

Synthesis and characterization of quaternized cellulose-based flocculant from cardboard waste for dye treatment

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ABSTRACT

The rapid accumulation of cardboard packaging waste poses severe municipal solid waste management and environmental challenges. In this study, cellulose was successfully extracted from cardboard waste with a high yield of 48% and quaternized using 3-chloro-2-hydroxypropyltrimethyl ammonium chloride (CHPTAC) in a NaOH/urea solvent system at varying molar ratios (QC5, QC10, and QC15). The material characterization performed via Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), and X-ray Diffraction (XRD) verified successful cationic functionalization, structural disruption, and morphological modification. FTIR confirmed the presence of quaternary ammonium groups ($-\text{CH}_3$ bending at 1663.8 – 1616.6 cm^{-1} and 1475 cm^{-1}). XRD analysis showed that extracted cellulose possessed a high crystallinity index (78.57%), whereas quaternization reduced crystallinity to 44.07% (QC5) due to lattice opening. SEM imaging revealed highly porous web-like networks in QC15. To establish practical applicability in wastewater treatment, jar-test flocculation performance was systematically evaluated for Methyl Orange (MO) dye removal. QC5 achieved a maximum dye removal of 87.48% at an optimal dosage of 40 mg/L. Solution pH evaluation showed superior decolorization under acidic conditions, with QC10 attaining 68.66% removal at pH 4 via electrostatic charge neutralization. Under varying initial dye concentrations, QC15 achieved up to 80.35% removal at 20 mg/L MO. Comparative jar tests revealed that quaternized cellulose significantly surpassed commercial aluminum sulfate (alum), which achieved a maximum removal of only 44.66%. This work demonstrates the valorization of cardboard waste into effective flocculants for the sustainable treatment of dye wastewater.

Keywords: cardboard waste, quaternized cellulose, flocculant, dye treatment, methyl orange

1. INTRODUCTION

The rapid growth of industrialization, urbanization, and packaging activities has resulted in a substantial increase in municipal solid waste worldwide. Although cardboard is widely discarded or recycled through conventional routes, its high cellulose content makes it a promising renewable feedstock for producing value-added materials. Converting waste cardboard into functional cellulose-based materials can therefore provide a dual benefit by reducing waste while recovering a renewable resource [1], [2].

Cardboard waste contains a high amount of cellulose, making it a promising renewable resource for material development [1]. Cellulose is one of the most abundant natural biopolymers and possesses desirable properties, including biodegradability, renewability, biocompatibility, and high mechanical strength [3], [4]. These characteristics make cellulose highly attractive for the development of environmentally friendly functional materials [5]. In addition, the hydroxyl groups present along the cellulose backbone provide reactive sites for chemical modification,

allowing its properties to be tailored for specific applications. However, the direct utilization of cellulose is often limited by its strong intra- and intermolecular hydrogen bonding, which contributes to its limited solubility and functionality in aqueous applications. Chemical modification is therefore employed to introduce additional functional properties. One promising approach is quaternization, which introduces positively charged quaternary ammonium groups onto the cellulose backbone [6]. The resulting cationic cellulose can interact with negatively charged particles and contaminants, making it particularly attractive for coagulation–flocculation processes in wastewater treatment [7]. CHPTAC is a well-established reagent for the quaternization of cellulose, and its ability to introduce cationic functionality has been demonstrated in previous studies. Thus, the novelty of this work does not arise from the use of CHPTAC itself but from the selection of waste cardboard as the cellulose source and its conversion into a functional flocculant. Previous studies on quaternized cellulose have predominantly used commercial or purified cellulose and cellulose derived from other biomass sources. In comparison, the use of cellulose

recovered from waste cardboard as a precursor for CHPTAC-quaternized cellulose and its subsequent application in flocculation remains insufficiently explored. This approach provides a direct waste-to-value pathway in which cardboard waste is recovered as cellulose, chemically transformed into a cationic biopolymer, and subsequently utilized for wastewater treatment.

Material characterization plays a crucial role in understanding the structural, chemical, and morphological changes occurring during cellulose extraction and quaternization processes. Advanced analytical techniques such as Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), and X-ray Diffraction (XRD) are commonly employed to evaluate the success of cellulose modification and determine the resulting material properties [8-12].

Accordingly, this study aims to develop a quaternized cellulose flocculant from cellulose extracted from waste cardboard. The extracted cellulose and resulting quaternized cellulose were characterized using FTIR, SEM, and XRD to evaluate their chemical structure, morphology, and crystallinity. The flocculation performance of the synthesized material was subsequently investigated to assess its potential as a cellulose-based flocculant for dye treatment applications.

2. MATERIALS AND METHODS

2.1. Materials

Cardboard packaging waste was collected from nearby commercial areas. Sodium hydroxide (NaOH, ≥98%), sodium chlorite (NaClO₂, 80%), sulfuric acid (H₂SO₄, 95–97%), urea (CH₄N₂O), 3-chloro-2-hydroxypropyltrimethyl ammonium chloride (CHPTAC, 60 wt% in H₂O), Methyl Orange (MO) dye, and aluminum sulfate (Al₂(SO₄)₃·18H₂O) were purchased from Sigma-Aldrich.

2.2 Extraction of Cellulose from Cardboard Waste

Cardboard waste was shredded, immersed in tap water for 3 days (water changed twice daily), boiled for 30 min, and dried at 60 °C for 24 h [13]. Forty-five kilograms dried cardboard was ground into fine fibers using a mill blender. Alkali treatment was conducted using 4 wt% NaOH (sodium hydroxide) solution at 60 °C for 2 h (liquor ratio 5:200 g/mL) to dissolve hemicellulose and swell the fiber structure. Subsequently, bleaching was performed using 1.7 wt% NaClO₂ (sodium chlorite) buffered at pH 4.5 using H₂SO₄ (sulfuric acid) at 60 °C for 4 h to remove residual lignin. Both alkali and bleaching treatments were repeated three times until pure white cellulose was obtained. The final yield of extracted cellulose was calculated as:

$$\text{Yield (\%)} = (\text{Weight of Extracted Cellulose} / \text{Weight of Raw Cardboard Waste}) \times 100\% \quad (1)$$

2.3 Synthesis of Quaternized Cellulose (QC)

Extracted cellulose was dissolved in a NaOH/urea solution with a weight ratio of 7 NaOH: 12 urea at -20 °C for 24 h. Upon thawing, the solution was vigorously stirred to form a transparent cellulose solution and centrifuged at 2500 rpm for 5 min to remove insoluble residues. Quaternization was carried out by adding CHPTAC at 5:1, 10:1, and 15:1 molar ratios (CHPTAC : cellulose) and was designated as QC5, QC10, and QC15 [14]. The solution was reacted in an incubator shaker at 25 °C, 150 rpm for 24 h, neutralized to pH 7.0 using 0.5 M HCl (hydrochloric acid), soaked against distilled water for 3 days using a membrane dialysis tube, and freeze-dried for 24 h to yield solid QC product.

2.4 Material Characterization

2.4.1 FTIR

A potassium bromide (KBr) pellet was prepared using an agate mortar and pestle. 5–10 mg of sample and 90–95 mg of KBr were mixed, triturated, and combined for 5 minutes. The mixture was then transferred to the IR sample instrument and compressed for 60 seconds with an IR hydraulic press. The KBr pellet was inserted and scanned by using a blank range. After completing the blank spectrum, the KBr sample was removed from the window, thinly coated with the test sample, and placed back in the FTIR window to evaluate the sample spectrum.

2.4.2 XRD

Five grams of sample was positioned in the holder plate. Prior to analysis, it was placed in the XRD chamber at a scan range from 20° to 80°. The XRD spectrum was then analyzed using High Score Plus Software [15].

2.4.3 SEM

The conductive tape was affixed on the specimen stub and attached to the sample. Subsequently, it was placed into the specimen holder. The interval between the specimen surface used is 1 mm. After that, the specimen was placed on the specimen stage, and the image was observed and saved into CDR format.

2.5 Flocculation Performance Studies (Jar Testing)

Synthetic MO dye solutions (10–100 mg/L) were prepared in distilled water. Jar tests (Stuart Flocculator) were conducted with 400 mL dye solution per beaker. Rapid mixing was performed at 250 rpm for 3 min, followed by slow mixing at 50 rpm for 30 min and settling for 1 h. Each treatment was performed in triplicates to ensure data accuracy and reduce experimental variability. Supernatant samples were collected and analyzed using a UV-Vis spectrophotometer (Shimadzu UV-1800) at λ_{max} = 465 nm. Dye concentrations were quantified using a linear calibration equation ($y = 0.0425x + 0.1268$, $R^2 = 0.9978$), where y corresponds to the absorbance measured at 465 nm and x is the MO concentration in mg/L. Dye removal percentage was calculated as:

$$\text{Dye Removal (\%)} = [(Cr - Ct) / Cr] \times 100\% \quad (2)$$

where Cr and Ct are initial and treated dye concentrations, respectively. Parameters evaluated included flocculant dosage (10–100 mg/L), pH (2–12), initial dye concentration (10–100 mg/L), and comparison with commercial alum.

3. RESULTS AND DISCUSSION

3.1. Yield of Extracted Cellulose

Cellulose is a chemical compound produced largely from various plant sources. This study indicates that the yield of extracted cellulose from cardboard waste is $48.09 \pm 0.26\%$. Consequently, the findings align well with a previous study reported by [16]), which indicated that cardboard waste has at least 50% cellulose.

3.2. FTIR Analysis

FTIR is one of the most prevalent techniques for identifying the various functional groups present in a substance. FTIR spectroscopy provides information about molecular fragments and the presence or absence of certain functional groups and can provide a more in-depth understanding of fiber structure. Figure 1 describes the FTIR spectrum of raw material, cellulose, and QC.

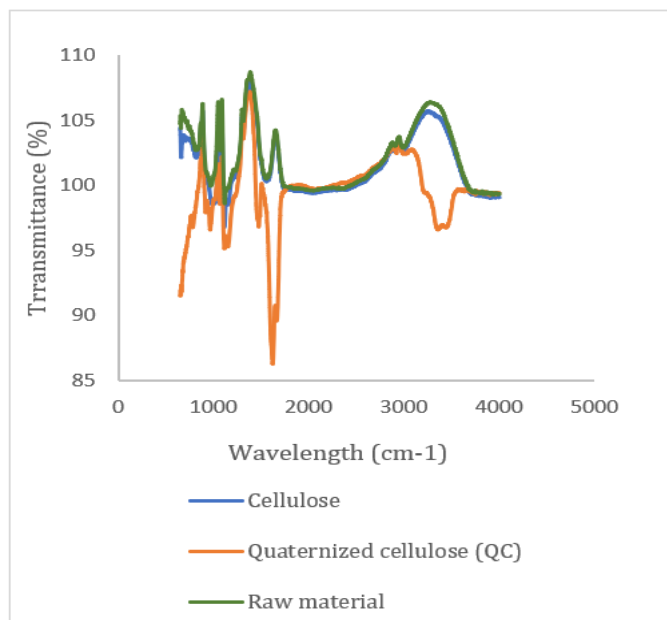


Figure 1. FTIR spectra of cellulose, QC, and raw material

The FTIR spectrum of QC exhibits strong absorption in the range of $3442.7\text{--}335.4\text{ cm}^{-1}$, indicating O-H in the hydroxyl group in Table 1. This signifies the existence of water. The results are similar to a previous study reported by [10], which identified a significant absorption band between $3650\text{ and }3250\text{ cm}^{-1}$, indicating the presence of a hydrogen bond in the material. This band clearly contains water, hydroxyl, and ammonium. The band at 2920 cm^{-1} was ascribed to the methyl (stretch) in the alkenes group. Upon

quaternization, new absorption peaks emerged at $1663.8\text{--}1616.6\text{ cm}^{-1}$ and 1475 cm^{-1} , corresponding to C-N bending and -CH_3 in the ammonium group. According to Md Noor et al. [11], an ammonium group is known to be non-toxic and biodegradable with a high positive charge. This peak was attributable to the bending stretching of the quaternary ammonium group due to the addition of 3-chloro-2-hydroxypropyl trimethylammonium chloride (CHPTAC).

This indicates that the quaternized cellulose possesses a positive charge. A previous study reported by Yawei et al. [17] shows that bending stretching of the quaternary ammonium group resulted in a peak near 1637 cm^{-1} .

Table 1. Summary of functional group for QC

Frequency (cm^{-1})	Bond	Functional Group
3442.7-3351.4	O-H (stretching)	Hydroxyl
2920	Methyl (Stretching)	Methyl
1663.8-1616.6	-CH_3	Ammonium
1475	-CH_3	Ammonium
1157	C-O (stretch)	Hydroxyl
1109.7-1071.9	C-O (stretch)	Hydroxyl
991.61-923.92	C-H (out of plane bend)	Aromatics
785.39	C-H (out of plane bend)	Aromatics

Furthermore, it was noted that quaternized cellulose exhibits wavenumbers ranging from $991.61\text{ to }923.92\text{ cm}^{-1}$ to 785.39 cm^{-1} , indicating the presence of C-H (out-of-plane bend) typical of aromatics. The wavenumber range of $1109.7\text{--}1071.9\text{ cm}^{-1}$ and 1157 cm^{-1} shows the existence of a hydroxyl group in C-O stretch. The ammonium group identified in this peak revealed that modified cellulose was the intended end product.

Table 2 illustrated that the FTIR spectrum of cellulose has the wavenumber range from $2989.3\text{ to }2901.2\text{ cm}^{-1}$, indicative of C-H stretching in methyl groups. Therefore, it indicates the presence of aliphatic compounds. The peaks seen at 1311.2 cm^{-1} and 662.6 cm^{-1} were attributed to the bending vibrations of the hydroxyl group, specifically the in-plane and out-of-plane bending modes.

Consequently, the study suggested that this cellulose has a high attraction for water. The band at 1556.8 cm^{-1} was ascribed to the C=C-C in the aromatic ring group. Cellulose also possesses a band in the range of $1163.2\text{--}1119.1\text{ cm}^{-1}$, which is assigned to the ether group. Additionally, the band at 986.89 cm^{-1} originates from the in-plane bending of C-H bonds inside the vinyl group. Furthermore, at 810.57 cm^{-1} ,

the C-O-O stretching vibration in the ether group is observed. This study confirms that cellulose has a functional group similar to the one described previously by [18], indicating that cellulose contains the highest concentration of hydroxyl and ether functional groups, as per the findings.

Table 3 illustrated the presence of over five peaks. Nandiyanto *et al.* [10] indicate that the examined chemical is not a simple compound. The FTIR spectra of cardboard display a wavenumber range of 2989.3 – 2901.2 cm^{-1} corresponding to C-H stretching in the methyl group. The double bond at 1556.8 cm^{-1} signifies the presence of an aromatic ring functional group.

Table 2. Summary of functional group for cellulose

Frequency (cm^{-1})	Bond	Functional Group
2989.3 – 2901.2	C-H(stretch)	Methyl
1556.8	C=C	Aromatic ring
1311.2	OH (in plane bend)	Hydroxy compound
1163.2- 1119.1	C-O stretch	Ether compound
986.89	C-H (in plane bend)	Vinyl
810.57	C-O-O stretch	Ether compound
662.6	OH (out of plane bend)	Hydroxy compound

3.3. XRD

A prior study by Fatimah *et al.* [9] indicated that sharp peaks signify strong crystallinity, while a less sharp peak signal a high amorphous structure. Table 4 shows cellulose has the highest percentage of crystallinity in comparison to raw material. Meanwhile, quaternized cellulose exhibits the lowest crystallinity in comparison to cellulose. Rasila *et al.* [8] indicate that alkali and bleaching treatments increase the cellulose content by removing a greater amount of amorphous polymer. The introduction of bulky quaternary ammonium side chains disrupts inter- and intramolecular hydrogen bonding, opening the crystalline lattice and converting cellulose into a partially amorphous, highly soluble polyelectrolyte. The crystalline structure of raw material, cellulose, and QC was examined by XRD spectroscopy as seen in Figures 2 and 3.

Therefore, these findings indicate that the cardboard possesses a relatively stable molecular structure. The band observed at 1119.1 cm^{-1} band can be attributed to the in-plane bending of C-H bonds in the aromatic group. The peaks at 1071.9 and 813.72 cm^{-1} are associated with the presence of ether groups. In addition, the band at 1311.2 cm^{-1} indicates the presence of a hydroxyl groups, which may be attributed to the cellulose and other oxygen-containing components present in the cardboard.

Table 3. Summary of functional group for raw material

Frequency (cm^{-1})	Bond	Functional Group
2989.3 – 2901.2	C-H(stretch)	Methyl
1547.3	C=C	Aromatic
1311.2	OH (in plane bend)	Hydroxy compound
1119.1	C-H (in plane bend)	Aromatic
1071.9	C-O stretch	Ether compound
974.3	C-H (out of plane bend)	Alkenes
813.72	C-O-O Stretch	Ether compound

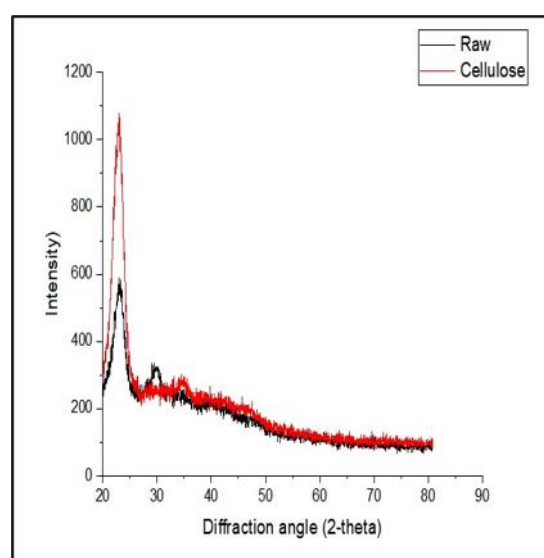


Figure 2. XRD pattern of raw material and cellulose

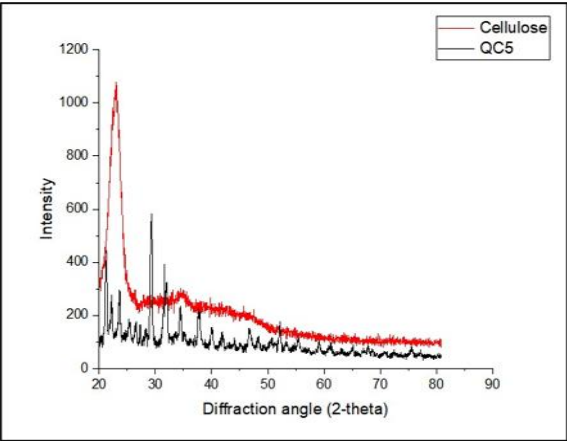


Figure 3. XRD pattern of cellulose and QC

3.4. SEM

Figures 4 (a) and (b) show the physical morphology of raw material and cellulose under magnification of 100 μm . Meanwhile, for all QC under magnification of (a) 1 mm and (b) 100 μm . The extracted cellulose fiber exhibited smoother surfaces and a white color compared to raw material (untreated). This is due to the elimination of lignin and other appealing components during treatment processes like bleaching and alkali treatment. The results are consistent with a previous study reported by [8], indicating smoother surfaces of cellulose. Figure 5 showed that the structure of all quaternized cellulose was irregularly formed. However, this observation shows that QC 15 has a more porous structure compared to QC 5 and QC 10, which is due to a hydrogen bond forming between the cellulose chain and cellulose crystal. As a result, the structure was disrupted

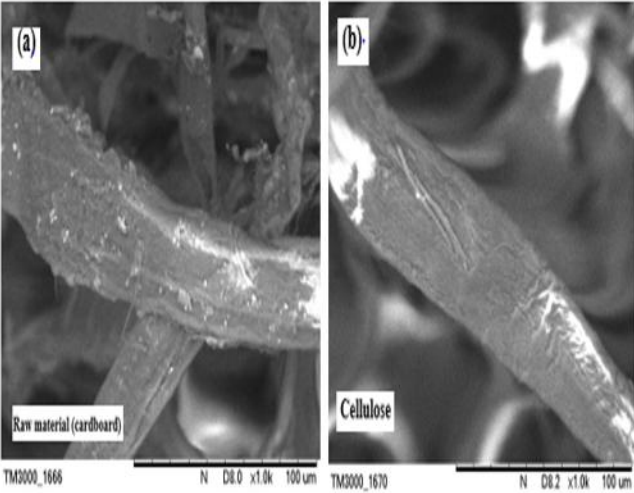


Figure 4. SEM images of raw material and cellulose samples, (a) before treatments, while (b) after treatments

Table 4. Percentage of crystallinity in raw material, cellulose and

Material	Percentage (%)
Cellulose	78.57%
Raw Material	67.24 %
QC5	44.07 %

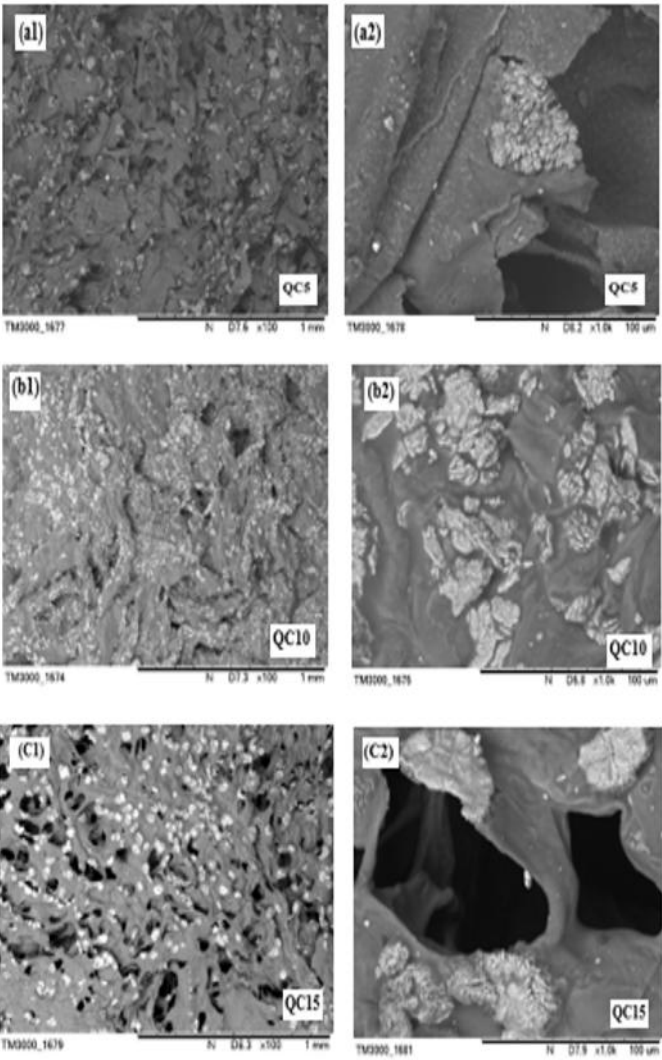


Figure 5. SEM images of QC at different magnifications and samples, (a1) and (a2) is QC5, (b1) and (b2) is QC10 while (c1) and (c2) is QC 15

3.5 PERFORMANCE OF QC AS FLOCCULANT IN DYE TREATMENT

3.5.1 Effect of QC flocculant dosage

A wide variety of flocculant dosages from 10 to 100 mg/L were investigated, as shown in Figure 6. In these studies, the variety of flocculant such as alum, QC5, QC10 and QC15 was analyzed by using jar test.

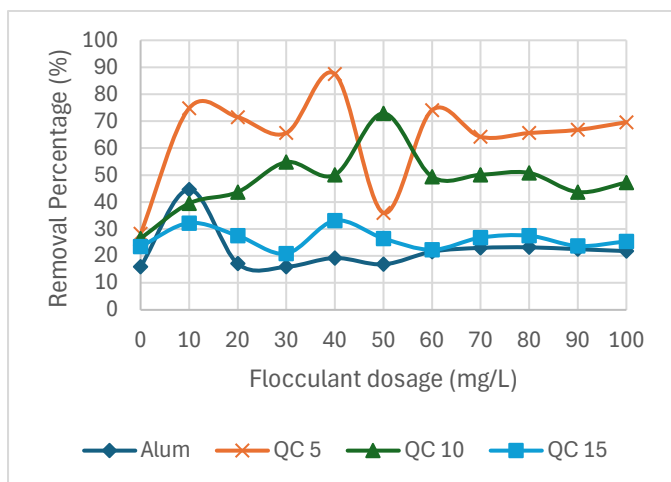


Figure 6. Effect of flocculant dosage on methyl orange dye removal efficiency

To effectively remove the methyl orange dye, QC is used as a flocculant. Meanwhile, alum is a commercialized flocculant that is used as a comparison jar test. The value of pH and 10 mg/L of methyl orange dye concentration was fixed. The percentage removal in QC10 was increased from 26.5% until 54.80% at 30 mg/L. However, when the dosage was increased to 40 mg/L, the reduction was roughly 50.10%. Then, at 50 mg/L, percentage removal in QC10 rises to 72.89 percent and then drops. QC5, QC15, and alum exhibited similar trends, with fluctuations in dye removal efficiency. The results also show that dye removal remained relatively stable at concentrations starting from 60 mg/L, possibly due to flocculant overdosing during the flocculation process. Among the tested flocculants, QC5 achieved the highest methyl orange dye removal efficiency, demonstrating its highest performance under the tested conditions. Md Noor et al. [11] attributed this improvement to the presence of sufficient cationic quaternary ammonium groups, which enhanced flocculation efficiency even at a lower flocculant dosage.

3.5.2 Effect of pH

Flocculation efficiency is also influenced by the pH. Even without the addition of a flocculant, pH influenced dye removal efficiency. As shown in Figure 7, QC5 and QC15 achieved their highest removal efficiencies at pH 2, reaching 55.01% and 51.95%, respectively. The removal efficiency of QC5 subsequently decreased as the pH increased towards 10, before rising again at pH 12. QC5

was discovered to have the highest removal percentages among other flocculants. The QC15 then continues to slowly drop and shows a fluctuating graph. The pH of the solution also had an effect on the performance of the blank, QC10, and alum. Thus, it also has a similar trend and shows the fluctuated graph.

Alum was discovered to have the lowest removal percentages among the other flocculants at pH 2 before rising to pH 4 and continuing to slowly drop until pH 12. The QC10 has shown the highest removal percentage of methyl orange dye among other flocculants, which is 68.66% at pH 4. Overall, the results suggest that acidic conditions favour color removal. A previous study reported by Banerjee & Chattopadhyaya [19] also shows that the lower the pH, the greater the number of positively charged sites available for the binding of negatively charged dye ions due to the electrostatic force of attraction. Consequently, a lower pH is preferable for dye removal in this circumstance.

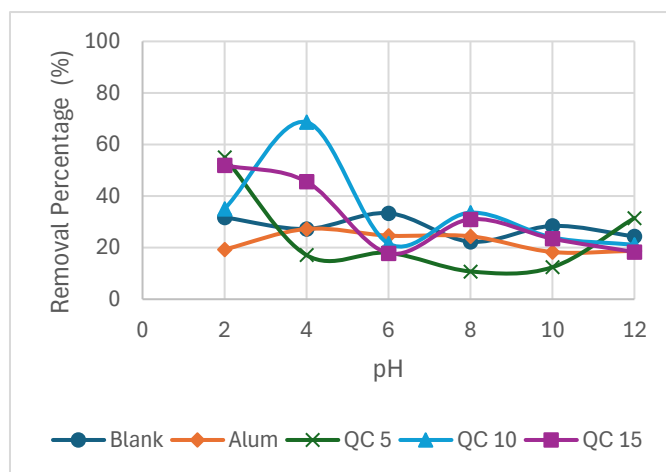


Figure 7. Effect of pH on methyl orange dye removal efficiency

3.5.3 Effect of Initial Dye Concentration

The effect of initial dye concentration on flocculation performance was also studied. A wide range of dye concentrations was used, ranging from 10 to 100 mg/L, as shown in Figure 8.

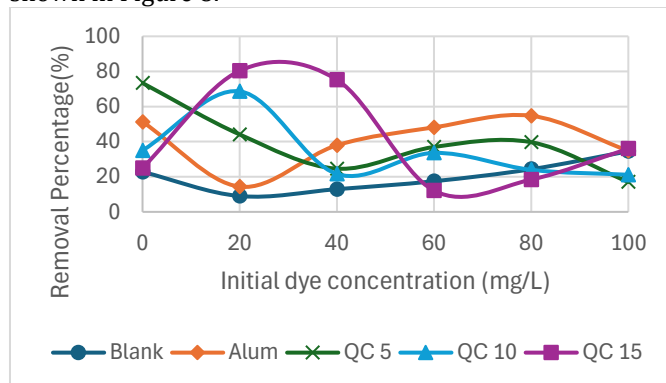


Figure 8. Effect of initial dye concentration on methyl orange dye removal efficiency

In these studies, the variety of flocculant such as alum, QC5, QC10 and QC15, was analyzed. Dye concentration influenced the flocculation performance of QC15. The removal percentage of QC15 and QC5 increases with dye concentration. The removal percentage of QC15 increased from 25.01% to 80.35% before slightly dropping to 35.97% at 100 mg/L. At 40 mg/L, this QC15 also shows the highest removal of dye compared to other flocculants. QC5 achieved the highest methyl orange dye removal at a concentration of 10 mg/L, with a removal efficiency of 73.40%. However, its removal efficiency gradually decreased at higher dye concentrations. The blank solution and QC10 exhibited lower removal efficiencies than QC15, QC5, and alum at 10 mg/L. The lower performance of QC10 may be attributed to its lower quaternary ammonium group content [11].

4. CONCLUSION

This study successfully demonstrated the extraction of cellulose from cardboard waste and the synthesis of quaternized cellulose through chemical modification using CHPTAC. The characterization analyses confirmed significant structural, chemical, and morphological transformations after cellulose extraction and quaternization. The jar test demonstrated the quaternized cellulose based flocculant had a better performance in terms of methyl orange dye removal as compared to commercial aluminum sulfate, mostly due to the electrostatic charge neutralization at the acidic pH. The results confirm that cardboard waste can be a sustainable and value-adding precursor for wastewater treatment under a circular economy concept.

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