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Total chlorine free bleaching of old corrugated containers pulp

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Abstract

Old corrugated container (OCC) pulp was upgraded to bleached pulp through a total chlorine-free (TCF) bleaching sequence consisting of formic acid (FA) delignification, followed by peroxyformic acid (PFA) treatment and two-stage alkaline hydrogen peroxide bleaching. In the FA delignification, the kappa number decreased from 51.5 to 42.9 after 2 h with minimal mass loss which shows selective lignin removal. The subsequent PFA treatment further reduced the kappa number to approximately 31 and increased the ISO brightness to about 24%. Two-stage alkaline peroxide bleaching resulted in a pulp brightness of 70–77% ISO and a yield of 49–52%. The bleached pulp exhibited a viscosity of 2.02–2.34 mPa·s, a holocellulose content of approximately 93%, and α-cellulose content of up to 84%. The pulp exhibited a tensile index of 38.1 N·m/g, a burst index of 1.09 kPa·m²/g, and a tear index of 2.03 mN·m²/g at a pulp freeness of 280 CSF. The FA/PFA-based TCF bleaching produced a moderate-strength, high-brightness pulp from OCC, which is suitable for paper and board applications.

Keywords: Pulp yield; Formic acid treatment; Delignification; Brightness; Viscosity

Introduction

Old corrugated containers (OCC), commonly referred to as corrugated cardboard boxes are the most available and widely used categories of recovered paper globally. OCC consist of primarily of unbleached long fiber pulps which are stronger compared to short fibers, making it a highly suitable material for the recycle paper and paperboard industry (Aguilar-Rivera, 2021). The OCCs are highly valuable in the recycling sector due to its abundant and the relatively low processing cost to new products (Anon, 2026).

The global demand of OCC is increasing rapidly compare to other paper products due to environmental awareness, stricter regulations on waste management, and the rising demand for sustainable packaging solutions. The market is forecasted to grow from USD 51.95 billion in 2026 to USD 79.90 billion by 2035, at a compound annual growth rate of 4.9% (Anon 2026). Asia Pacific region currently leads the market with the largest share, while North America is observed to be the fastest-growing region. The OCC consumption increased from 22.5 million tonnes in 2018 to a historical level of 22.8 million tonnes in 2020 in the United States. Moreover, US export a substantial amount of OCC. China imported a significant amount of OCC from US but the ban on importing solid waste enacted by China resulted in significant decline on US OCC export trade from 9.2 million tonnes in 2015

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to 7.6 million tonnes in 2020 (Zambrano *et al.* 2021). This shift created both challenges and opportunities, prompting the development of domestic upcycling strategies and the exploration of new export markets in South Asia.

Although the statistical data on OCC availability in Bangladesh are limited, the country's industrial development and regional perspective provide valuable information. Bangladesh is the largest producer of ready-made garments (RMG) and substantial development of manufacturing sectors generates a significant quantity of corrugated packaging waste, creating a locally available feedstock for pulp and paper production. OCC is an excellent source of recycled fiber but it has several challenges for the production of high-quality paper. The material is complex and heterogeneous, comprising fibers from different pulping processes: semi-chemical pulping with high lignin content and kraft pulping with lower lignin content (Zambrano *et al.* 2021). Unbleached OCC typically exhibits a kappa number of 85–105 and low brightness (approximately 18.9% ISO) (Jackson *et al.* 1994).

OCC contains a high amount lignin, which results in stiffer fiber and poor bonding ability that reduces the paper strength properties (Zambrano *et al.* 2021). Additionally, the fibers undergo hornification and degradation during repeated recycling, leads to decrease the physical properties with each reprocessing cycle (Hossen *et al.* 2024, Rahman *et al.* 2024). Furthermore, OCC contains many contaminants including adhesives, wax coatings, inks, and other pollutants, further complicate the recycling process (Wang *et al.* 2023).

To produce high-quality bleached pulp suitable for printing, writing, and tissue papers from OCC pulp, various bleaching technologies have been studied. OCC pulp bleached to ISO brightness levels of 75–80% through oxygen-alkaline treatment followed by peroxide and ozone bleaching sequences (Jackson *et al.* 1994a). Shafiei and Latibari (2015) investigated alkaline-sulfite anthraquinone pulping of OCC to produce pulp containing a lower amount of lignin without significant mass loss. The pulps were bleached using an elemental chlorine-free (ECF) bleaching sequence. The pulp produced under the conditions of 18% active alkali, 0.1% AQ and 150 min cooking followed by bleaching of D_0EPD_1 sequences, produced a pulp of 82.6% brightness. In an another study, OCC pulp bleached to 70% IS0 brightness at high temperature by hydrogen peroxide delignification and bleaching (Nguyen and Simard, 1996). The oxygen delignification of OCC pulp was studied by Danielewicz and Surma-Slusarska (2011), and found that oxygen delignification and bleaching was effective for OCC pulp.

A recent studies on bleaching of OCC pulp in an environmentally friendly elemental chlorine-free bleaching sequences have significantly reduced the kappa number (from about 89 to near zero) and increased the brightness to approximately 75% ISO. The resultant pulp showed improved tensile strength and absorbency, although some loss in bulk and softness occured (Zambrano *et al.* 2021). Reynolds *et al.* (2025) upgraded the OCC pulp to dissolving pulp by a fiber fractionation and removal of fines followed by delignification and bleaching. In addition, optimization of process parameters and bleaching chemicals in recycled pulp systems continues to focus on balancing brightness development with fiber strength preservation and chemical consumption (Moyo, 2026). A combination of formic acid delignification with alkaline extraction followed by multi-stage bleaching ($D_0EPD_1EPD_2$) could achieve high brightness (~85%) and high α-cellulose content (~95%), indicating the feasibility of producing high-value pulp from OCC (Jahan *et al.* 2016). Overall, current research trends focus on integrating efficient pretreatment, chlorine-free bleaching chemistry, and process optimization to enhance sustainability, reduce chemical load, and improve the end-use properties of recycled OCC fibers.

The objectives of this study are to delignification and bleaching of OCC using formic acid and peroxyformic acid treatments, followed by two-stage alkaline peroxide bleaching. This approach aims to produce high-brightness pulp from OCC through a TCF process.

Materials and methods

Materials

The OCC was collected from the local market and originated from both domestic and international manufacturers. The collected OCC was torn into small pieces (approximately 3–5 cm) by hand and soaked in water for 24 h. It was then disintegrated for 15 min in a disintegrator and subsequently screened through a flat

vibrating screener. The pulp was air-dried for further experiments.

The pulp was characterized for kappa number and α-cellulose in accordance with TAPPI standard methods (T 236 cm-85 and T 203 om-88), respectively. Holocellulose was determined by treating the pulp with $NaClO_2$ solution at pH 4 until it became white. The holocellulose content was then measured gravimetrically.

Formic acid treatment

The OCC pulp was refluxed with formic acid (90% v/v) for 1, 2, 3, and 4 h. The treatment was carried out using a 50 g oven-dry (o.d.) sample in a 1 L flat-bottom boiling flask equipped with a condenser. After treatment, the pulp was washed with 80% (v/v) formic acid, followed by hot water, and finally tap water until neutral pH (pH 7) was reached. The pulp yield and kappa number were determined gravimetrically and in accordance with TAPPI Test Method (T 236 om-99), respectively.

Peroxyformic acid treatment

After FA treatment, the pulp was further delignified with peroxyformic acid (PFA) at 80°C in a water bath. The PFA composition was varied with 1–4% H_2O_2 (on o.d. FA pulp) in 90% formic acid. Delignification was carried out for 1, 2, 3, and 4 h. After delignification, the pulp was first washed with formic acid and then with distilled water. The pulp yield and kappa number were determined gravimetrically and according to the TAPPI test method (T 236 om-99), respectively.

Bleaching

Bleaching of unbleached OCC pulp was performed using 30 g pulp at 10% consistency. Hydrogen peroxide was applied at 1–4% (on o.d. pulp) at each stage, while maintaining the pH at 11. The bleaching was performed at 80 °C for 1 h, and a similar procedure was followed in the 2nd stage of peroxide bleaching.

Evaluation of formic acid pulp

Bleached pulp was refined PFI and hand sheets were prepared for tensile (T 494 om-96), tear (T 414 om-98) and burst (T 403 om-97) tests. All characteristics were determined according to Tappi Standard Methods given in the parenthesis.

Results and discussion

The starting kappa number of OCC pulp was 51.5. The OCC pulp also had 81.3% holocellulose, 68.2% α-cellulose and 10.3% ash content (data are not shown). As shown in Fig. 1, OCC pulp was a mixture of chemical pulp of mechanical pulp, With Graff “C” stain, chemical pulp fibers show a vivid blue color, whereas mechanical pulp fibers exhibit a yellow-brown color.

Formic acid treatment of OCC

The delignification efficiency of formic acid (FA) for OCC was evaluated by monitoring the changes in pulp yield, kappa number, and brightness over a different reaction time, and results are presented in Table I.

As shown in Table I, the FA treatment significantly reduced the kappa number. The initial OCC pulp, characterized by a starting kappa number of approximately 51.5, underwent rapid delignification within the first hour, with the kappa number dropping to 44.5. This initial phase represents a reduction of about 13.6%, suggesting a high accessibility and reactivity of the residual lignin in the OCC fibers to the FA. FA cleave the aryl-ether bonds in lignin macromolecules and solubilize it which leads to the reduction of kappa number of pulp (Caridad *et al.* 2004, Dapia *et al.* 2002, Jahan *et al.* 2014). The delignification increased with the reaction time but rate decline. The kappa number of the OCC pulp reduced to 41.5 corresponding to a total reduction of 19.4% from the initial value after 3 h treatment. However, extending the treatment 1 h more to 4 h, the kappa number decreased only 0.7 points reaching 40.8. This indicates that lignin removed in two phases: an initial and rapid phase where bulk delignification occurred followed by a slow delignification happened where lignins strongly associated with carbohydrates are removed (Pande and Roy, 1996).

The pulp yield decreased gradually from 93.5% after 1 h to 90.4% after 4 h, with the kappa number. The decrease in yield is not exclusively due to lignin removal. FA catalyzed the hydrolysis of cellulose and hemicellulose, resulting in carbohydrate degradation (Vázquez *et al.* 1995). The relatively small decrease in yield (3.1%) for a 0.7point reduction in kappa number suggests a significant degradation of carbohydrate, especially in the second phase of delignification of the process. The brightness of the pulp showed minimal variation throughout the treatment, fluctuating between 16.5 and 17.2%.

Table I. Formic acid delignification of OCC

Time (h)	Yield (%)	Kappa number	Brightness
1	93.5	44.5	16.6
2	92.1	42.9	16.5
3	91.2	41.5	17.2
4	90.4	40.8	16.8

Fig. 1. Micro-photograph of OCC pulp

Based on the data of the kappa number against yield loss, an optimal treatment time for process efficiency appears to be around 2 h.

Peroxyformic acid treatment of FA treated pulp

The FA-treated OCC pulp, obtained after 2 h FA delignification, was afterward subjected to PFA treatment to evaluate its effectiveness in additional delignification. PFA was produced by the reaction of FA and hydrogen peroxide which generate highly reactive peracids and hydroxyl radicals that are strong oxidizing agents for lignin (More *et al.* 2021). The results of the PFA treatment over a period of 1 to 4 h are presented in Table II.

The PFA effectively delignified the FA treated pulp. The kappa number reduced to from 42.9 to 34.9, representing a reduction of 18.6% within the first 1 h treatment. The strong oxidative action of PFA significantly removed the residual lignin that remain after FA treatment. Peracids that produced in the reaction system are known to selectively cleave the phenolic and non-phenolic aromatic rings of lignin, leading to their fragmentation and solubilization (More *et al.* 2021; Sun *et al.* 2000; Zare-Zardini and Ghanipour-Meybodi, 2025). When the PFA treatment extended to 3 h, the kappa number further reduced to 31.1, achieving a total reduction of 27.5% from the starting value, and the kappa number decreased to 30.4 after 4 h. This behavior suggests that the most accessible residual lignin was removed during the

Table II. Peroxy formic acid treatment of FA OCC pulp

Time (h)	Yield (%)	Kappa number	Brightness
1	88.0	34.9	23.8
2	87.5	33.3	23.9
3	85.6	31.1	24.5
4	83.4	30.4	24.6

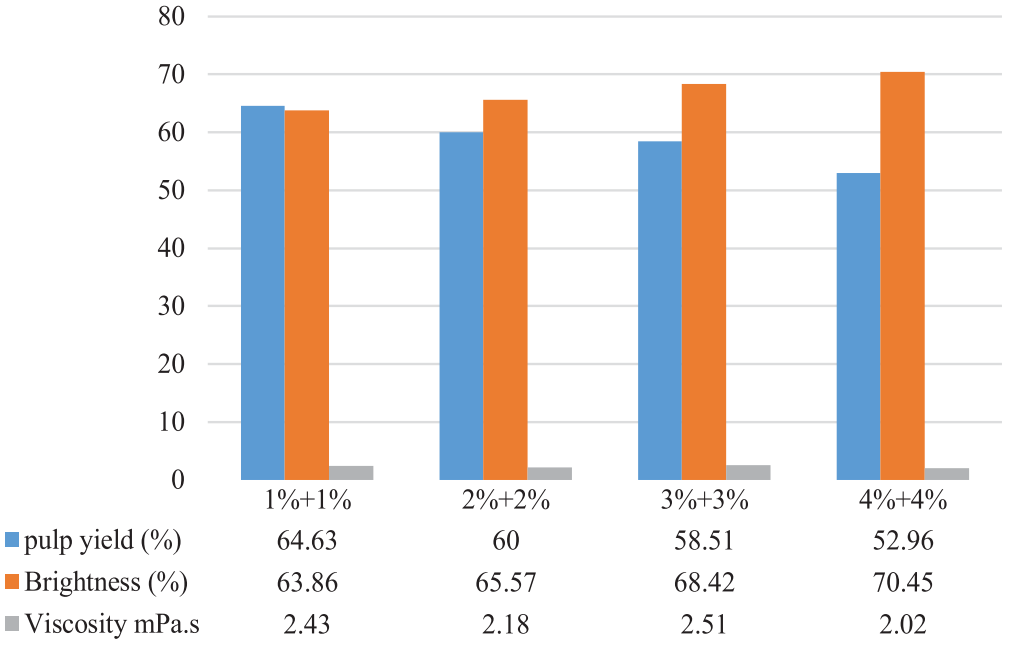

Fig. 2. Two stage alkaline peroxide bleaching of FA/PFA pulp (pH 11, Temp =80°C Time=1 h)

initial 3 h, while further treatment resulted in slow down the removal rate due to the limited accessibility of the remaining lignin or increased its resistance due to condensation reactions (Zhao and Liu, 2013). The PFA treatment achieved an additional reduction of 9.6 kappa points at 1 h PFA treatment, further reduction of 4.5 kappa points at 4 h treatment (Table II). The PFA is more selective delignifiying agent than FA alone. PFA formed *in situ* by reacting FA with H_2O_2, PFA generates highly electrophilic hydroxonium ions (HO^+) under acidic conditions. The HO^+ ion preferentially acts as an electrophile, selectively attacking regions of high electron density, specifically the aromatic rings and olefinic double bonds of the lignin macromolecule (Sun *et al.* 2000a).

The pulp yield decreased with increasing PFA treatment time. The pulp yield dropped from 93.5% to 88.0%, representing a loss of 5.5% after 1 h treatment. The pulp yield further reduced to 83.4% after 4 h PFA treatment, representing a total loss of 8.7 % from the starting FA-treated pulp.

The yield loss is not only attributable to the removal of lignin alone. The PFA also degrades hemicelluloses and, to a lesser extent, cellulose. Peracids are known to generate hydroxyl radicals, which can non-selectively attack carbohydrate polymers, particularly hemicelluloses, leading to their hydrolysis and solubilization (Jääskeläinen *et al.* 2000). The yield loss and reduction in kappa number after the first hour suggests a relatively

Table III. Two stages alkaline peroxide bleaching of FA/PFA treated OCC pulp

FA Treatment time	Yield (%)	Brightness (%)	Viscosity (mPa.s)	Holocellulose (%)	α-cellulose
1	52.46	70.45	2.02	93.67	80.33
2	51.35	74.45	2.34	93.45	83.59
3	49.71	76.67	2.22	93.22	83.91
4	49.38	77.21	2.07	93.35	84.13

(4% +4%) H_2O_2, pH 11, t=80°C Time=1 h

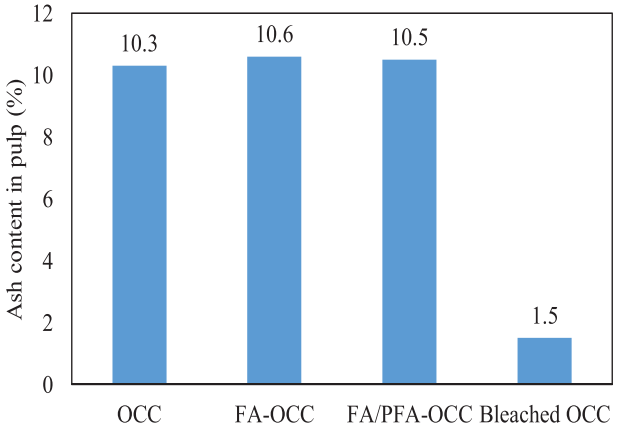

Fig. 3. Ash content at different stage of OCC treatment

favorable selectivity; however, the selectivity diminishes with longer treatment times, as the yield loss continues while delignification slows.

A remarkable improvement in pulp brightness was observed following PFA treatment. The brightness increased from 16.6% (FA pulp, as shown in Table I) to 23.8% after 1 h of PFA treatment, further increased brightness to 24.6% after 4 h of PFA treatment. The increase in brightness is directly associated with the removal of lignin chromophores by the oxidative action of peroxyformic acid. Peracids are effective in depolymerizing and solubilizing lignin (Ma *et al.* 2021). The relatively small increase in brightness beyond the first hour suggests that most color-imparting structures were eliminated early in the PFA process, consistent with the plateau observed in kappa number reduction. This implies that while prolonged PFA treatment yields marginal gains in brightness and delignification, it comes at the cost of substantial yield loss.

In conclusion, PFA treatment of FA-delignified OCC pulp proved highly effective for further delignification and brightness improvement.

Bleaching of FA/PFA treated OCC pulp

The FA/PFA-treated OCC pulp (obtained after 2 h of PFA treatment) was then subjected to a two-stage alkaline hydrogen peroxide bleaching sequence. The bleaching was carried out at a temperature of 80°C for 1 h in each stage, with varying peroxide charges. The results, showed in Fig. 2, illustrate the significant impact of alkaline peroxide bleaching on pulp yield and overall process efficiency.

Final brightness of pulp reached to 63.86% by using 1% peroxide in each stage, which increased progressively with the increase of peroxide charge and finally reached to 70.45% by using 4% peroxide in each stage.

The most striking observation from the two-stage alkaline peroxide bleaching was the substantial reduction in pulp yield. The yield decreased dramatically from 87.5% to 64.63% following the bleaching sequence. This represents a total yield loss of approximately 22.9% points, which is considerably higher than what would be expected from lignin removal alone. Further, the yield loss continued with increasing peroxide charges.

The primary explanation for this pronounced yield loss can be explained by higher alkaline conditions (pH 11) during the bleaching process. Several non-fibrous (fillers such as calcium carbonate) components that were retained during the FA and PFA treatments, those were solubilized in such alkaline conditions. During the acidic FA and PFA treatments, these minerals remained largely intact, contributing to the higher observed yields. In addition, alkaline conditions, particularly in the presence of hydrogen peroxide, can promote the peeling reaction and alkaline

degradation of hemicelluloses and amorphous cellulose. The β-alkoxy elimination mechanism leads to the stepwise degradation of reducing end groups, resulting in the loss of carbohydrate polymers (Hosoya *et al.* 2018). This phenomenon has been documented in other studies on organic acid pulping, where the retention of inorganics contributed to inflated yield values (Jahan *et al.* 2006; 2015). As shown in Fig. 3, ash content of OCC pulp increased after FA and FA/PFA treatments. Ash content in the acid-treated OCC pulp increased due to the removal of lignin and hemicelluloses, which is consistent with findings reported in other studies (Jahan *et al.* 2015). The ash content in OCC pulp after alkaline peroxide bleaching drastically decreased to 1.5%. Under the alkaline conditions of peroxide bleaching, silica in the pulp dissolved into sodium silicate (Jahan *et al.* 2006), which was reflected by lower ash content. This result supports the above explanation.

The continuous decrease in pulp yield with increasing peroxide charge further supports the role of alkaline solubilization. Higher peroxide concentrations lead to more extensive generation of HOO^- anion and hydroxyl radicals, which not only enhance delignification but also worsen carbohydrate degradation (Ofoedu *et al.* 2021; Mittal *et al.* 2017).

It can be concluded that the optimization of bleaching conditions is essential to balance the competing objectives of yield retention, delignification, and brightness improvement.

To enhance the brightness of the FA/PFA-treated OCC pulp, a two-stage alkaline peroxide bleaching sequence was also applied under constant conditions: 4% hydrogen peroxide charge in each stage, pH 11, 80°C, and 1 h per stage. The FA/PFA-treated pulps were prepared from OCC subjected to varying initial FA treatment times (1, 2, 3, and 4 h), followed by a fixed PFA treatment of 2 h at 80°C. The bleaching results, including yield, brightness, viscosity, holocellulose content, and α-cellulose content, are presented in Table III.

The pulp yield after the two-stage bleaching sequence ranged from 52.46% to 49.38% for pulps derived from OCC with initial FA treatment times of 1 to 4 h, respectively. The variation in yield across the different FA treatment times was relatively modest, decreasing by only 3.08 % as the initial FA treatment time increased. This suggests that once the FA/PFA delignification and bleaching sequence is applied, the final bleached yield is primarily determined by the cumulative effects of the overall process rather than the specific duration of the initial FA stage, provided that the PFA and bleaching stages are consistently applied.

The overall yield values (approximately 49–52%) are similar to bleached chemical hardwood pulp yields (Jahan *et al.* 2010) but are consistent with yields reported for organic acid pulping of non-wood and recycled fibers (Jahan *et al.* 2007). This yield level reflects the combined removal of lignin, hemicelluloses, and inorganic fillers during the sequential FA, PFA, and alkaline bleaching stages.

Pulp brightness improved substantially with increasing initial FA treatment time, rising from 70.45% for the 1 h FA-treated pulp to 77.21% for the 4 h FA-treated pulp. This progressive brightness improvement correlates with the cumulative delignification achieved during the FA stage (as shown in Table 1), where longer FA treatment times resulted in lower kappa numbers prior to bleaching. The final brightness levels of 76–77% are typical for bleached recycled pulps intended for high-quality paper products (Bajpai, 2012).

The alkaline peroxide bleaching stage serves a dual function: it oxidizes and solubilizes residual lignin through the action of hydroperoxide anions (HOO^-), and it effectively bleaches chromophoric groups that survive during the FA/PFA treatments. The increasing brightness with longer FA pretreatment indicates that enhanced initial delignification facilitates subsequent bleaching, requiring less oxidative demand on the peroxide stage.

The viscosity of the bleached pulps, a key indicator of cellulose chain length and pulp strength potential, ranged

Table IV. Papermaking properties of bleached OCC pulp

CSF	Tensile (N.m/g)	Burst index (kPa.m2/g)	Tear index (mN.m2/g)
400	8.5	0.54	1.46
280	38.1	1.09	2.03

from 2.02 to 2.34 mPa·s. The highest viscosity (2.34 mPa·s) was observed for the pulp from 2 h FA/PFA treatment, with slightly lower values at both shorter and longer treatment times. Viscosity values in this range are typical for bleached chemical pulps and suggest moderate cellulose degradation during the combined FA/PFA and bleaching sequence.

The holocellulose content of the bleached pulps ranged from 93.22% to 93.67%, while the α-cellulose content ranged from 80.33% to 84.13%. The α-cellulose content increased progressively with FA treatment time, from 80.33% at 1 h to 84.13% at 4 h, indicating improved cellulose purity with more extensive delignification and hemicellulose removal.

A notable finding is the difference between holocellulose (approximately 93–94%) and α-cellulose (approximately 80–84%), which reveals that the bleached pulps still contain approximately 9–13% hemicelluloses. This is consistent with the behavior of organic acid pulping, where FA is known to partially dissolve hemicelluloses but may retain a significant fraction, particularly xylan, within the fiber structure (Dapia *et al.* 2002; Jahan *et al.* 2015). The presence of residual hemicelluloses is not necessarily detrimental; indeed, hemicelluloses can contribute to fiber bonding and paper strength, though they may also affect pulp refining behavior and optical properties (Sjöström, 1993).

Papermaking properties

The bleached OCC pulp was refined in a PFI mill to a Canadian Standard Freeness (CSF) of 280 mL, and the resulting papermaking properties are presented in Table IV. The tensile index of 38.1 N·m/g, burst index of 1.09 kPa·m2/g, and tear index of 2.03 mN·m2/g indicate that the pulp possesses moderate strength properties suitable for certain paper and board applications.

The presence of residual hemicelluloses (9–13%) may contribute positively to fiber-fiber bonding and tensile strength development during refining (Rahman *et al.* 2025). However, the tear strength is somewhat lower than values typically reported for virgin bleached kraft pulps, reflecting the cumulative effects of fiber degradation during the recycling, delignification, and bleaching processes. The combination of moderate strength properties and high brightness positions this pulp for applications such as white-lined chipboard, printing papers, or as a partial substitute for virgin pulps in various paper grades.

Conclusion

A total chlorine free bleaching sequence based on formic acid, peroxyformic acid, and alkaline peroxide was successfully applied to OCC pulp. Formic acid treatment was efficient in initial delignification, while PFA significantly enhanced lignin removal and pulp brightness. Two-stage alkaline peroxide bleaching resulted in a pulp brightness of 70–77% ISO and a yield of 49–52%. Two-stage peroxide bleaching caused substantial yield loss due to dissolution of inorganic materials present in the OCC pulp. The resulting pulp exhibited moderate viscosity (2.02–2.34 mPa·s), high cellulose content (93%), and acceptable strength properties (tensile index of 38.1 N·m/g, a burst index of 1.09 kPa·m²/g, and a tear index of 2.03 mN·m²/g) for paper and paperboard production. The study confirms that formic acid/Peroxyformic acid delignification combined with peroxide bleaching is a viable chlorine-free approach for OCC upgrading.

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