Conversion of waste derived cellulose to Carboxymethyl cellulose (CMC)

A dissertation submitted in the partial fulfillment of
the requirement for the degree of
Bachelor of Science
in
Chemistry

Submitted by:
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DECLARATION

I declare that the thesis entitled **“Conversion of waste derived cellulose to Carboxymethyl cellulose (CMC)”** has been prepared by me under the supervision of **Dr. Shailey Singhal and Dr. Shilpi Agarwal** from **Chemistry, Applied Science Cluster, School of Advanced Engineering, UPES, Dehradun, India.**

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CERTIFICATE

I certify that, **Ashmeet Bhogal** has prepared his project entitled **“Conversion of waste derived cellulose to Carboxymethyl cellulose (CMC)”** for the award of **B.Sc. (Hons) Chemistry,** under our guidance. He has carried out the work at the **Chemistry, Applied Science Cluster, School of Advanced Engineering, UPES, Dehradun, India.**

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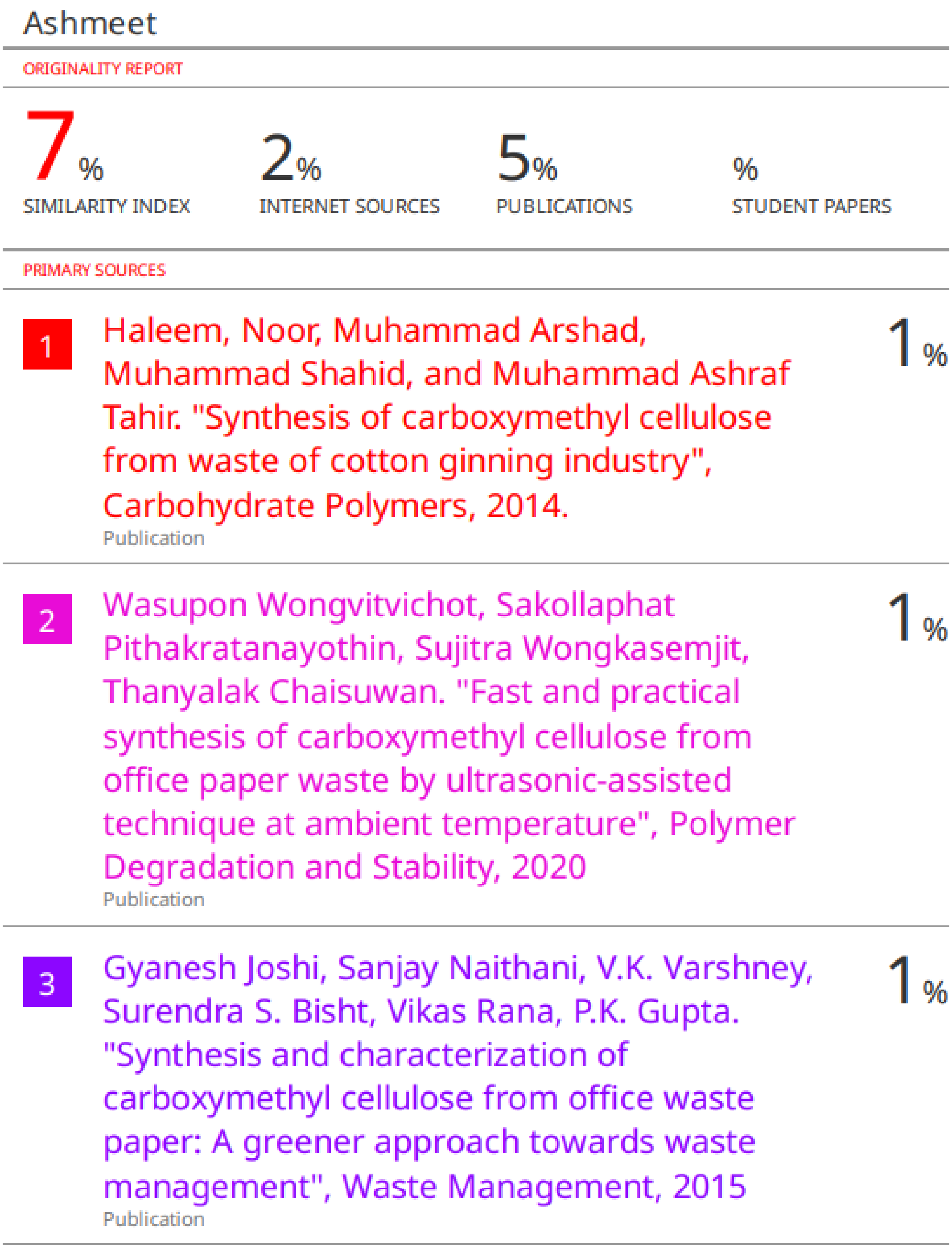

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Abstract

This research focuses on the sustainable synthesis of carboxymethyl cellulose (CMC) from waste-derived cellulose, specifically waste paper and waste textiles. The study aims to optimize the reaction conditions, chemical modification, characterization and best processes to produce high quality CMC. The method involves collecting and sorting samples of waste paper and textiles, by the cellulose-containing material through shredding, grinding, and purification steps, followed by alkalization and etherification reactions to produce CMC. Various process parameters such as NaOH concentration, reaction temperature and reaction time are optimized to achieve maximum efficiency and desired product characteristics. Characterization techniques including FTIR (Fourier transform infrared spectroscopy), XRD (X-ray diffraction), degree of substitution determination and melting point analysis were used to analyse the chemical composition, crystalline nature, degree of substitution and thermal stability of the synthesized CMC. The results indicate successful extraction of CMC from waste materials, which has similar characteristics to commercial CMC and literature references. Key findings include the potential of waste paper and waste textiles as viable feedstock for CMC production. In conclusion, this research demonstrates the feasibility and sustainability of CMC production from waste-derived cellulose, paving the way for future optimization, scalability, and exploration of additional feedstock varieties for CMC synthesis.

Keywords: cellulose, carboxymethyl cellulose (CMC), mono-chloroacetic acid (MCA), waste paper, waste fabric

ACKNOWLEDGEMENT

In fact, there are many people for whose contribution I must express my gratitude towards the successful completion of my dissertation.

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Ashmeet Bhogal

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CHAPTER 1
INTRODUCTION

1. Introduction to Carboxymethyl Cellulose (CMC)

Cellulose, an abundant natural polymer found in plant cell walls, consists of repeating glucose units linked by β(1→4) glycosidic bonds, making it the most prevalent organic compound on our planet.
Carboxymethyl Cellulose (CMC) is derived from cellulose through chemical alteration. This process involves adding carboxymethyl groups ($-CH_2COOH$) to the cellulose structure, leading to water solubility and various beneficial characteristics.

Structure of CMC

- The structure of CMC is represented by [$C_6H_{10}O_5$] n,
- where the backbone is composed of D-glucose units linked through -1,4-linkages.
- Carboxymethyl groups ($-CH_2-COOH$) are attached to certain hydroxyl groups of the glucopyranose monomers in the cellulose backbone.
- In practical applications, CMC is commonly utilized in its sodium salt form, known as sodium carboxymethyl cellulose.

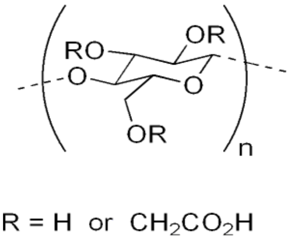


***Fig1:** Structure of CMC*

Reaction for converting cellulose to CMC

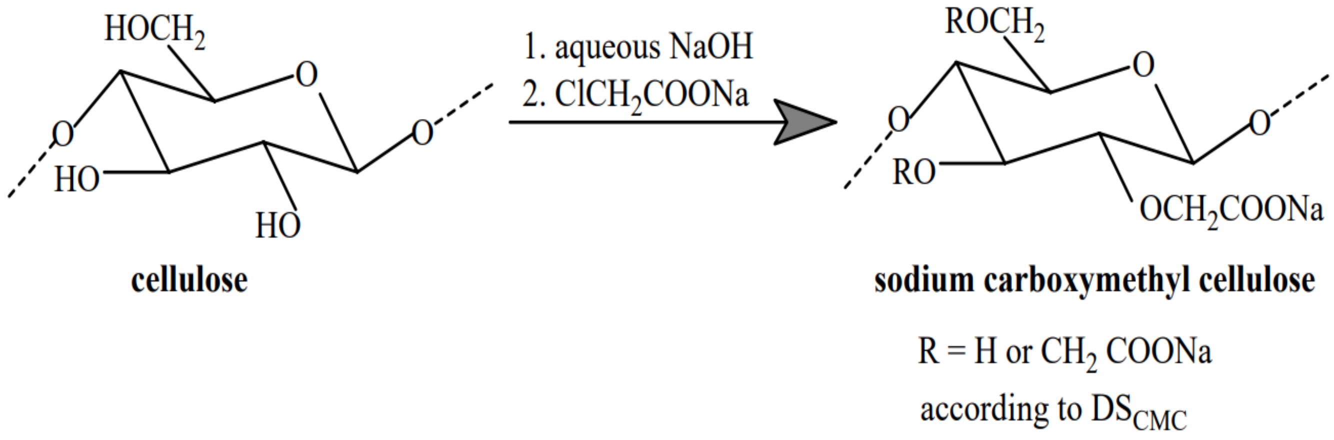


Fig2: *Reaction for converting cellulose to CMC* (Pushpamalar, 2005)

Properties of CMC

- CMC is a white or light-yellow powder with no odor, taste, or toxic properties.
- It absorbs moisture from the air and dissolves easily in hot or cold water, forming a thick solution.
- CMC does not dissolve in organic solvents like methanol, ethanol, acetone, chloroform, or benzene.
- Its properties, such as viscosity and stability, depend on the degree of substitution in the cellulose structure, chain length, and arrangement of carboxymethyl groups.
- It's commonly used as a thickener and stabilizer in various products, both food and non-food, due to its high viscosity, safety, and hypoallergenic nature.

Potential Applications of CMC

CMC is widely used across industries for its versatility as a thickener, stabilizer, film-former, binder, and emulsifier. It finds applications in food, pharmaceuticals, textiles, paper products, cosmetics, and various industrial processes.

Food industry: CMC is used as a thickener, stabilizer, emulsifier, and gelling agent in food products (e.g., sauces, dressings, dairy products, desserts).

Pharmaceutical industry: CMC serves as a binder, disintegrant, film-former, and viscosity modifier in tablet formulations, oral suspensions, topical creams, and controlled-release formulations.

Textile industry: CMC is utilized in textile printing, dyeing, sizing, and finishing processes as a thickener, binder, and anti-migration agent.

Paper industry: CMC enhances paper strength, surface properties, printability, and ink absorption, leading to improved paper products (e.g., packaging materials, specialty papers, coatings).

Cosmetics and personal care industry: CMC is found in skincare products, hair care formulations, oral care products, and cosmetics as a thickener, stabilizer, and texture modifier.

Industrial applications: CMC is used in adhesives, coatings, paints, detergents, drilling fluids, ceramics, and construction materials for its rheological properties, water binding capacity, and film-forming characteristics.

The versatility of CMC makes it a valuable ingredient in product formulations, providing functionalities such as viscosity control, moisture retention, adhesion, and film formation.
(Pettignano, 2019)

2. Review of literature

The synthesis of CMC from various cellulose sources has been extensively studied in the scientific literature. Research has explored CMC production from agricultural residues (e.g., sugarcane bagasse, corn husks), wood residues (e.g., sawdust, pulp), cotton fibers, and other biomass sources. Different extraction methods, chemical modification techniques, and process parameters have been investigated to optimize CMC yield, purity, and properties.

Steps for conversion of cellulose to CMC –

These methods are chosen based on factors such as efficiency, environmental impact, and the properties desired in the resulting CMC product. Some common methods for this conversion include:

Ethanol-Isobutyl Alcohol Mixture as Solvents: Involves using a mixture of ethanol and isobutyl alcohol as solvents in the carboxymethylation reaction.

Alkaline Hydrolysis and Esterification: Cellulose is hydrolyzed under alkaline conditions followed by esterification to obtain CMC.

Acetylation Followed by Carboxymethylation: Cellulose is first acetylated and then carboxymethylated to produce CMC.

Etherification Reaction: Involves reacting cellulose with chloroacetic acid in the presence of alkali to form carboxymethyl cellulose.

Fast and Practical Synthesis: A rapid synthesis method often involving ultrasonic assistance or other energy-efficient techniques.

Studies have demonstrated the feasibility of using alternative cellulose sources for CMC synthesis, highlighting the potential for waste materials utilization and sustainable production practices.

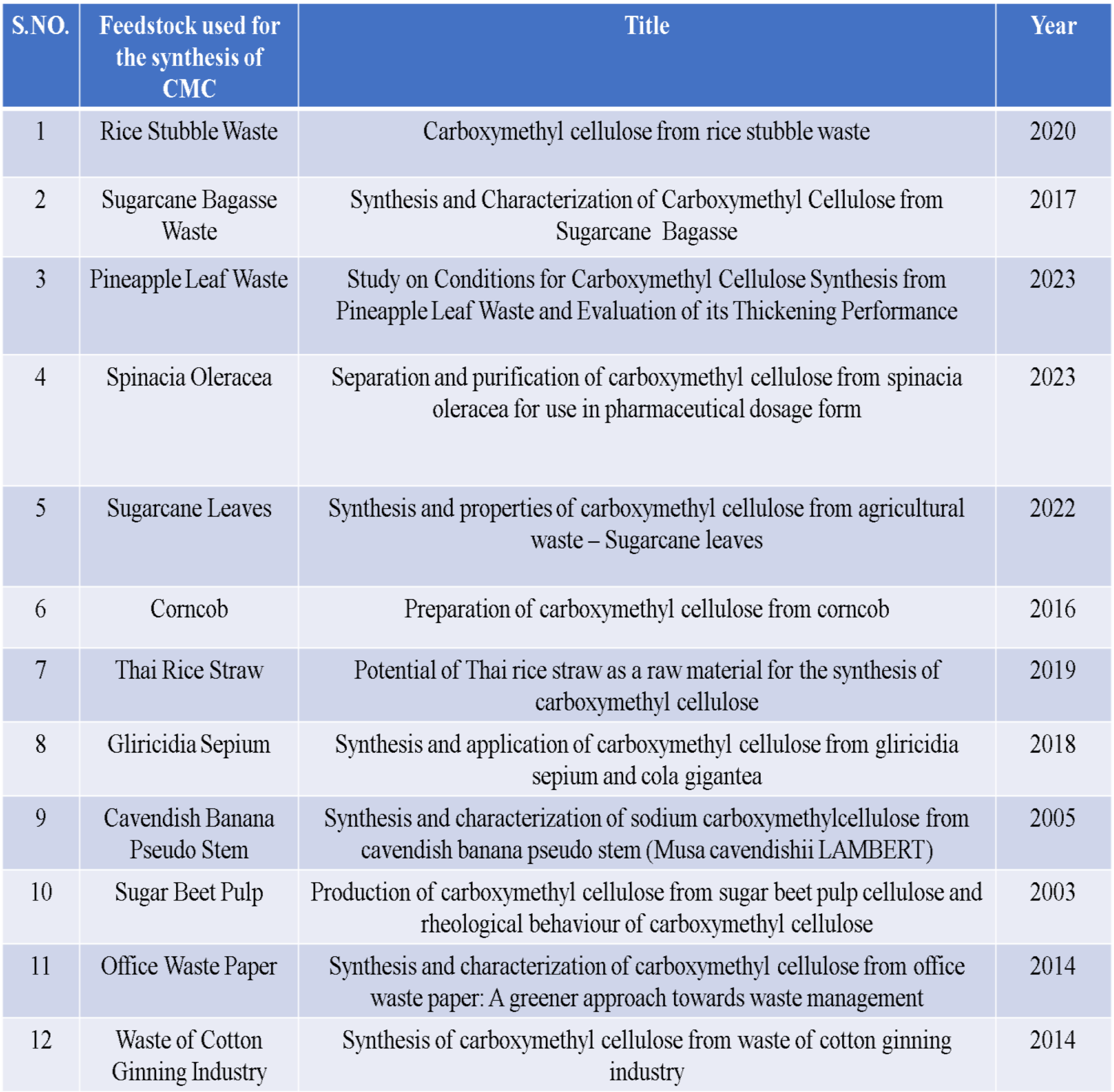
Here are some common waste materials used in research to convert cellulose to carboxymethyl cellulose (CMC):

Sago Waste: Waste generated from processing sago palm, which includes cellulose rich residues.
Pineapple Leaf Waste: Waste materials obtained from pineapple cultivation, particularly the leaves, which contain cellulose.

Rice Straw: Agricultural waste from rice farming, consisting of the stalks and leaves of the rice plant.
Sugar Beet Pulp Cellulose: Waste generated from processing sugar beets, containing cellulose-rich pulp.
Sugarcane Leaves: Waste from sugarcane cultivation, specifically the leaves that contain cellulose.
Corncob: Waste material from corn processing, specifically the cob part rich in cellulose.

Optimizing reaction conditions, such as temperature, pH, reaction time, and chemical concentrations, is crucial to achieving high yields and quality CMC products.

Table 1 - Studies of Conversion of Agricultural waste to CMC



S.NO.	Feedstock used for the synthesis of CMC	Title	Year
1	Rice Stubble Waste	Carboxymethyl cellulose from rice stubble waste	2020
2	Sugarcane Bagasse Waste	Synthesis and Characterization of Carboxymethyl Cellulose from Sugarcane Bagasse	2017
3	Pineapple Leaf Waste	Study on Conditions for Carboxymethyl Cellulose Synthesis from Pineapple Leaf Waste and Evaluation of its Thickening Performance	2023
4	Spinacia Oleracea	Separation and purification of carboxymethyl cellulose from spinacia oleracea for use in pharmaceutical dosage form	2023
5	Sugarcane Leaves	Synthesis and properties of carboxymethyl cellulose from agricultural waste – Sugarcane leaves	2022
6	Corncob	Preparation of carboxymethyl cellulose from corncob	2016
7	Thai Rice Straw	Potential of Thai rice straw as a raw material for the synthesis of carboxymethyl cellulose	2019
8	Gliricidia Sepium	Synthesis and application of carboxymethyl cellulose from gliricidia sepium and cola gigantea	2018
9	Cavendish Banana Pseudo Stem	Synthesis and characterization of sodium carboxymethylcellulose from cavendish banana pseudo stem (Musa cavendishii LAMBERT)	2005
10	Sugar Beet Pulp	Production of carboxymethyl cellulose from sugar beet pulp cellulose and rheological behaviour of carboxymethyl cellulose	2003
11	Office Waste Paper	Synthesis and characterization of carboxymethyl cellulose from office waste paper: A greener approach towards waste management	2014
12	Waste of Cotton Ginning Industry	Synthesis of carboxymethyl cellulose from waste of cotton ginning industry	2014

(HUANG, 2016)

3. Research Gap:

Limited work on waste feedstock for this process
Stand-alone studies without proper mapping between properties and application

4. Proposed Objectives:

Synthesis of CMC from waste paper and waste fabric
Characterization of CMC synthesized
Comparison of properties with Commercial CMC

5. Significance of Proposed Work:

Challenges in Waste Management

Waste management is a global challenge with significant environmental and economic implications. The improper disposal of waste materials leads to pollution, resource depletion, and land degradation.

Waste clothes and waste paper are two major components of municipal solid waste. While they are recyclable, a considerable amount still ends up in landfills, contributing to environmental harm.

Addressing waste management challenges requires innovative approaches such as recycling, repurposing, and sustainable resource utilization.

Potential of Waste Clothes as Raw Material

Waste clothes, including discarded textiles from households, industries, and fashion production, represent a substantial portion of municipal waste streams.
These textiles are rich in cellulose, especially those made from natural fibers such as cotton, linen, and hemp. Cellulose constitutes the structural framework of these fibers, making them potential raw materials for CMC production.

Converting waste clothes into CMC offers several advantages:

Reduces textile waste and landfill pressure.
Utilizes renewable resources effectively.
Creates value-added products with diverse applications.

Promotes sustainable practices in the fashion and textile industries.

Significance of Utilizing Waste Paper

Waste paper, generated from offices, households, packaging industries, and printing presses, is another significant component of municipal solid waste.

Despite being recyclable, a substantial amount of waste paper is discarded due to various factors such as contamination, lack of recycling infrastructure, and low market demand for recycled paper products.

Repurposing waste paper into CMC presents an opportunity to:

Reduce paper waste and conserve forest resources.

Enhance the circular economy by closing the loop on paper recycling.

Develop eco-friendly alternatives to conventional paper-based products.

Address environmental concerns related to deforestation and carbon emissions.

(Joshi, 2014)

Environmental and Economic Implications

- Utilizing waste clothes and waste paper for CMC synthesis offers significant environmental and economic benefits.
- Environmental benefits include waste reduction, resource conservation, energy savings, and reduced carbon footprint.
- Economic benefits include cost savings through waste valorization, development of new revenue streams, job creation in recycling and manufacturing sectors, and enhanced market competitiveness.
- CMC derived from waste materials aligns with sustainability goals, circular economy principles, and corporate social responsibility initiatives.
- Innovations and Challenges in CMC Synthesis from Waste Materials
- Innovations in CMC synthesis from waste materials focus on:
- Developing efficient and scalable extraction methods. Enhancing chemical modification processes for higher yields and improved product properties. Characterizing waste-derived CMC for quality control and performance assessment.

CHAPTER 2

EXPERIMENTAL DETAILS

1. Materials:

Waste clothes, Waste papers, Commercial Cellulose, Commercial Carboxy Methyl Cellulose (CMC), Isopropyl Alcohol IPA), (NaOH), Mono-Chloro-Aceticacid (MCA), Hydrogen Peroxide (H_2O_2), Acetic Acid (CH_3COOH),

2. Methodology:

The primary objective of this research is to synthesize CMC from waste clothes and waste paper, optimizing extraction, chemical modification, and characterization processes.

Conversion of Cellulose to CMC
(Heinze, 1999)
The methodology involves:
Collecting and sorting waste clothes and waste paper samples. Preparing cellulose-rich materials through shredding, grinding, and purification steps. Conducting alkalization and etherification reactions to produce CMC.

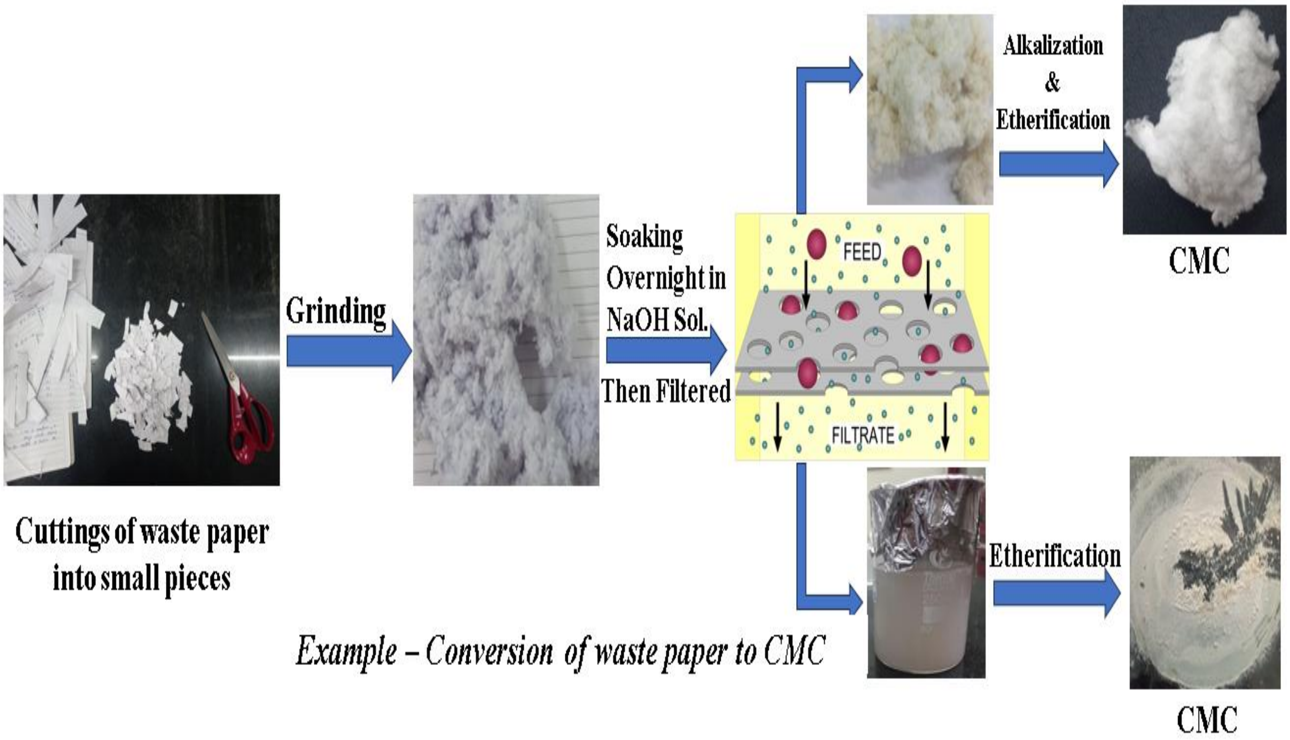


Fig3: Steps to synthesize CMC

Treating waste paper for the extraction of cellulose-

The paper waste used in this study was collected from labs – (lab files), Comprising primarily of handwritten paper, photocopier and computer printout papers, the paper waste underwent sorting to remove non-paper elements such as stickers, staples, and rubber bands. All chemicals utilized in the study were of analytical grade. The recycling of waste paper heavily relies on effective pulping and deinking processes. In this study, the waste paper was pulped and deinked, Deinking, a crucial step, involves the removal of ink particles from fibre surfaces and their separation from fibre suspensions. Flotation deinking, specifically, utilizes rising air bubbles in the pulp suspension to remove ink particles and hydrophobic contaminants. The waste paper was initially torn into squares pieces then grinded by using grinder and soaked in water overnight. After deinking, the pulp was recovered, washed, and drained, resulting in deinked MOW pulp ready for further processing.
(Joshi, 2014) (Haleem, 2014)

Preparation of Na-MCA by using MCA

Solution-1 - (MCA + Water)
Solution-2 - (Sodium Carbonate + Water)

Mix Solution-1 and Solution-2
And keep it on magnetic stirring at $80^{o}C$ for 2 hours

Synthesis of Carboxymethyl Cellulose (CMC) from Waste Paper

Paper waste and clothes waste extracted cellulose were prepared by drying at 80°C overnight to remove moisture. Alkalization was carried out by dissolving NaOH in deionized water (DI) before slowly adding it to Paper waste or clothes waste derived cellulose. The amounts of NaOH used varied. Isopropanol was added in different amounts to create a heterogeneous solution, which was stirred for 1 hour at room temperature.

Etherification Process:

Sodium mono chloroacetate (SMCA) was added to the solution, After the reaction, the solution separated into two layers. The upper layer was drained, and the lower layer was neutralized with glacial acetic acid.
The obtained product was washed multiple times with ethanol or methanol to remove impurities.

Carboxymethylation of Waste Paper Pulp:

The carboxymethylation of paper pulp proceeded through Williamson's ether synthesis reaction steps, with NaOH reacting first with cellulose hydroxyl groups to form alkoxide. Carboxymethyl groups were then formed in an SN2 reaction between cellulose alkoxide and SMCA. The conditions for carboxymethylation were optimized by varying process parameters such as NaOH concentration, reaction temperature, and reaction time.

3. Conversion to CMC:

Isolated cellulose from cotton gin waste was also converted to CMC through alkalization and etherification under heterogeneous conditions. The cellulose-NaOH activation reaction, known as mercerization, was performed at room temperature by adding varying concentrations of NaOH and stirring for 2 hours.
Carboxymethylation was initiated by adding varying amounts of SMCA and heating the reaction mixture at different temperatures for 3 hours. The resulting product was filtered, washed with methanol, neutralized with dilute glacial acetic acid, and dried at 60°C overnight to obtain CMC powder.
(Haleem, 2014) (Joshi, 2014)

4. Characterization of CMC:

Melting Point Analysis –

Measures the temperature at which CMC transitions from solid to liquid state, indicating its thermal stability and purity, important for various applications.

FTIR (Fourier Transform Infrared Spectroscopy) -

Used to analyse the chemical structure of CMC by measuring the absorption of infrared radiation, identifying functional groups like -OH, -CH_2, and carboxymethyl (-COO-) groups.

XRD (X-ray Diffraction) -

Determines the crystalline structure of CMC, measuring diffraction patterns when X-rays interact with the sample, providing information about its molecular arrangement and degree of crystallinity.

Degree of Substitution Determination -

Calculates the average number of hydroxyl groups in cellulose substituted by carboxymethyl groups, crucial for understanding CMC's chemical modification and properties.

(Rachtanapun, 2011)

CHAPTER 3
RESULTS AND DISCUSSION

Melting point:

Melting point of CMC is 274^{O}C, but commercial CMC degraded around 265^{O}C. Same observation we got for these samples, these also degraded around 265^{O}C.

FTIR- (Fourier Transform Infrared Spectroscopy):

It is a technique that measures the absorption of infrared light by a sample. This absorption occurs at specific frequencies for different functional groups and chemical bonds, allowing to identify the presence of specific functional groups in compounds. After doing FTIR analysis we got results which having similar characters of FTIR results which we got from literature**,** carboxymethyl - 1620 cm^{-1},1423 cm^{-1}, hydroxyl functional groups – 1328 cm^{-1}, carbon-hydrogen (C-H) -2920 cm^{-1}, COO^{-}- 1620 cm^{-1}, CH_2 scissoring - 1423 cm^{-1}, hydroxyl group - 3423 cm^{-1}, hydroxyl group bending - 1328 cm^{-1}, >CH-O-CH_2- 1054 cm^{-1} (Haleem, 2014)

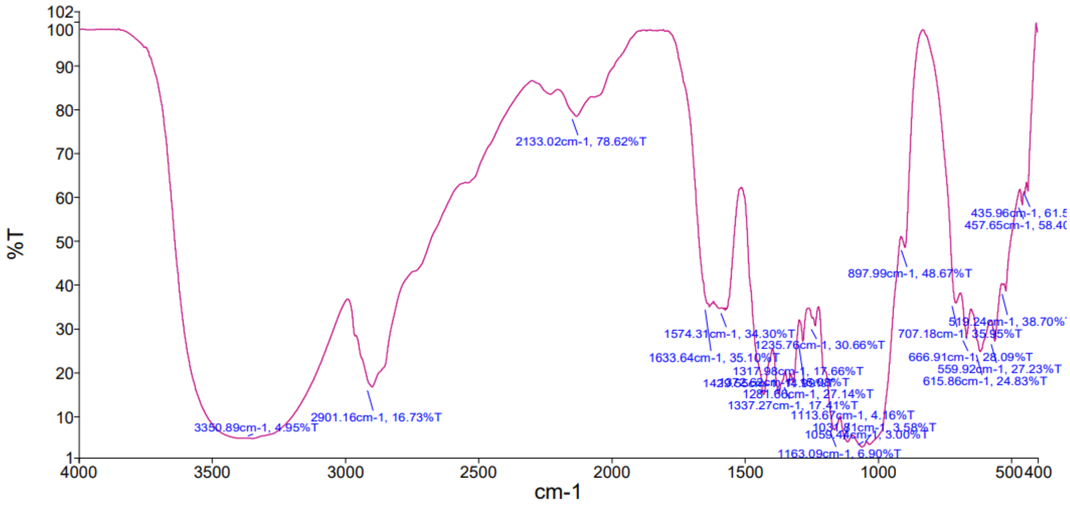


Fig4: *FTIR (WC- CMC) Waste Cotton to CMC*

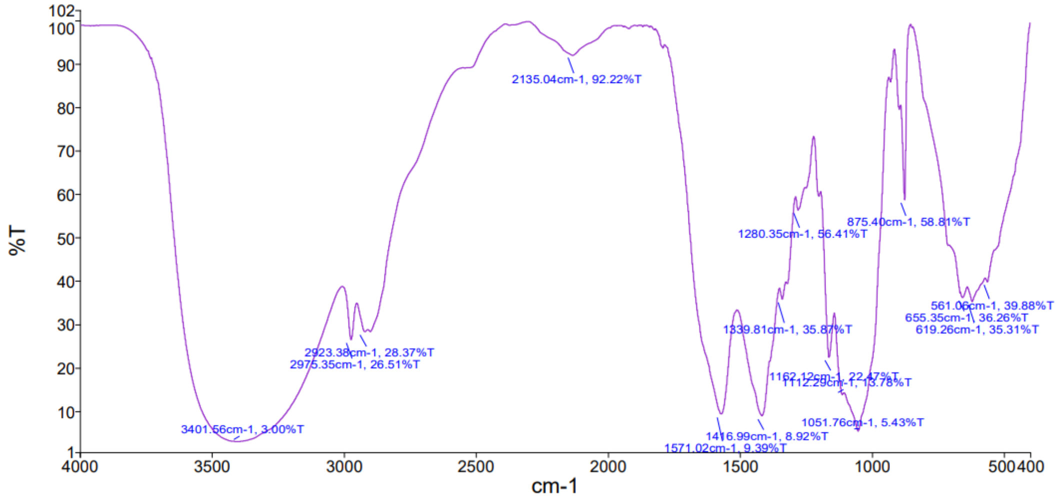


Fig5: FTIR (WP-CMC) Waste Paper to CMC

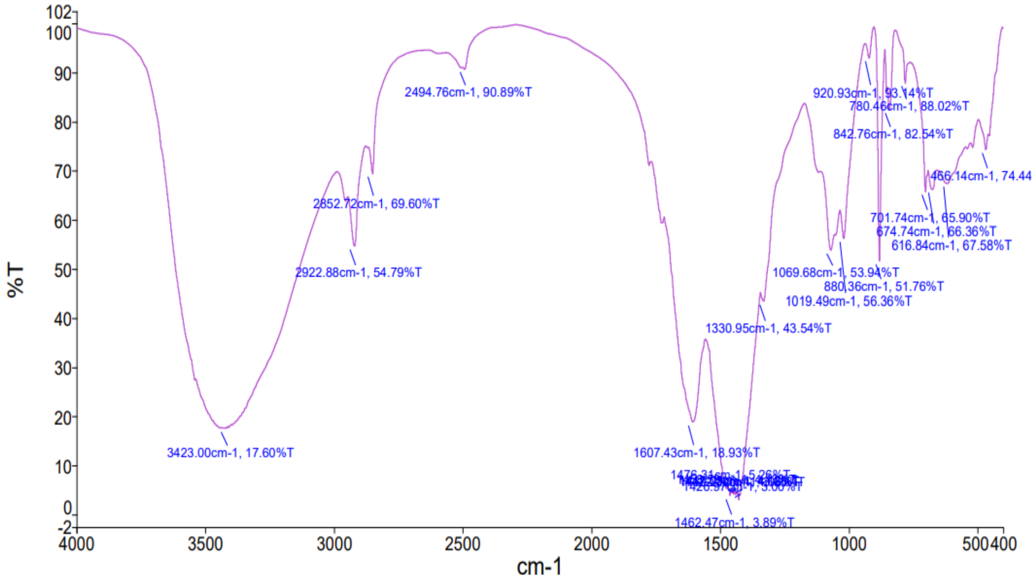


Fig6: FTIR (CP-CMC) Waste Cotton's pulp to CMC

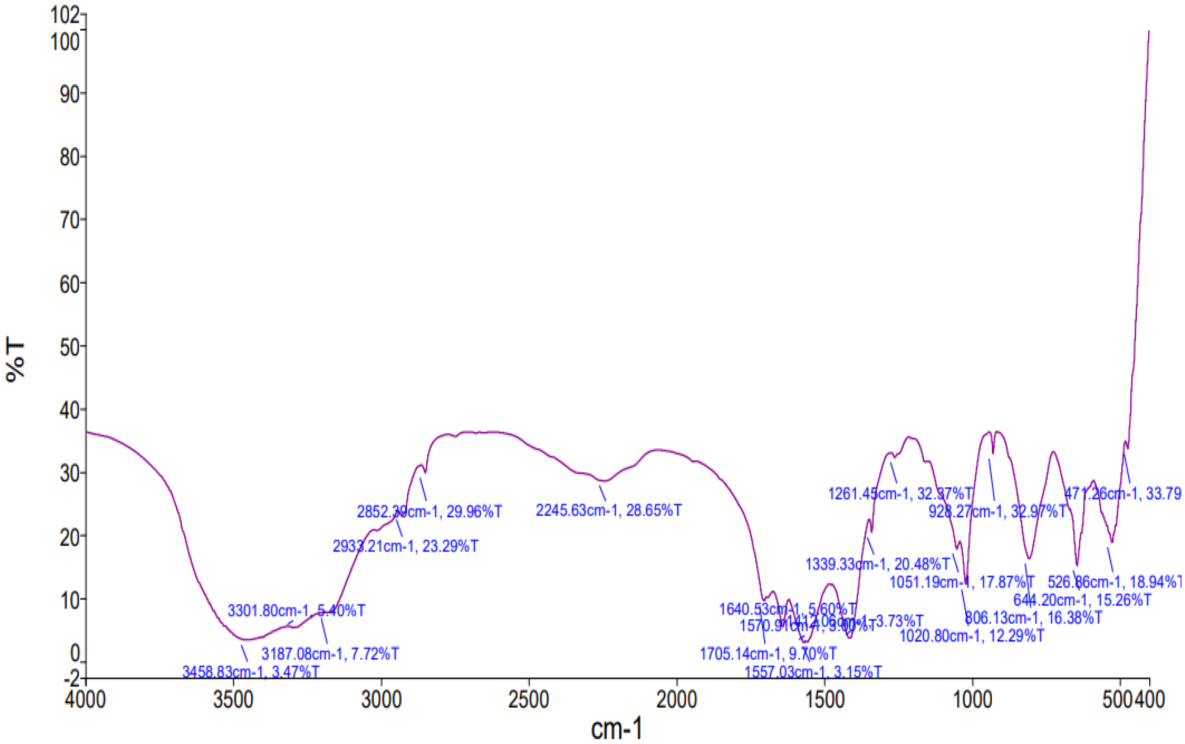


Fig7: *FTIR (PP-CMC) Waste Paper's pump to CMC*

XRD- (X-Ray Diffraction):

It is used to study the crystalline structure of materials. When X-rays are directed at a sample, they are diffracted in specific patterns depending on the arrangement of the atoms in the crystal lattice. By analyzing these diffraction patterns,

we can determine the crystal structure, phase structure, crystallographic orientation of materials using XRD.

After doing XRD analysis we got results which having similar characters of XRD results which we got from literature**,** major peak around 20.76 and sample's showing its crystalline nature

(Haleem, 2014)

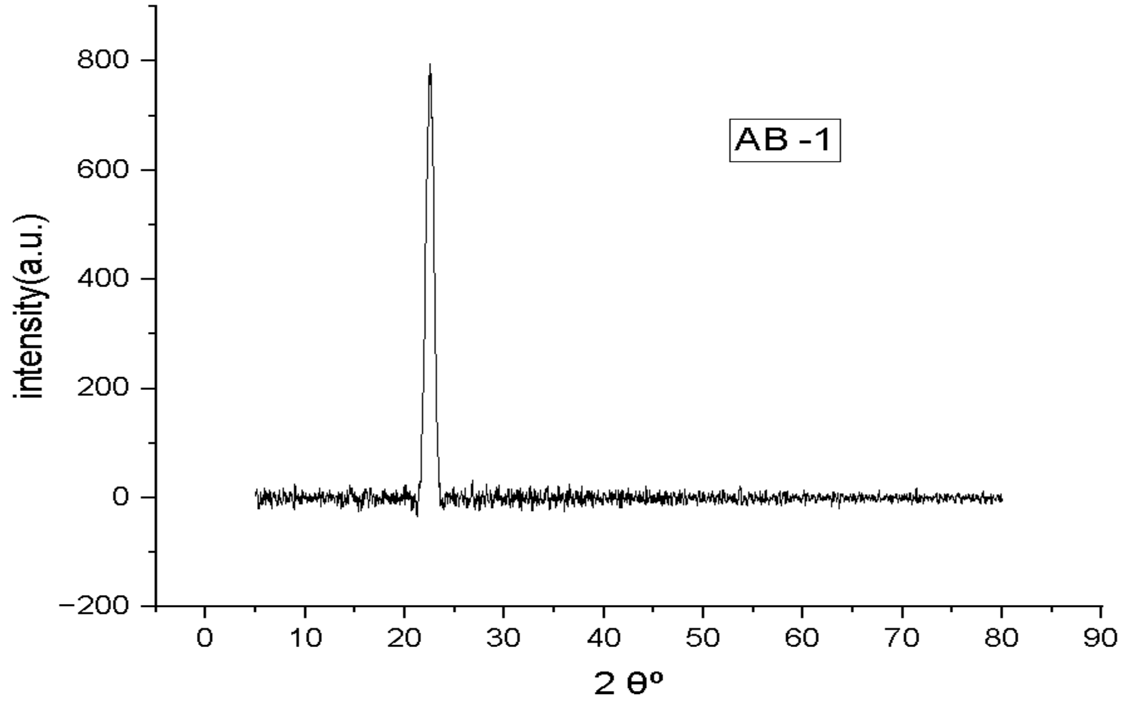


Fig8: *XRD AB-1 Waste derived cellulose hydrolyzed under acidic conditions to CMC (set -1)*

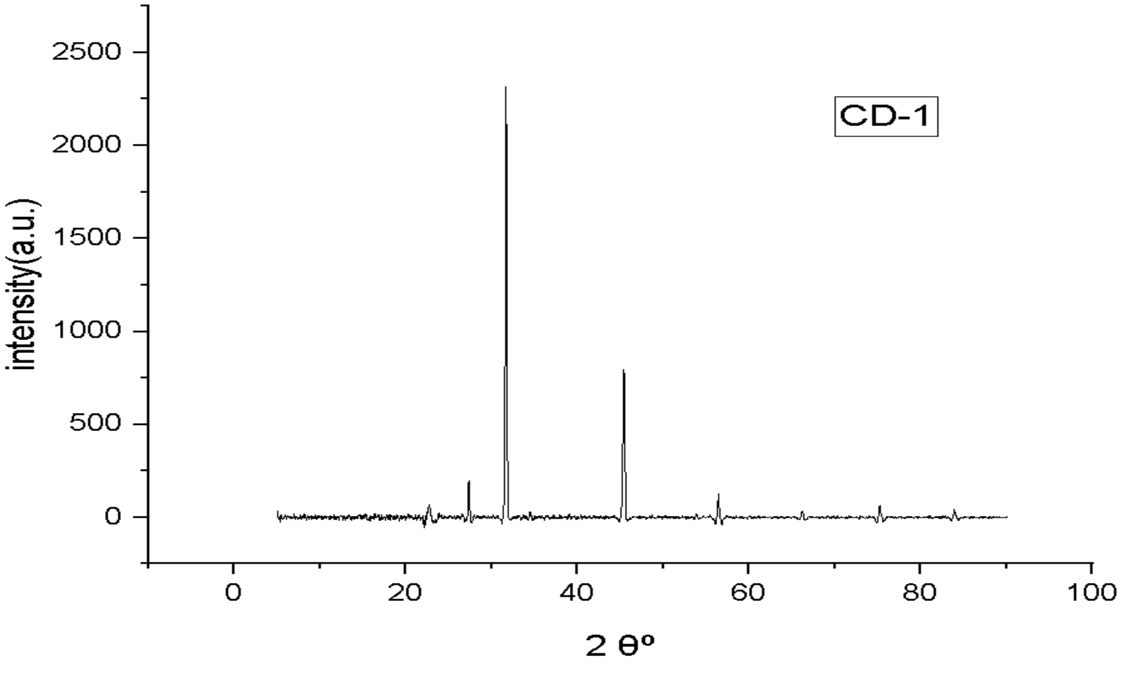


Fig9: *XRD CD-1 Waste derived cellulose hydrolyzed under acidic conditions to CMC (set -2)*

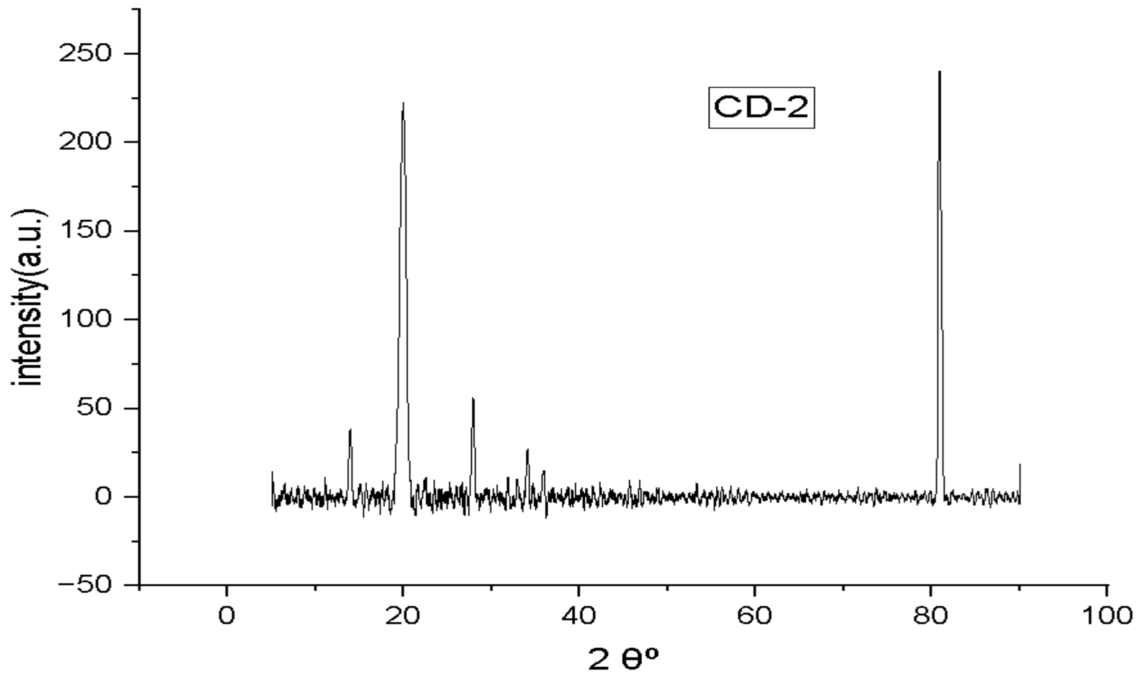


Fig10: *XRD CD-2 Waste derived cellulose hydrolyzed under acidic conditions to CMC (set -3)*

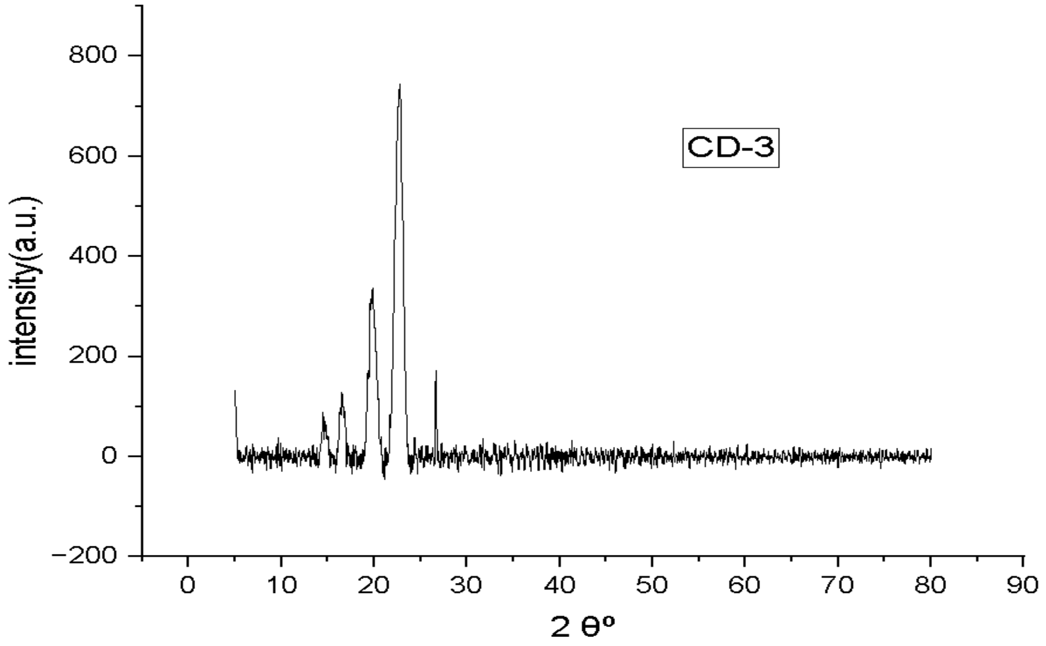


Fig11: *XRD CD-3 Commercial cellulose to CMC*

Degree of Substitution:

We calculated degree of substitution of 1 set of synthesized CMC and got 20% substitution in result, so then we worked with different reaction conditions and concentrations for getting better degree of substitution value.

Important Findings:

- CMC could be successfully extracted from all the starting materials.
- Characterization of all the sample was similar to commercial CMC and the references in the literature.
- The identified waste can serve as the potential feedstock for the generation CMC.

CHAPTER 4

Conclusion and Future Prospects

Conclusion:

The synthesis of carboxymethyl cellulose (CMC) from waste paper involved several crucial steps, including alkalization, etherification, and neutralization. These processes were optimized to achieve maximum efficiency and desired product characteristics. By repurposing waste paper and cellulose from fabric waste, valuable materials like CMC can be produced sustainably, contributing to waste reduction and resource utilization in eco-friendly ways
The extraction of CMC from all starting materials was successful, with characteristics matching those of the commercial sample and literature indicates that the identified waste materials having great potential as feedstock for CMC production.

Future Prospects:

- Optimization of process parameters for converting waste to CMC
- Defining the suitability of the synthesized CMC for the appropriate application
- Scale up of the process
- Exploration of more varieties of feed stock for this purpose

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