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Modified cellulose-chitosan polyelectrolyte for enhancing wet and dry strength of paper

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Abstract

Carboxymethyl cellulose (CMC) and water-soluble chitosan-based polyelectrolyte systems were developed as wet and dry strength additives for paper. Chitosan was chemically modified through carboxymethylation to produce water-soluble carboxymethyl chitosan (CMCh). The FTIR peaks at 1586 cm^{-1} and 1400 cm^{-1} confirmed the successful modification. The water solubility of CMCh increased with temperature and was accompanied by higher conductivity. The water solubility and polyelectrolytic properties make CMCh suitable for papermaking applications. The use of CMCh, CMC, and their combinations improved mechanical properties, air resistance, and filler retention to varying degrees. At a 1% dosage, CMCh increased the wet tensile strength from 2.48 N·m/g to 3.10 N·m/g and enhanced filler retention from 3.6% to 6.89% . The combination of CMCh and CMC exhibited synergistic effects on dry-strength properties such as tensile and tear strength, but not on wet strength.

Keywords: Carboxymethyl cellulose; Water-soluble chitosan; Carboxymethyl chitosan; Dry strength agent; Wet strength agent

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Introduction

Paper is produced from a suspension of cellulose fibers, where interweaving and physical bonding result in a strong and flexible sheet after drying. The dry strength of a paper sheet determines its ability to withstand various external mechanical stresses (Lindström *et al.* 2005). To reduce production costs, fillers such as calcium carbonate, talc etc, are often added during the papermaking process, although their addition reduces the paper strength (Hubbe and Gill, 2016; Lourenço *et al.* 2013; Shen *et al.* 2010). In addition, other additives like internal sizing chemicals can also hinder the interfiber bonding (Chauhan and Bhardwaj, 2015; Shen *et al.* 2010). Increasing filler content typically decreases paper strength because inorganic fillers disrupt the bonding between fibers (Chauhan and Bhardwaj, 2015). Despite these drawbacks, fillers improve optical properties such as whiteness, opacity, brightness, and their lower cost compared to cellulosic fibers makes them economically attractive. To achieve the required dry strength of paper, the utilization of additives, fillers, and process optimization is controlled. Thus, the challenge lies in achieving high filler content without compromising paper strength.

As dry strength additives improve the strength of pulp, the water-soluble polymers, forming hydrogen bonds with cellulose fibers, are mostly used. These include both natural and synthetic polymers. Some examples of natural polymers used are starch (cationic/anionic starch) (Abdullah, 2010), nanocellulose and modified starch (Hubbe, 2019), carrageenan (Liu *et al.* 2017), carboxymethyl cellulose (Wang *et al.* 2015) and guar gum. Polyvinyl alcohol, polyacrylamide and its derivatives are some of the synthetic polymers which are used among others (Ashori *et al.* 2013; Ghasemian *et al.* 2012).

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Wet strength is another critical parameter that must be maintained alongside dry strength. Wet strength refers to the paper's ability to retain its resistance and character when exposed to moisture (Francolini *et al.* 2023). To enhance the wet strength of paper, various resins and polymeric materials, copolymers, were used previously. Some examples are phenol- formaldehyde resin, melamine-formaldehyde resin, polyacrylamide, urea, and vinyl acetate-vinyl chloride (Hubbe, 2014; Shen and Fatehi, 2013; Song *et al.* 2011). However, using non-renewable elements has increased health issues prompting the development of bio-based alternatives.

Chitin, a natural polysaccharide, is converted into chitosan, which is cationic in acidic solutions, making it suitable for electrostatic interactions with anionic pulp fibers. However, its solubility is limited to weak acidic media, restricting its application. To overcome this limitation, water-soluble chitosan derivatives have been developed. For instance, Bobu *et al.* (2011) introduced alkyl groups into the chitosan chain to create a hydrophobic yet cationic derivative. Nada *et al.* (2006) synthesized carboxymethyl chitosan to improve the strength properties of paper. Similarly, Fatehi *et al.* (2009) used carboxymethyl chitosan and carboxymethyl cellulose in a layer-by-layer assembly to enhance paper strength. Additionally, chitosan-starch nanoparticle complexes have been developed to enhance the physical characteristics of reused paper (Salam *et al.* 2018).

In this study, carboxymethyl chitosan was synthesized from shrimp shells and characterized based on FTIR spectroscopy, solubility, degree of substitution. CMCh, CMC, and a combination of CMCh and CMC were evaluated as dry and wet strength agents for paper. Additionally, their effects on the retention of filling material in paper were also investigated.

Materials and methods

Acacia wood pulp was collected from the Bashundhara Paper Mills Ltd., Bangladesh. Carboxymethyl cellulose of sodium salt was used as modified cellulose, whose viscosity ranges from 400 to 800cps. CMC was obtained from Research Lab Fine Chemicals, Mumbai, India. Carboxy-methyl chitosan was processed from shrimp shells. Hydrochloric acid, sodium hydroxide and sodium chloride used in our experiment were also obtained from Merck, Germany.

Preparation of chitosan

The chitin was extracted using the previously mentioned paper (Tolaimate *et al.* 2003). First, the shrimp shells were heated in distilled water at 85°C for 3 hours to remove dirt and surface impurities. Afterward, the shells were demineralized by using 1.90N HCl at a material-to-liquor ratio of 1:10 for 1 hour. Next, deproteinization was conducted using a 10% NaOH solution for 3 hours. This treatment yielded chitin, which was then subjected to deacetylation to obtain chitosan. Chitin was deacetylated using 60% NaOH at a material to liquid ratio of 1:15, followed by heating at 95°C for 18 hours. The resulting chitosan was slightly brown and was subsequently bleached with sodium chlorite to get a white product.

Preparation of carboxymethyl chitosan

Chitosan was dispersed in distilled water and allowed to swell for 10 minutes. Next, a fivefold volume of 40% NaOH solution was added to the swollen chitosan and kept at 25°C for 15 minutes with continuous stirring. Following this, isopropyl alcohol at a ratio of 1:20 and monochloroacetic acid at a ratio of 1:4 were added to the mixture. The reaction was conducted at various temperatures with continuous stirring for 3 hours. After the reaction was complete, the solid carboxymethyl chitosan (CMCh) was cleaned with pure IPA. Finally, the CMCh was dried at 40°C in the oven.

Degree of substitution of chitosan

Degree of substitution (DS) of chitosan was measured following Haqiqi *et al.* (2021) which was a titration procedure. 20 mL of absolute ethanol was added to 0.5 g CMCh and was stirred at room temperature for 5 mins. Then it was boiled with 5 mL of 16 M nitric acid for 10 minutes. The residual CMCh was then filtered, and thoroughly washed with hot ethanol. It was then dried and dissolved in distilled water to prepare a 1% solution, with the addition of 25 mL of 0.3 M NaOH. The solution was heated for 15 minutes and then brought back to room temperature. At the end, the solution was titrated with 0.3M hydrochloric acid using phenolphthalein as an indicator.

The degree of substitution was measured according to the following equations

$$A = \frac{(BC-DE)}{F} \qquad \text{------------------------}1$$

$$DS = \frac{0.162\times A}{(1-(0.058\times A))} \qquad \text{------------------------}2$$

Where, A = milliequivalent of consumed acid of 1g sample, B= Volume of sodium hydroxide, C= Concentration of sodium hydroxide, D= Volume of consumed hydrochloric acid, E= Concentration of hydrochloric acid, F= sample in grams.

Conductivity of polyelectrolyte

The conductivity of the CMCh, CMC and their mixtures solutions were measured using ET4510 LRC Benchtop LCR Tester Meter.

FTIR analysis

FTIR analysis was carried out with a PerkinElmer Frontier FTIR spectrometer (USA). The spectral data were processed using PerkinElmer Spectrum software (Version 10.4.4). Each sample was subjected to 32 spectral scans, with a 4 cm^{-1} interval and a spectral resolution of 16 cm^{-1}.

Application of CMCh and CMC in sheet making

Stock solutions of CMCh and CMC were made and a predetermined amount of CMCh and CMC, based on the oven-dried pulp, was added to pulp suspension. The pulp mixtures were then stirred continuously for 40 minutes for properly mixing the additives. Finally, paper sheets with a basis weight of 63 g/m² were made using a Rapid-Köthen sheet former.

Physical properties of paper-sheet

The physical strength of the prepared paper sheets was assessed following TAPPI test methods. Dry and wet tensile strength were estimated following T 494 om-96 and T 456 om-03, respectively. Burst strength was determined following T 403 om-97, and tear strength was estimated following T 414 om-98.

Table I. Carboxymethylation of chitosan

Temperature (°C)	Degree of substitution
26	1.31
35	2.05
65	2.49

Result and discussion

Carboxymethylation of chitosan at different temperatures

The carboxymethylation of chitosan was conducted at varying temperatures while keeping all other parameters constant. As illustrated in Table I, the degree of substitution (DS) increased with temperature. The DS was 1.30 at 25°C, rose to 2.49 at 65°C. The increase in DS at higher temperatures is likely attributable to the increased mobility of the reactants. In a study, Ge and Luo (2005) prepared CMCh using a microwave-assisted technique at a power of 260 W and 100°C for 20 minutes, followed by alkalization at 13.5 pH for 2 h. The product had a DS of 0.85 and exhibited good water solubility. Islam *et al.* (2023) reported DS values of 0.69 and 0.90 for CMCh. Song *et al.* (2011) observed that the DS of CMCh increased with longer reaction times and higher concentrations of chloroacetic acid. They obtained CMCh with 1.32 DS under reaction conditions of 90°C for 4 h and a mass ratio of 5.

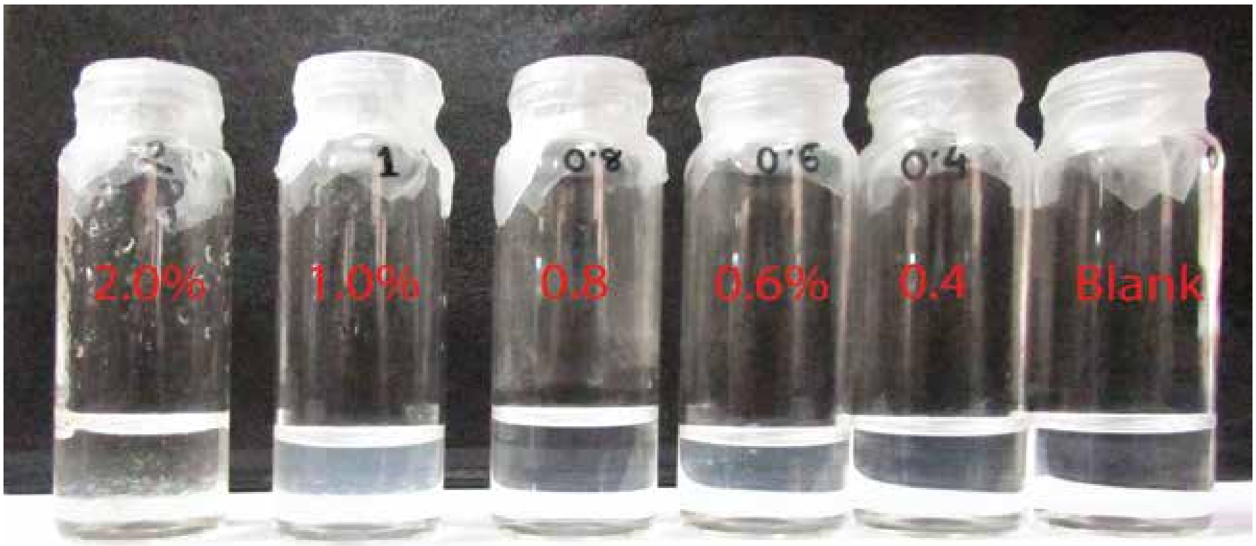

Fig. 1. Image showing solubility of Carboxymethyl chitosan at different CMCh (%) ratio at room temperature

Solubility of CMCh in water

To evaluate the solubility of CMCh, aqueous solution was prepared at varying concentrations (1-20 g/L). The mixtures were stirred for 2 hours at both room temperature and 50°C, followed by filtration through 0.45 µm filter paper. Solubility was determined based on the change in filter paper weight and expressed as the percentage of soluble CMCh. As shown in Fig. 1, CMCh formed a homogeneous solution in water. It was found that CMCh exhibited solubility in water up to 0.6% at room temperature, which increased to 2.0% in hot water (50°C). In a separate study, Islam *et al.* (2023) reported that CMCh was up to 0.3% soluble in water at 25°C. The solubility percentage is sufficient for the requirements of additives dosing in wet-end of the papermaking operations.

Table II. Conductivity of CMCh, CMC and CMCh+CMC solution

Solution, %	Conductivity, µs/cm
Distill water	4.20
0.05 CMCh	122.30
0.25 CMCh	594.00
0.50 CMCh	951.00
1.0 CMCh	2890.00
0.05 CMC	101.40
0.25 CMC	512.00
0.50 CMC	829.00
1.0 CMC	2700.00
0.05 CMCh + 0.05 CMC	133.50
0.25 CMCh+ 0.25 CMC	641.00
0.50 CMCh+0.50 CMC	915.00

Conductivity of CMCh as polymer electrolytes

Chitosan derivatives represent a relatively new field of research in polymer electrolytes. The conductivity of CMCh, CMC, and their mixtures in water was determined, and the results are presented in Table II. The conductivity had an increase with higher concentrations of CMCh or CMC. For example, the conductivity of distilled water was 4.20 µS/cm, which rose to 2890 µS/cm and 2700 µS/cm upon the addition of 1.0% CMCh or CMC, respectively. A mixture of 0.5% CMCh and 0.5% CMC in water reached a conductivity of 915 µS/cm. Mobarak *et al.* (2013) demonstrated that the conductivity of chitosan improves through carboxymethylation because of the increased presence of oxygen. Incorporating carboxymethyl groups into chitosan increases conductivity. Enabling its use in green polymer electrolytes. Furthermore, the conductivity of chitosan-based polymer electrolytes has been remarkably enhanced by reacting them with lithium and ammonium salts (Majid *et al.* 2005).

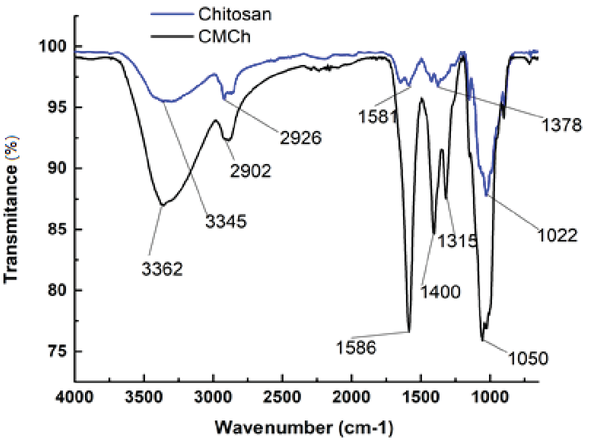

Fig. 2. FTIR spectra of chitosan and CMCh

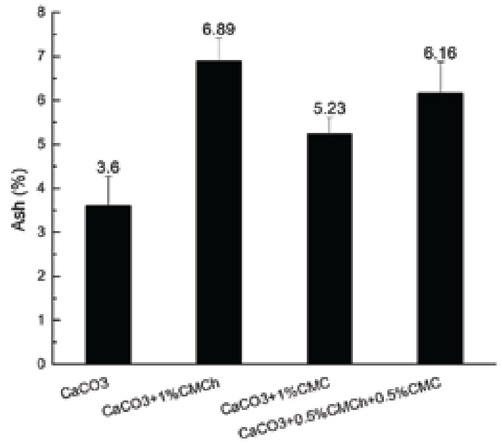

Fig. 3. Effect of CMCh and CMC on ash content

FTIR analysis

The FTIR spectra of chitosan and carboxymethyl chitosan are presented in Fig. 2. Principal characteristic peaks for both samples include 3310-3390 cm^{-1} (O-H stretching), 2910-2920 cm^{-1} (C-H stretching), 1590 cm^{-1} (N-H bending), 1310 cm^{-1} (C-N stretching), and 1020-1055 cm^{-1} (C-O stretching) (Farag and Mohamed, 2012). The spectrum of CMCh showed significant changes due to the substitution of carboxymethyl groups. The carboxymethylation at the C6 position of chitosan was indicated by the C-O absorption peak as it became more intense and shifted to 1050 cm^{-1}. Additionally, the intensity of the absorption peaks corresponding to the carboxyl group (overlapping with N-H bending and CH_2COOH) at 1585 cm^{-1} and 1410 cm^{-1} enhanced, confirming the successful substitution of carboxymethyl groups. The characteristic peak for the carboxyl group at 1735 cm^{-1} was overlapped in the spectrum. Compared to chitosan, the peaks of CMCh at 1585 cm^{-1} and 1410 cm^{-1} showed increased intensity, further confirming that carboxymethylation took place on both the amino and hydroxyl groups of chitosan. Similar spectral changes have been reported previously for carboxymethyl chitosan preparation (Ge and Luo, 2005; Mobarak *et al.* 2013).

Table III. Effect of CMCh and CMC on dry strength of paper

CMCh/CMC (%)	°SR	Tensile index (N·m/g)	Tear index mN·m^2 /g	Bursting index kPa·m^2 /g	Air resistance, sec/100ml
CMCh					
0	40	55.48±4.23	11.64±1.05	4.92±0.76	77±5
0.1	43	61.79±5.54	10.49±0.94	5.72±1.01	90±5
0.25	43	69.89±3.95	12.10±0.75	5.16±0.87	97±4
0.5	44	67.52±5.10	10.02±1.03	5.01±0.55	111±6
1.0	45	66.67±3.79	12.59±0.89	5.73±0.66	159±8
CMC					
0.1	47	60.68±5.19	12.89±1.53	5.21±0.84	92±4
0.25	48	63.04±2.86	12.59±2.04	5.66±1.00	96±4
0.5	49	65.12±3.35	12.49±1.08	5.72±0.39	99±6
1.0	52	67.97±5.05	12.05±1.93	5.07±0.51	103±6
CMCh + CMC					
0.05 + 0.05	45	65.65±4.15	11.38±0.97	5.07±0.19	92±5
0.1 + 0.1	48	71±3.80.71	12.46±1.15	5.65±0.76	110±7
0.5 + 0.5	49	77.29±5.26	14.59±2.13	5.46±1.01	103±4

Dry and wet strength analysis

The properties of paper sheets without the addition of CMCh or CMC are referred to as the blank. The pulp was beaten to 43 °SR and then 0.1-2.0% dosage of CMCh, CMC, or their combination was added. The results demonstrate the effectiveness of CMCh, CMC, and their combination (CMCh+CMC) as dry and wet strength agents as shown in Table III and IV, respectively. Significant improvements in tensile, tear, and bursting indices, as well as air resistance were observed, with the addition of these agents.

Tensile strength

The tensile strength, which measures the paper's resistance to breaking under tension, showed notable improvements with CMCh, CMC, and their combination. Chitosan improves the inter fiber bonding resulting in an increase in tensile index (Jahan, *et al.* 2009; Lertsutthiwong *et al.* 2002). Wennman *et al.* (2022) reported that both dry and wet strength of paper improved significantly using cationic starch and chitosan based polyelectrolyte. The tensile index increased from 55.48 N·m/g to 69.89 N·m/g using 0.25% CMCh, indicating the enhancement interfiber bonding. However, the tensile index reduced to 66.67 N·m/g at 1.0% CMCh indicated the decreasing trend at higher concentration, possibly due to the high electronegativity of the system, which hindered cellulose fiber bonding. Conversely, CMC exhibited a gradual improvement in tensile index and reaching 67.97 N·m/g at 1.0% CMC. Excellent result was observed in case of the combination use of CMCh and CMC. The tensile index was 77.29 N·m/g at a dosage of 0.5% CMCh + 0.5% CMC, demonstrating a clear synergistic effect between the two agents.

Tear strength

The tearing strength of paper refers to the resistance of a paper sheet that has already been torn. Both CMCh and CMC showed an increasing tendency for tearing strength by improving the fiber fiber bonding. It was observed that the tearing strength of paper increased when chitosan is added to the paper furnish (Nada *et al.* 2006). The tear index varied between 10.02 and 12.59 mN·m²/g at the experimented dosages of CMCh while the highest value (12.59 mN·m²/g) was obtained at 1.0% dosage. For CMC, the tear index remained relatively stable (12.05-12.89 mN·m²/g) across all dosages. It indicated that the effect of CMC on tear index was negligible. Interestingly, the combination of CMCh and

Table IV. Effect of CMCh and CMC on wet strength of paper

CMCh/CMC (%)	°SR	Tensile index (N·m/g)
CMCh		
0	40	2.48±0.57
0.1	43	3.18±0.79
0.25	43	2.97±0.35
0.5	44	3.27±0.55
1.0	45	4.10±0,49
CMC		
0.1	47	2.36±0.57
0.25	48	2.42±0.67
0.5	49	2.89±0.36
1.0	52	3.10±0.51
CMCh + CMC		
0.05 + 0.05	45	2.66±0.29
0.1 + 0.1	48	2.46±0.38
0.5 + 0.5	49	2.76±0.55

CMC produced a synergistic effect, particularly at higher dosages. The combined system consistently exhibited a higher tear index than either additive alone. The tear index reached a maximum of 14.59 mN·m^2/g at a combined addition of 0.5% of CMCh and + 0.5% of CMC. This enhancement can be attributed to the complementary functional properties of CMCh which may provide the cationic sites for electrostatic bonding and CMC improving hydrogen bonding among fiber network, thereby improving tearing resistance.

Burst strength

Bursting index measures the paper's resistance to rupture under pressure which showed moderate improvements with CMCh and CMC. Marcello and Salam (2023) observed that N-hydroxymethyl starch-amide increased the paper burst index by 38% at 1% addition. The bursting index increased from 4.92 kPa·m^2/g to a maximum of 5.73 kPa·m^2/g at 1.0% dosage of CMCh. For CMC, the bursting index remained consistently higher than the non-additive paper sheet across all dosages. The highest value of 5.72 kPa·m^2/g was observed at 0.5%, likely due to enhanced hydrogen bonding which strengthen the fiber network and improve resistance to multi-directional stress during bursting. In case of the combination of CMCh and CMC resulted in moderate improvements, but no strong synergistic effect was observed for bursting strength, with the highest value (5.65 kPa·m^2/g at 0.1% + 0.1%) being comparable to the performance of the individual additives.

Wet strength

The addition of CMCh, CMC, and their combinations influenced the wet tensile strength of the paper sheets to varying degrees, as demonstrated in Table IV. For CMCh, the wet tensile index increased steadily with dosage, from 2.48 N·m/g in the control to a maximum of 4.10 N·m/g at 1.0%. This improvement can be attributed to the strong interactions with the cellulose fibers under wet conditions. This trend suggests that CMCh is highly effective in improving wet tensile properties due to its ability to form water-resistant bonds. In contrast, the addition of CMC resulted in a more modest improvement, with tensile index values ranging from 2.36 N·m/g at 0.1% to 3.10 N·m/g at 1.0% The combination of CMCh and CMC did not produce a synergistic effect, as tensile index values for the mixtures (2.46-2.76 N·m/g) were lower than those obtained with CMCh alone at comparable total dosages. This suggests that competitive adsorption between the two polymers on fiber surfaces may limit their individual effectiveness. Overall, these results indicate that CMCh is more effective than CMC in enhancing wet tensile strength, and their combination offers no substantial advantage for this property.

Air resistance

Air resistance of paper is an important property that reflects its porosity. It needs to be controlled to meet specific end-use requirements. The addition of CMCh, CMC, and their combination increased the air resistance (Table III). The air resistance increased from 77 sec/100 mL to 159 sec/100 mL at 1.0% CMCh, indicating improved sheet formation and reduced porosity. At the same dosage of CMC, the air resistance increased to 103 sec/100 mL. Furthermore, the combination of CMCh and CMC moderately enhanced the air resistance to 110 sec/100 mL, suggesting that CMCh effectively reduces the porosity of paper

Filler retention

The retention of fillers, such as calcium carbonate ($CaCO_3$), is a critical aspect of papermaking, as it directly influences the paper's optical properties, smoothness, and cost efficiency. The filler retention can be improved by adding different types of polymers (Bhardwaj *et al.* 2023; Chauhan and Bhardwaj, 2015). Precipitated calcium carbonate (PCC) improvisation with chitosan increased filler retention, opacity and brightness (Chen *et al.* 2014). The results demonstrate the effectiveness of CMCh and CMC in improving filler retention, as indicated by the ash content in the paper sheets. The addition of 1% CMCh significantly increased the ash content from 3.6% (control) to 6.89%, indicating a substantial improvement in $CaCO_3$ retention. This improvement can be attributed to the adsorption of CMCh onto negatively charged fibers and filler particles. The electrostatic interactions between CMCh and $CaCO_3$ promote better retention within the paper matrix. The results highlight CMCh's effectiveness as a dry strength agent for improving filler retention, which is crucial for enhancing properties of paper such as opacity, brightness, and printability. The addition of 1% CMC also improved filler retention, increasing the ash content to 5.23%, although this improvement is less pronounced compared to CMCh. As an anionic polymerCMClikely improves retention by enhancing fiber-filler bonding and promoting better sheet formation. It may also act as a stabilizer, preventing filler particles from agglomerating and improving their distribution within the paper. The combination of 0.5% CMCh + 0.5% CMC resulted in an ash content of 6.16%, which is higher than the retention achieved with CMC alone and slightly lower than that with CMCh alone. This suggests a synergistic effect between the two agents. Where CMCh provides strong electrostatic interactions with filler particles, enhancing retention While CMC improves fiber-filler bonding and sheet formation, ensuring better distribution and retention of fillers. The improved filler retention with CMCh and CMC offers several benefits for papermaking, including enhanced paper opacity, brightness, and smoothness, making it suitable for high-quality printing and packaging applications.

Conclusion

The CMCh, prepared at varying temperatures, exhibits a high degree of substitution (DS). CMCh displayed good water solubility and significantly enhanced conductivity, confirming its potential as a polymer electrolyte. FTIR analysis verified successful carboxymethylation of chitosan. When applied in papermaking, CMCh proved to be an effective dry and wet strength additive, improving tensile, tear, burst indices, air resistance, and filler retention more prominently than CMC. CMCh was particularly superior in enhancing wet tensile strength and calcium carbonate retention, owing to its strong interactions with cellulose fibers and filler particles. CMC, while less effective in wet strength, contributed consistently to dry strength development and sheet formation. The combination of CMCh and CMC yielded synergistic benefits in certain properties (tensile and tear strength, filler retention), though not in wet tensile strength, where CMCh alone remained more effective.

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