



# Synthesis of carboxymethyl cellulose from waste of cotton ginning industry



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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.

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## 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, & Kirici, 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 sub-continent 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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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  $\beta$ -D-glucose and  $\beta$ -D-glucopyranose 2-O-(carboxymethyl)-monosodium salt which are connected via  $\beta$ -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 stirred 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,

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