherification was carried out with MCA as the organic acid that was replaced by substitution of NaOH on cellulose fibers. NaOH reacts with hydroxyl groups to form alkoxide. The ratio of NaOH/MCA is critical for etherification. It affects degree of substitution but very high concentration of NaOH may lead to formation of other by products (Bi, Liu, Wu, & Cui, 2008). Use of isopropanol as solvent was very efficient in the preparation of the CMC as a desirable product with high DS (0.874). Isopropanol has been reported in literature a very good reaction solvent (Bi et al., 2008; Tijsen, Kolk, Stamhuis, & Beenackers, 2001).

3.3.3. Degree of substitution

3.3.3.1. Effect of various NaOH concentrations on degree of substitution (DS) of CMC. The effect of sodium hydroxide concentration was studied by varying the concentration of the sodium hydroxide solution from 5% to 40% (Fig. 6). It was observed that the DS of CMC increased with sodium hydroxide concentration and attained a maximum DS of 0.874 at an alkali concentration of 30 mL of 20% (w/v). The increase in the DS of CMC improved the ability of CMC to immobilize water in a system. At particular alkali concentration, the DS was maximum after which it started declining. Beyond 20% of NaOH concentration, the DS was decreased, probably due to the degradation of cellulose structure and glycolate formation leading to inactivation of monochloroacetate and its utilization by this side reaction. Similar observation has reported in literature (Pushpamalar et al., 2006).

3.3.3.2. Effect of various amounts of sodium monochloroacetate on DS. The effect of concentration of sodium monochloroacetate was also studied on DS of CMC. A maximum DS of 0.874 was obtained with 5.0 g of sodium monochloroacetate. There was an increase in the DS with amount of sodium monochloro acetate up to 5.0 g. The increase probably was due to the greater availability of the acetate ions at higher concentrations in the closeness of cellulose molecules. At a concentration higher than 5.0 g, glycolate formation seems to be preferential and the reaction efficiency decreases (Ren, Sun, & Peng, 2008).

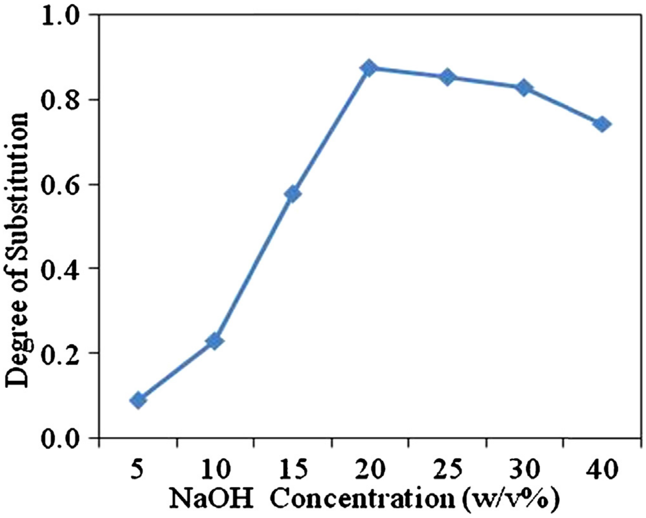

Fig. 6. Effect of NaOH concentration on degree of substitution of carboxymethyl cellulose.

4. Conclusion

A multi-step process for isolation of cellulose from CGW was optimized. The cellulose obtained from CGW using refined protocol was of high quality, with an increased crystallinity, representing closely packed polymer chains which do not dissolve in H_2SO_4. The chemical configuration of cellulose was confirmed by FT-IR, SEM and XRD. Hydrolysis of the cellulose fibers with 10% (w/v) H_2SO_4 for 1 h at 70–80 °C was observed to be optimum because it resulted in the removal of most of the amorphous domains without any significant damage to the crystal structure. The isolated cellulose from CGW was successfully converted to CMC by etherification using sodium monochloroacetic acid and NaOH. The optimum concentration for carboxymethylation was found to be 20 g/100 mL. The resulted CMC showed high degree of substitution (DS = 0.874). This CMC can be further used in different industrial processes to manufacture food products, cosmetics, pharmaceutics, ceramics, etc. To develop this method for the industry, pilot scale production will be done in collaboration with Shafi Reso-Chem, Lahore, Pakistan–a local industry desirous of producing CMC on commercial scale.

Acknowledgments

The authors would like to thank the directorate of research, National University of Sciences and Technology (NUST), Islamabad, Pakistan for providing financial support to carry out this research. We are also thankful to Shafi Reso-Chem, Lahore for their support to perform this work. The authors also thank Dr. Nauman Ul Haq, School of Chemical and Material Engineering–NUST for very valuable suggestions and critical comments in preparing this manuscript.

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