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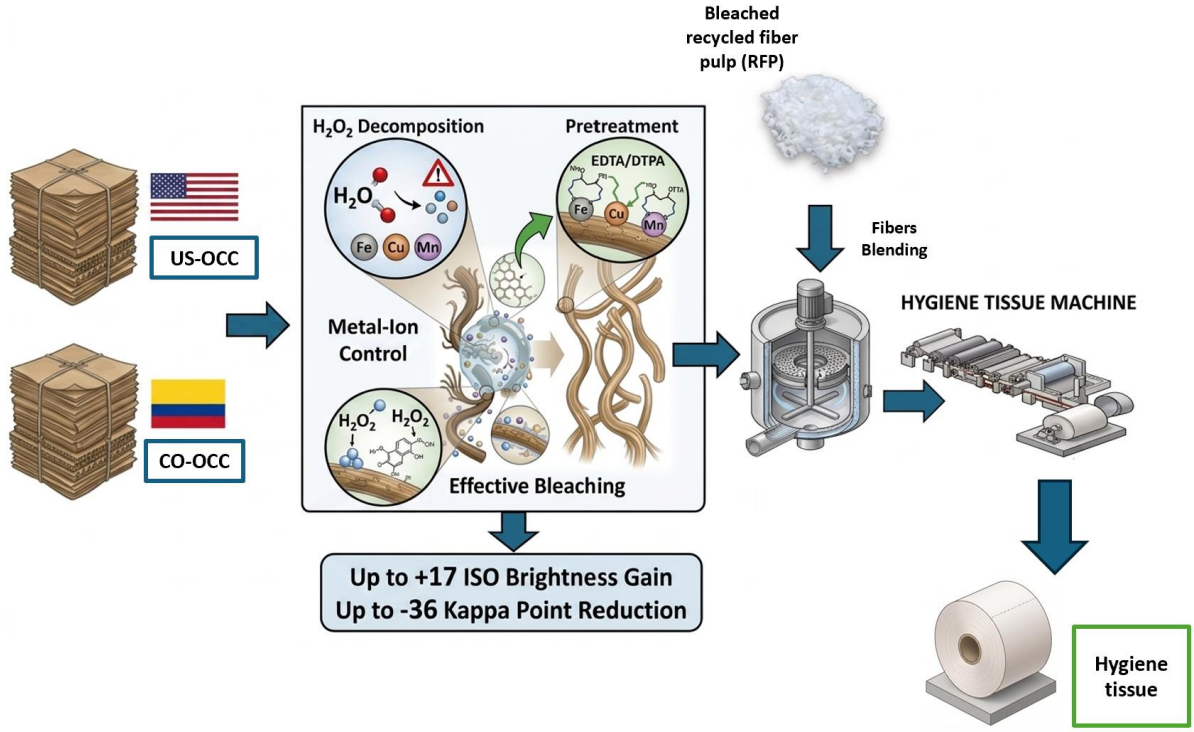
Old Corrugated Containerboard (OCC) Bleaching for Tissue Application: Metal Ion Removal Pretreatments and Hydrogen Peroxide-Based Delignification

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Abstract This study investigates the oxidative bleaching of Old Corrugated Containers (OCC) using alkaline hydrogen peroxide to improve pulp brightness and evaluate its suitability for hygiene tissue applications. As conventional recycled fiber supplies decline, OCC represents a promising alternative recovered fiber source. Metal-ion control pretreatments, including EDTA/DTPA chelation, acid washing, and flotation deinking, were evaluated to remove Fe, Cu, and Mn, which catalyze peroxide decomposition through Fenton-type reactions. Two OCC sources from the southern United States and Colombia were compared to assess the effect of furnish origin on bleachability. The U.S. OCC exhibited higher lignin content (Kappa 89 vs. 61.8), longer fibers (1.7 vs. 0.9 mm), and lower ash content (2.1% vs. 10.1%) than the Colombian OCC. Metal-ion removal improved peroxide stability, resulting in brightness gains of up to 17 ISO points and Kappa reductions of up to 36 points. Bleached OCC was evaluated alone and in blends containing 5–20% commercial bleached recycled fiber pulp (RFP). Although OCC reduced blend brightness, it improved softness, bulk, and compressibility without significant losses in tensile strength, demonstrating its potential as an alternative recycled fiber for tissue manufacturing.

Graphical Abstract



Keywords: Bleaching, Hydrogen Peroxide, Recycling, Tissue, Waste paper, Metal ions.

Introduction

The ongoing digitalization megatrend is driving a steady decline in the production of printed and writing paper (PWP), which directly affects the availability of sorted office paper (SOP), a traditionally high-quality recycled fiber widely used in tissue manufacturing (Vivas et al. 2024). As SOP becomes scarcer and more expensive, the tissue industry faces challenges in maintaining both cost efficiency and product quality (Moore 2019; Morris 2013). In this context, upgrading old corrugated containerboard (OCC) as a complementary fiber source is not only desirable but necessary to ensure a stable and sustainable fiber supply chain (Morris 2019). However, OCC fibers are typically lower in brightness and contain high levels of contaminants, including transition metal ions such as iron (Fe), copper (Cu), and manganese (Mn) (Brown et al. 2022). These transition metals—particularly cuprous (Cu^{+}), ferrous (Fe^{+2}), and manganous (Mn^{+2}) ions—poses a significant challenge during hydrogen peroxide (H_2O_2) bleaching. These metals catalyze peroxide decomposition through Fenton-type reactions, generating highly reactive radicals that reduce bleaching efficiency, decrease peroxide stability, and may compromise fiber integrity (Potucek and Milichovsky 2000; Hosseinpour et al. 2014) As a result, uncontrolled metal content can lead to excessive chemical consumption, limited brightness development, and potential degradation of cellulose chains (Martinsson et al. 2023).

The presence of these transition metals in recovered paper fibers can originate from multiple sources. Residual chemicals used during the original papermaking process, metallic contamination from processing equipment and pipelines, and mineral components inherent to the pulp furnish all contribute to metal accumulation. In addition, fillers, coatings, pigments, and other functional additives incorporated during paper production may introduce or retain metal ions within the fiber matrix (Hubbe et al. 2007; Södö et al. 2007). Moreover, the concentration of transition metals in OCC fibers can vary considerably depending on the contamination level and geographic origin of the material. Differences in containerboard manufacturing practices across regions—such as variations in wood species, recycled fiber availability, industrial infrastructure, economic constraints, and environmental regulations—can influence the metal profile of the final product (JPT 2023; PG Paper 2018; TLP Packaging 2022). To address this issue, effective control and removal of metal ions prior to bleaching are essential, as these metals catalyze the decomposition of bleaching agents and negatively affect brightness development (Goto et al. 2023).

EDTA (ethylenediaminetetraacetic acid) and DTPA (diethylenetriaminepentaacetic acid) act as strong chelating agents capable of forming stable complexes with transition metal ions, thereby reducing their availability to catalyze bleaching reactions (Pinto et al. 2015), Acid washing treatments, on the other hand, promote the solubilization and removal of metal ions by protonating fiber surfaces and disrupting metal–fiber interactions, facilitating their extraction into the aqueous phase (Jahan et al. 2016a).

In addition to chemical pretreatments, deinking is widely used in the recycling of printed fibers and can help reduce the overall metal load. Metal ions are often associated with printing inks, coatings, and fillers, which can remain attached to OCC fibers during recycling. Their partial removal during deinking may therefore further improve bleaching efficiency and chemical stability (Jiang and Ma 2000).

In this study, five pretreatment strategies were systematically evaluated to mitigate the catalytic activity of transition metal ions in OCC fibers prior to hydrogen peroxide bleaching. These strategies included chelation with ethylenediaminetetraacetic acid (EDTA) and diethylenetriaminepentaacetic acid (DTPA), acid washing using dilute sulfuric acid (H_2SO_4) and hydrochloric acid (HCl), and a deinking treatment.

To account for source-related variability, two OCC samples from distinct geographical regions were examined to assess the influence of fiber origin on pretreatment efficiency and bleaching performance. One sample was obtained from the southern United States (US-OCC), and the other was collected in Colombia (COL-OCC).

This research aims to evaluate the effectiveness of the selected pretreatments in controlling transition metal ions prior to hydrogen peroxide bleaching and to quantify their impact on key performance indicators. In addition to ISO brightness, the study assesses critical tissue properties such as softness, tensile index, bulk, and other relevant functional characteristics required for hygiene tissue applications.

Furthermore, the bleached OCC fibers are examined not only as a standalone furnish but also in blends with recycled fiber pulps (RFP) commonly used in hygiene tissue production. This complementary evaluation enables a comprehensive assessment of how pretreated and bleached OCC can integrate with conventional recycled furnish systems, balancing optical, mechanical, and tactile properties. Through this approach, the study determines the feasibility of incorporating bleached OCC as a partial substitute or complementary component in hygiene tissue formulations while maintaining product quality and performance standards.

Experimental

Old container cardboard (OCC) sample collection and preparation

US-OCC pulp samples were collected from a material recovery facility (MRF) located in the southeastern United States, while CO-OCC samples were obtained from a recovered-fiber tissue mill in central Colombia, specifically in the department of Cundinamarca. The selected materials will undergo a series of processing steps, as can be seen in Fig. 1, including repulping and screening, to disintegrate the fibers and remove unwanted contaminants such as large and medium-sized particles. These preparatory treatments will be completed prior to the application of pretreatments preceding the hydrogen peroxide bleaching stage.

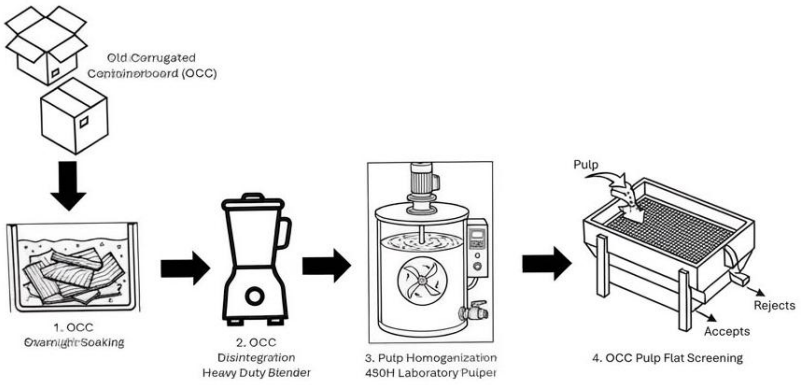


Fig. 1 OCC pulps preparation flow sheet.

Repulping

The OCC samples were initially soaked for at least 12 hours, followed by disintegration in a 1-gallon heavy-duty blender, model Waring CB15 at 20,800 rpm (high speed). Subsequently, the pulp was processed at 70 °C for one hour using a Formax 450H High Consistency Laboratory Pulper at 17 hertz. The resulting pulp reached a consistency of approximately 20%, serving as the arting point for subsequent treatments.

Screening

A lab-scale flat vibrating screen with 0.006-inch slot widths was used to screen the repulped OCC, removing particles of large (100-150 μm) and medium sizes (10-100 μm). In Table 1, are summarized the initial characteristics of both OCC samples.

Table 1 Initial characterization of the OCC samples after repulping and screening

OCC ID	Kappa Number	Brightness (%)	Viscosity (Cp)	Ash Content (%)
US-OCC	89.2	17.2	38.6	2.1
CO-OCC	61.8	23.4	12.6	10.1

Pretreatments for metal ion removal

Chelation (Q)

Chelation pretreatments using EDTA or DTPA were applied to remove transition metals, particularly iron (Fe^{2+}) and manganese (Mn^{2+}), by forming stable metal–chelant complexes. The treatment was carried out by adding 1% (w/w, oven-dried pulp basis) EDTA or DTPA to a pulp slurry at 3% consistency and maintaining it at 50 °C for 60 minutes in a polyethylene bag immersed in a temperature-controlled water bath at pH 5.00.

Acid washing (A)

During the papermaking process, printing inks introduce metal ions and other inorganic materials into the fiber matrix. These contaminants, commonly quantified as ash content, can be effectively removed from OCC pulps through an acid-washing treatment. According to Jahan et al. (2016b), maximum ash removal is achieved at pH 2.0. Therefore, the pulp was acid-washed at this pH using either dilute sulfuric acid (H_2SO_4) or hydrochloric acid (HCl). Sulfuric acid concentrations of 0.020 M and 0.052 M were used for the US-OCC and COL-OCC pulps, respectively, while hydrochloric acid concentrations of 0.032 M (US-OCC) and 0.098 M (COL-OCC) were required to reach the same pH. The pulp consistency was maintained at 3% (w/w), and the treatment was conducted in sealed polyethylene bags immersed in a water bath at 70 °C for 60 minutes.

The required acid concentration depends on both the type of acid and the origin of the OCC. For both OCC samples, lower molar concentrations of H_2SO_4 were needed compared to HCl, reflecting the higher proton efficiency of sulfuric acid. Additionally, CO-OCC sample required substantially higher acid concentrations than the US-OCC sample, indicating a higher buffering capacity or inorganic content. These results suggest that pulp composition and acid selection play a critical role in optimizing acid washing efficiency and chemical consumption.

Deinking (Deink)

Flotation experiments were conducted using a Wemco laboratory flotation machine. A commercial anionic surfactant reagent supplied by Buckman Laboratories International, Inc. under the product line BRD®, was employed to render the ink particles and metal contaminants hydrophobic, facilitating their removal by flotation.

Bleached recycled fibers pulp (RFP)

RFP was obtained from a tissue mill located in the south-central region of North Carolina. The furnish composition consisted of 40% Sorted Office Waste (SOW), 40% Lightly Printed Bleached Kraft (LPBK), and 20% White Ledger (WL), a high-grade deinked office furnish reflecting an essentially typical recycled furnish blend used in hygiene tissue production.

Hydrogen peroxide bleaching (P)

Following pretreatment, the OCC samples were subjected to an alkaline hydrogen peroxide (H_2O_2) bleaching stage to increase the brightness. The bleaching conditions were selected to maximize the formation and stability of the perhydroxyl anion (HOO^-), the primary active species in peroxide bleaching. In aqueous systems, hydrogen peroxide behaves as a weak acid, with reported pKa values between 9.5 and 11.0 over the temperature range of 50–110 °C (Bajpai 2021). Under alkaline conditions, H_2O_2 dissociates to form HOO^-, thereby increasing its nucleophilic reactivity (Karlsson and Agnemo 2010).

The perhydroxyl anion reacts preferentially with carbonyl and conjugated carbonyl groups present in residual kraft lignin and other chromophoric structures. These reactions disrupt light-absorbing groups, resulting in increased ISO brightness (Chen et al. 2003). In addition, HOO^- can promote cleavage of β-O-4 linkages—the most abundant interunit bond in lignin—facilitating lignin fragmentation and improving solubility (Li et al. 2025). Oxidation of phenolic hydroxyl groups may also lead to the formation and subsequent transformation of quinone-type structures (Fig. 2), contributing to chromophore modification and brightness development.

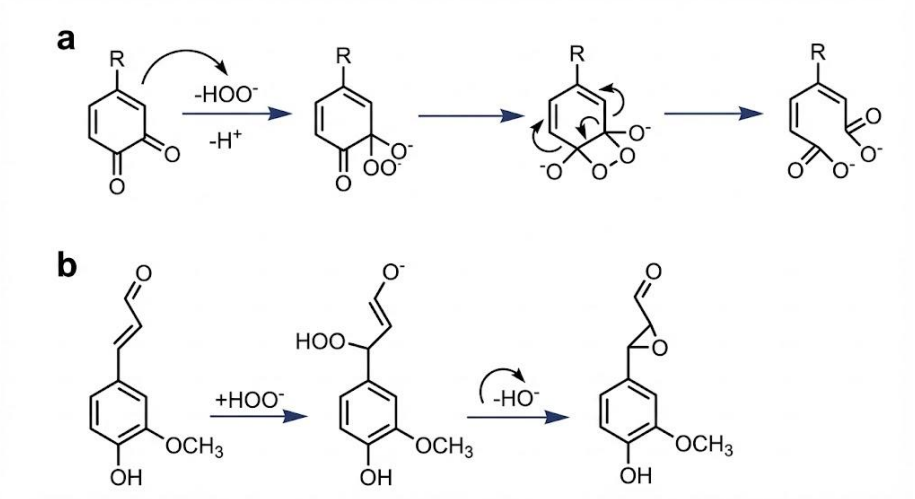


Fig. 2 Alkaline hydrogen peroxide oxidation pathways of phenolic lignin structures. Adapted from Brogdon and Hart (2012).

The efficiency of the peroxide (P) stage depends strongly on the initial kappa number and brightness of the pulp, as well as the applied peroxide dosage. Low-kappa pulps (kappa < 5) typically require modest peroxide charges (0.5–1.5% H_2O_2 on oven-dry pulp) to achieve significant brightness gains due to their relatively low chromophore content.

In contrast, high-kappa pulps such as brown OCC contain elevated levels of residual lignin and oxidized chromophores, resulting in a diminished brightness response at low peroxide dosages (Bajpai 2018; Brogdon and Hart 2012). As can be seen in Fig 2, literature reports indicate that peroxide charges of ≥3% H_2O_2 are generally required to achieve meaningful brightness improvements in high-kappa pulps (Koç et al. 2017; Gulsoy et al. 2013). Based on these considerations, a peroxide dosage of 4.0% H_2O_2 (on an oven-dry pulp basis) was selected to provide sufficient oxidative capacity while maintaining reasonable chemical efficiency. Elevated temperature (90 °C), and extended retention time (2 h) were employed to enhance peroxide reactivity and chromophore removal, conditions consistent with effective bleaching of high-kappa recycled fibers (Karlsson and Agnemo 2010).

Sodium hydroxide was added to establish the alkaline environment necessary for in situ generation of HOO^-. For non-pressurized peroxide stages, NaOH dosages typically range from 80–120% of the peroxide charge (Bajpai 2018; Brogdon and Hart 2012). Accordingly, 3.5% NaOH was applied with 4.0% H_2O_2. Sodium silicate (3.0% on oven-dry pulp) was included as a peroxide stabilizer to mitigate catalytic decomposition by residual metal ions and improve peroxide utilization. Bleaching was conducted at 10% pulp consistency in sealed polyethylene bags immersed in a temperature-controlled water bath. Pulps characterization methods

The OCC samples—both untreated and after pretreatment and peroxide bleaching—were thoroughly characterized using standard TAPPI and ISO methods to monitor chemical, optical, and physical changes during processing for tissue-grade pulp production.

Chemical and compositional analysis

Residual lignin content was quantified by kappa number (TAPPI 1993a) and intrinsic viscosity, determined after holocellulose isolation to mitigate lignin interference in unbleached pulps (TAPPI 1999a; ISO 2010). Holocellulose was prepared by treating 5.0 g pulp with 2.5 mL glacial acetic acid, 7.5 g sodium chlorite, and 200 mL deionized water at 70 °C for 60 min. Ash content (inorganic fraction) was carried out in a muffle furnace at 575 °C in accordance with TAPPI T 211 om-93 (TAPPI 1993b), before/after repulping and screening to assess mineral removal; structural carbohydrates and acid-(in)soluble lignin followed NREL protocols.

Handsheet preparation and tissue properties

Handsheets (30 g m^{-2}) of untreated/treated OCC and 80:20 RFP:OCC blends were formed (ANSI/TAPPI 2024), conditioned, and tested for brightness (TAPPI 2018), tensile index (ANSI/TAPPI 2022), caliper and bulk (TAPPI 2013; 2015), water absorbency (TAPPI 2021), and freeness (TAPPI 1999b). Softness was directly evaluated using a Tissue Softness Analyzer (TSA; Emtec Electronic GmbH, Leipzig, Germany), focusing on the TS7 peak (~6.5 kHz) for micro-surface softness.

Fiber morphology

Fiber fractionation used a Bauer–McNett classifier (TAPPI 2025); dimensions (length, width, etc.) were measured via Fiber Quality Analyzer (FQA) (TAPPI 2023).

Results and discussion

OCC different location comparison

Morphology characterization

Fig. 3.a. and Fig. 3.b. presents the length-weighted fiber length and width distributions of US-OCC and CO-OCC, illustrating differences in fiber morphology between the two sources. Complementing this distribution, Fig. 4 summarizes the results of fiber fractionation using the Bauer–McNett classifier, reporting the length-weighted average fiber length and percentage composition retained in each mesh fraction (14, 28, 48, 200 mesh, and fines). Together, the figure and table provide insight into the structural characteristics of the fibers, which are directly related to their mechanical performance and suitability for tissue applications.

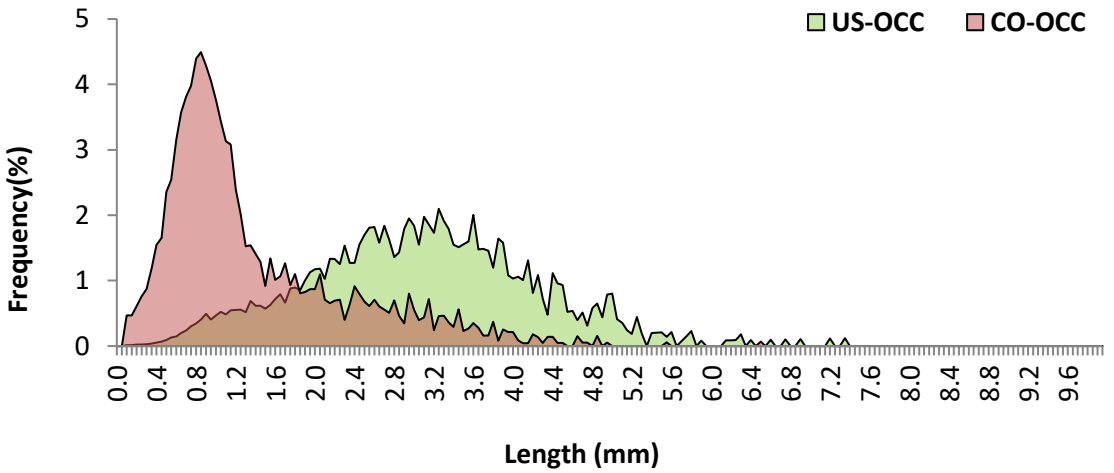


Fig 3.a US-OCC & CO-OCC pulps length weighted frequency.

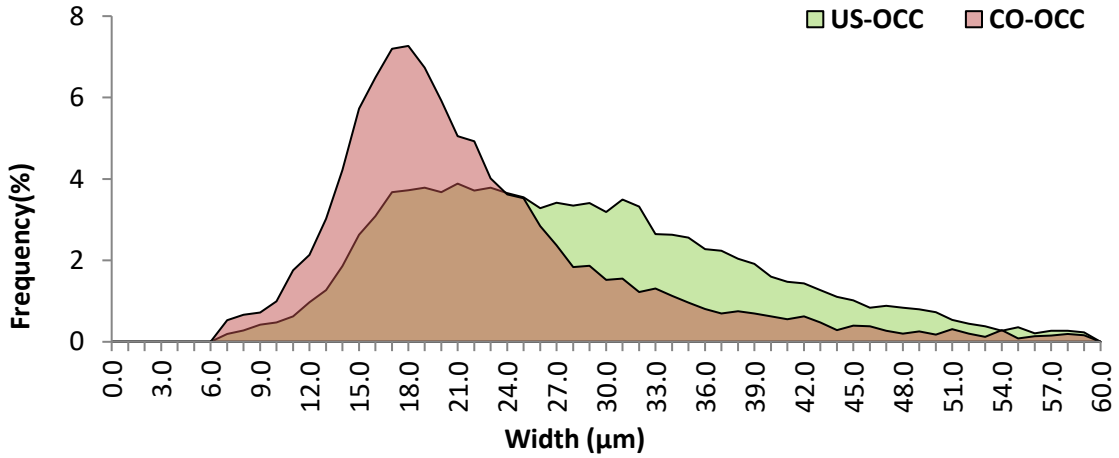


Fig. 3.b US-OCC & CO-OCC pulps Width frequency.

Fig. 3 US-OCC & CO-OCC pulps Fiber Quality Analysis results.

The results show a markedly different fiber length distribution between the two OCC sources. US-OCC contains a substantially higher proportion of long fibers, with 51.0% retained on the 14-mesh screen (1.4 mm) and a length-weighted average of 2.2 mm in this fraction. In contrast, CO-OCC has only 14.5% in the same fraction, although with a slightly higher average length (2.4 mm). This indicates that while CO-OCC contains some very long fibers, they represent a much smaller proportion of the total furnish.

CO-OCC exhibits a greater proportion of medium and short fibers, with 31.0% retained on the 28-mesh screen (0.595 mm) and 22.5% on the 200-mesh screen (0.074 mm), compared to 18.0% and 13.6% for US-OCC, respectively. Additionally, the fines content in CO-OCC (15.1%) is nearly double that of US-OCC (8.2%). These differences are consistent with the expected furnish compositions (Reynolds et al. 2025).

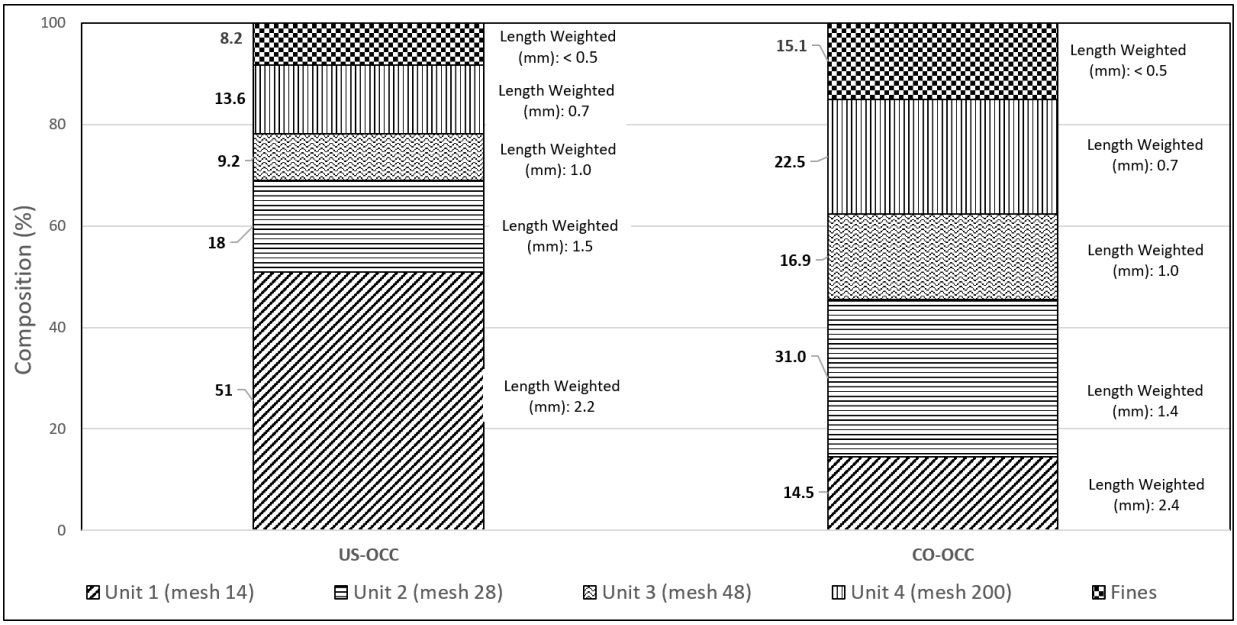


Fig. 4 US-OCC & CO-OCC fiber length results after fractionation.

Notes:
- 14 mesh (1.18 mm).
- 28 mesh (0.6 mm).
- 48 mesh (0.3 mm).
- 200 mesh (0.075 mm).

US-OCC, sourced from the U.S., likely contains a higher proportion of virgin softwood fibers used in linerboard, contributing to the larger fraction of long fibers. In contrast, CO-OCC from Colombia, with a higher recycled fiber content and possible inclusion of hardwood and nonwood fibers, shows a greater proportion of short fibers and fines. From a tissue manufacturing perspective, the higher long-fiber content in US-OCC may contribute to improved tensile strength, whereas the higher short-fiber and fines content in CO-OCC may enhance softness and sheet formation.

Chemical composition

Table 2 summarizes the chemical composition of the two OCC samples and highlights clear differences between US-OCC and CO-OCC, reflecting variations in furnish composition and manufacturing practices.

Table 2. US-OCC & CO-OCC chemical compositional analysis.

Sample Type	'Cellulose'	'Hemicellulose'			'Lignin'		Ash %	TOTAL %
	Glucan %	Xylan %	Galactan %	Arabinose % + Mannose %	AIL %	ASL %		
US-OCC	61.4	7.5	1.8	9.1	10.6	0.4	2.2	93.0
CO-OCC	60.3	8.4	2.0	6.8	9.0	0.7	10.1	97.3

US-OCC is expected to be produced primarily from softwood fibers for linerboard and semichemical hardwood for corrugated medium from the containerboard made in the southern US. According to (Veijalainen 2022), containerboard production in this region (North America) typically contains approximately 70% virgin fibers. This fiber profile is consistent with the relatively high glucan content observed in US-OCC (61.4%), indicative of softwood-rich furnish, along with moderate hemicellulose fractions. The total lignin content is slightly higher than that of CO-OCC, which aligns with the greater contribution of virgin kraft and semichemical pulps. Notably, US-OCC exhibits a low ash content (2.2%), supporting the assumption of a higher virgin fiber proportion and lower recycled mineral contamination.

In contrast, CO-OCC from Colombia likely contains a higher proportion of recycled fibers—estimated at around 75%—with virgin components derived from hardwood and possibly nonwood sources. This is reflected in its slightly lower glucan content (60.3%) and higher xylan content (8.4%), consistent with greater hardwood contribution. Most notably, its ash content (10.1%) is substantially higher, indicating increased mineral content typical of recycled fiber streams and filler accumulation.

Consistent with the compositional differences, the Inductively Coupled Plasma (ICP) results presented in Table 3 further differentiate US-OCC and CO-OCC in terms of inorganic and transition metal content. A direct comparison shows substantially higher concentrations of several metal ions in CO-OCC, which correlates with its higher ash content and supports the presence of greater mineral accumulation and residual contaminants typically associated with recycled fiber streams.

Table 3. Initial US-OCC & CO-OCC Inductively Couple Plasma (ICP) results.

Sample ID	Na (ppm)	K (ppm)	Ca (ppm)	Mg (ppm)	Cu (ppm)	Fe (ppm)	Mn (ppm)
US-OCC	354.0	77.0	1,236.0	178.0	4.0	122.0	6.0
CO-OCC	122.0	134.0	6,968.0	420.0	9.7	435.0	8.0

Regarding transition metals associated with hydrogen peroxide decomposition, both samples contain copper (Cu) and iron (Fe), while manganese (Mn) is detected only in CO-OCC. Iron concentration is markedly higher in CO-OCC (420 ppm) compared to US-OCC (178 ppm), and copper is also higher (12 ppm vs. 9.7 ppm). The presence of Fe, Cu, and Mn—particularly at elevated levels in CO-OCC, highlights the increased potential for catalytic peroxide decomposition via Fenton-type reactions.

These findings reinforce the importance of transition metal control through pretreatment prior to hydrogen peroxide bleaching, especially for OCC sources with higher inorganic content.

Recycled Fiber pulps (RFP)

Figure 4 shows the length-weighted fiber length distribution of the recycled fiber pulp (RFP) used as the reference furnish in the tissue blend formulations. The main physicochemical and morphological characteristics of the RFP are an ash content of 4.5%, an ISO brightness of 74.6%, and a length-weighted average fiber length of 1.1 mm, values that are consistent with recycled-fiber-based pulps used in sanitary and tissue applications (Meyer and Turnbull 2023). The ash content of 4.5% reflects a moderate inorganic fraction attributable to fillers and residual mineral matter typically present in recovered-fiber pulps. The ISO brightness of 74.6% indicates that the RFP is a relatively high-brightness furnish suitable for hygiene tissue products, in line with brightness ranges reported for deinked and bleached tissue-grade recycled pulps (Xin et al. 2016). Morphologically, the length-weighted average fiber length of 1.1 mm places the RFP in the short-to-medium fiber range, which is commonly used in tissue furnishes to balance tensile strength, softness, and formation.

As shown in Figure 4, the fiber length distribution is centred on these intermediate fractions, which are typically associated with good sheet formation, adequate tensile strength, and acceptable softness in tissue products.

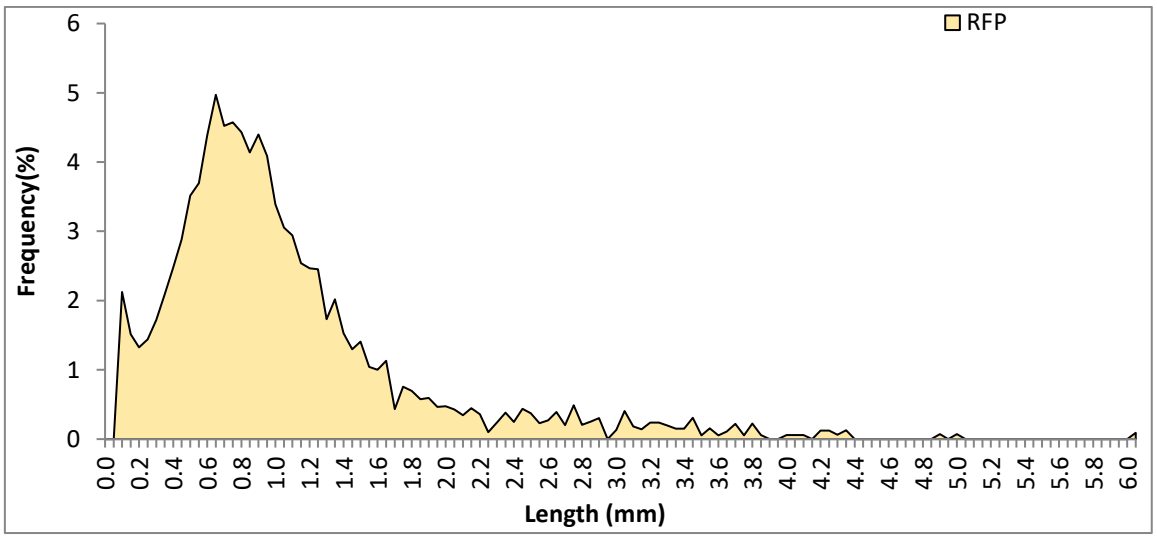


Fig 5. RFP length weighted frequency.

OCC after metal ion control pretreatments

Table 4 and Fig.6 summarize the effects of the different pretreatments—Q1 (EDTA chelation), Q2 (DTPA chelation), A1 (sulfuric acid washing), A2 (hydrochloric acid washing), and deinking—on the main chemical and physical properties of US-OCC and CO-OCC. The results reflect the distinct initial compositions of the two sources: US-OCC, with lower ash and higher lignin content, and CO-OCC, with higher ash and lower kappa number.

For US-OCC, chelation treatments (Q1 and Q2) primarily targeted metal ions without substantially removing mineral fillers, resulting in relatively small reductions in ash content. Consequently, changes in kappa number were limited. In contrast, acid washing (A1 and A2) and deinking promoted greater ash removal. As ash content decreased, the relative proportion of lignin in the remaining pulp solids increased, leading to an apparent increase in kappa number. This increase does not necessarily indicate lignin formation, but rather a shift in composition due to mineral removal. Brightness changes after pretreatments were influenced by both filler removal and partial extraction of coloured impurities, while viscosity tended to increase when carbohydrate content became proportionally higher. The increase in viscosity is attributed to the greater contribution of cellulose and hemicelluloses, which enhance resistance to flow during viscosity measurement.

For CO-OCC, the effects were more pronounced due to its higher initial ash content. Acid washing and deinking resulted in substantial ash reduction, which correspondingly increased the relative lignin fraction and thus the measured kappa number.

Chelation reduced metal content but had less influence on total ash. In several cases, viscosity increased after pretreatment, consistent with enrichment of structural carbohydrates following removal of inorganic components. Brightness responses reflected the balance between filler removal, metal extraction, and changes in chromophore structures.

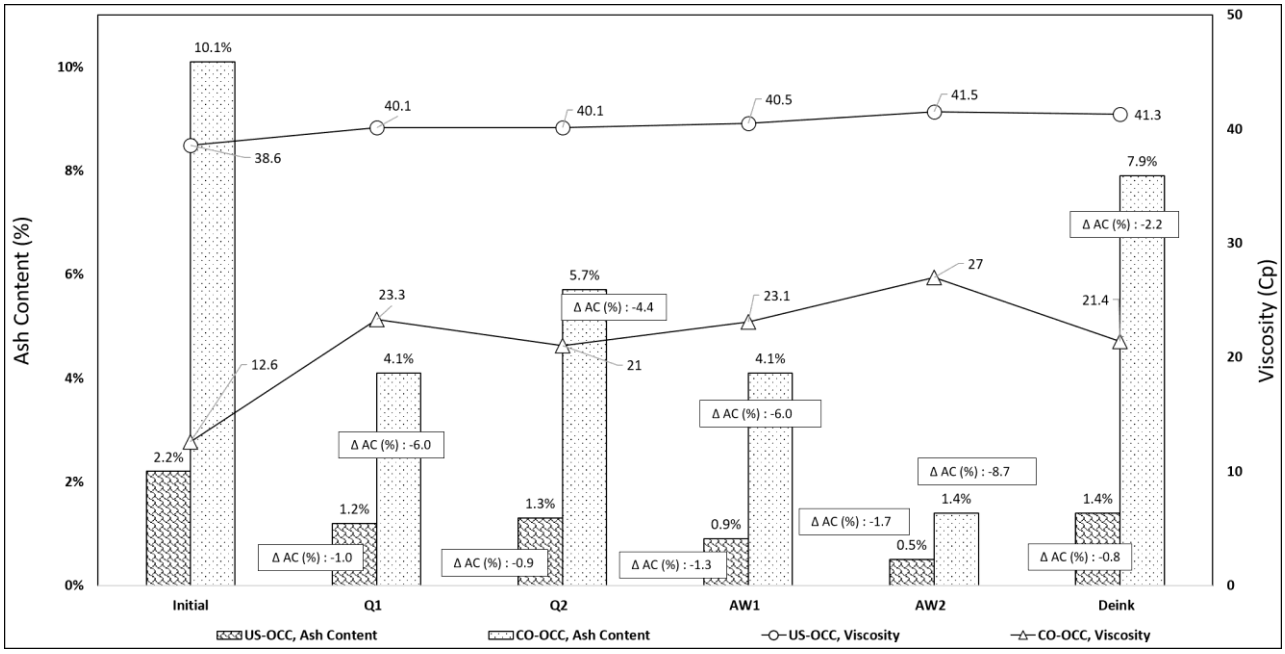


Fig. 6 UC-OCC & CO-OCC ash content and viscosity results after the metal ion control pretreatments.

Note: ΔAC (%) indicates the change in ash content, calculated as the difference between post-treatment and pre-treatment values.

Table 4. US-OCC & CO-OCC characterization results with and without metal ion removal pretreatments.

Treatment	Yield US-OCC (%)	Yield CO-OCC (%)	Kappa Number US-OCC	Kappa Number CO-OCC	Brightness US-OCC (%)	± SD (%)	Brightness CO-OCC (%)	± SD (%)
Initial OCC	—	—	89.3	61.8	17.1	0.20	23.4	0.10
Q1 (EDTA)	96.6	91.7	91.2	61.0	19.1	0.20	23.9	0.30
Q2 (DTPA)	95.3	92.8	84.5	60.1	19.2	0.30	24.1	0.30
A1 (H2OS4)	93.6	89.8	87.9	63.3	18.1	0.20	21.9	0.20
A2 (HCl)	91.5	85.7	85.6	60.4	19.8	0.20	25.20	0.20
Deink	83.7	80.6	87.8	57.2	17.8	0.30	22.60	0.10

Notes:
All standard deviations (± SD) were calculated from samples tested in triplicate.

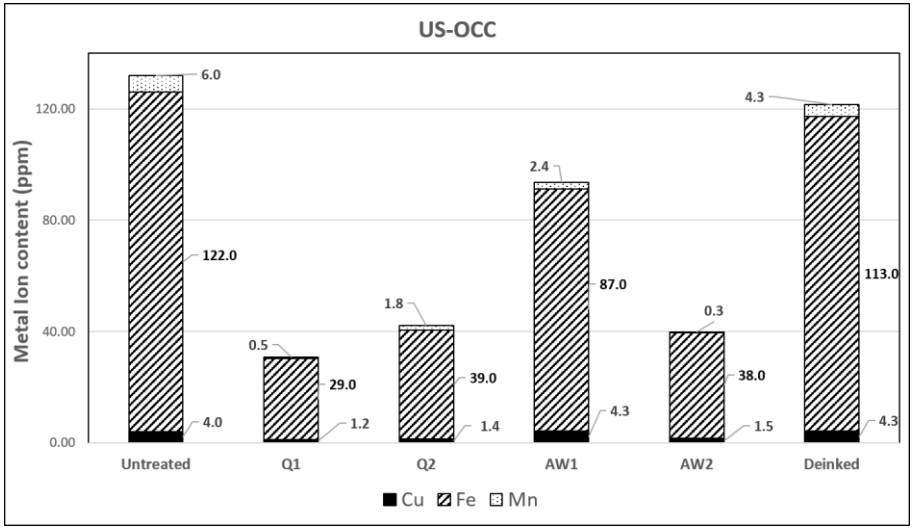


Fig. 7.a US-OCC ICP results after metal ion control pretreatments.

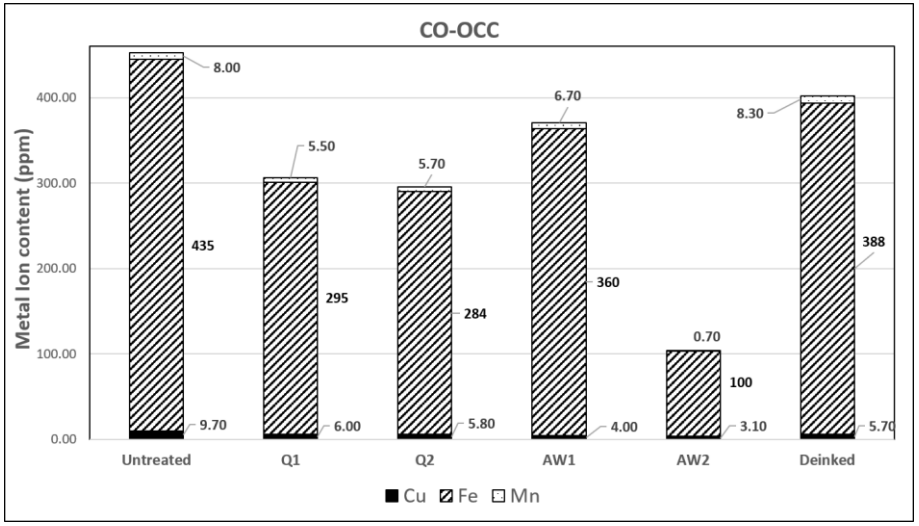


Fig. 7.b CO-OCC ICP results after the metal ion control pretreatments.

Fig. 7 US-OCC & CO-OCC Inductively Couple Plasma (ICP) results after the metal ion control pretreatments.

ICP Analysis after Metal Ion Control Pretreatments

Fig. 7 presents the residual Cu, Fe, and Mn concentrations in the US-OCC and CO-OCC samples after the different metal ion control pretreatments. Overall, all chelation and acid washing treatments reduced metal concentrations to varying degrees, whereas deinking alone showed limited effectiveness.

For the US-OCC (Fig. 7a), EDTA (Q1) and hydrochloric acid washing (AW2) were the most effective treatments, producing the greatest reductions in Fe and Mn. DTPA (Q2) also decreased all three metals but to a lesser extent, while sulfuric acid washing (AW1) showed only moderate removal and did not reduce Cu. Deinking resulted in only minor changes compared to the untreated sample.

The CO-OCC (Fig. 7b) contained substantially higher initial concentrations of Cu, Fe, and Mn than the US-OCC. Among the pretreatments, AW2 was the most effective, reducing Cu, Fe, and Mn to 3.1, 100, and 0.7 ppm, respectively. EDTA and DTPA achieved moderate reductions, whereas AW1 and deinking were less effective.

The superior performance of hydrochloric acid washing (AW2) over sulfuric acid washing (AW1) can be explained by the different chemical behavior of chloride and sulfate ions during iron dissolution (Figure 8).

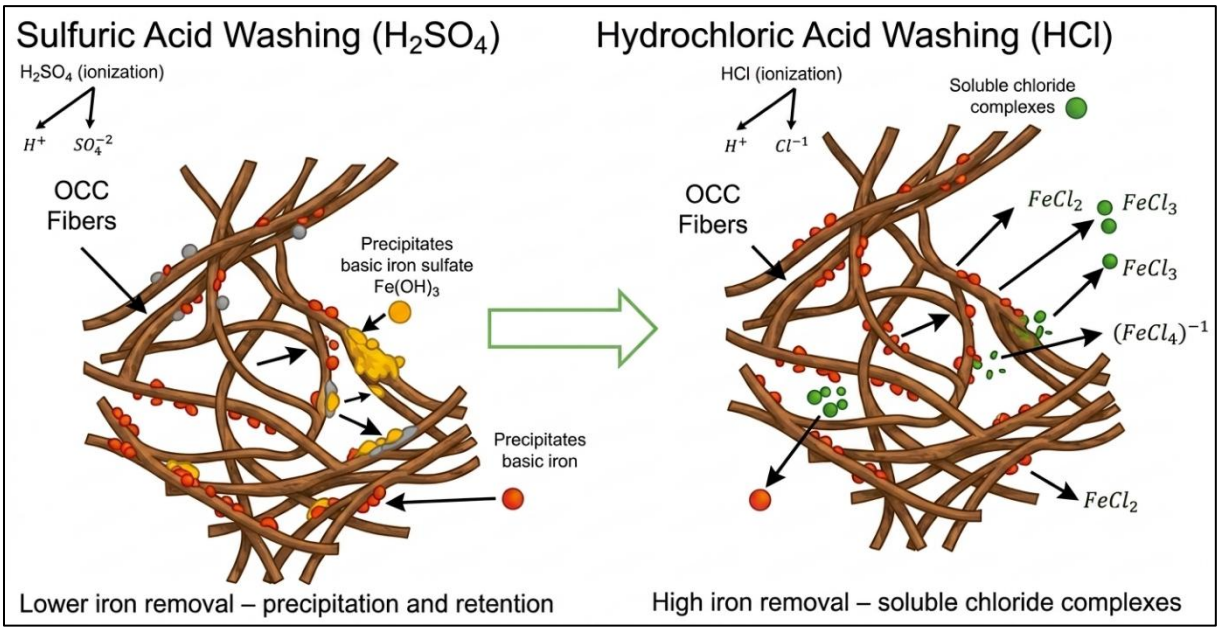


Fig. 8 Comparison between AW1 and AW2 iron dissolution mechanisms.

Although both acids protonate the fiber surface and promote the release of metal ions, chloride ions readily form soluble iron-chloride complexes (e.g., $FeCl_2$, $FeCl_3$, and $FeCl_4^-$), which remain in solution and are efficiently removed during washing. In contrast, sulfate ions tend to promote the formation of sparingly soluble basic iron sulfate and iron hydroxide species that can precipitate and remain entrapped within the fiber network, thereby reducing the overall iron removal efficiency. This mechanism is consistent with the significantly lower residual Fe concentrations obtained after AW2 in both OCC sources (Stumm and Morgan 1996; Cornell and Schwertmann 2003; Pourbaix 1974).

Hydrogen peroxide bleaching results

Table 5 presents the characterization results of OCC samples after hydrogen peroxide bleaching (P), emphasizing how each pretreatment affected brightness development and kappa number reduction compared with the untreated pulps. All pretreatments improved bleaching performance in both US-OCC and CO-OCC, as shown by higher final brightness and lower kappa numbers.

For US-OCC, the untreated pulp reached 27.1% brightness and a kappa number of 68.8 after bleaching. Chelation treatments produced the most significant improvements: Q1 (EDTA) achieved the highest brightness (34.3%) and the greatest brightness gain (Δ17.3 points) as well as the largest kappa reduction (Δ35.6). Q2 (DTPA) yielded comparable results, with 33.1% brightness and a Δ31.7 kappa reduction. In contrast, acid washing (A1 and A2) and deinking treatments offered moderate benefits, raising brightness to about 29.5–29.9% and reducing kappa by 26.3–29.0 points.

A similar trend occurred in CO-OCC, though the magnitude of change varied due to its lower initial kappa number and higher ash content. The untreated CO-OCC reached 34.0% brightness after bleaching, whereas Q1 (EDTA) and A2 (HCl) yielded the brightest pulps—38.6% and 40.1%, respectively—and the greatest kappa reductions (Δ20.0 and Δ21.0). These findings suggest that transition metal removal via chelation enhances peroxide reactivity more effectively than acid washing or deinking alone.

Bleached OCC fibers as complement to recycled fibers pulps (RFP)

Bleached OCC & RFP blends brightness

To assess the potential of using bleached OCC fibers as a potential raw material to RFP in tissue production, blends of RFP with OCC—both bleached with and without metal ion control pretreatments—were prepared. Fig. 9 presents the changes in brightness as the proportion of bleached OCC fibers in the blends increases, illustrating how OCC incorporation and pretreatment influence optical properties.

Blends were prepared at varying OCC proportions: 5%, 10%, 15%, and 20% of the total pulp. Fig. 9 also includes a comparison of the resultant brightness from each blend and the brightness expected for a consumer (at-Home) and commercial (away-from-home) quality level, as stablished by McKinney 1995. These results illustrate how both the type of OCC pretreatment and its proportion in the blend influence the optical properties of the final tissue pulp.

The data reveal a clear trend: increasing the proportion of bleached OCC in the blend results in a gradual decrease in ISO brightness for all treatments. This is expected, as OCC fibers, even after bleaching, exhibited lower brightness than RFP (74.6% ISO Brightness). Among the pretreatments, Q1 (EDTA) and Q2 (DTPA) chelation generally produced the highest brightness values for both US-OCC and CO-OCC blends, indicating that controlling transition metal ions prior to peroxide bleaching enhances optical performance in the resulting pulp blends.

Comparing the two OCC sources, CO-OCC blends consistently show slightly higher brightness than US-OCC blends at the same incorporation levels, likely due to its higher initial brightness after bleaching and effective metal ion stabilization during pretreatment.

Table 5. OCC characterization results after Hydrogen Peroxide bleaching (P).

Sample ID	Brightness US-OCC (%)	± SD (%)	Δ Brightness US-OCC (%)	Brightness CO-OCC (%)	± SD (%)	Δ Brightness CO-OCC (%)	Kappa Number US-OCC	Δ Kappa Number US-OCC	Kappa Number CO-OCC	Δ Kappa Number CO-OCC
Untreated	27.1	0.2	10.0	34.0	0.2	10.6	68.8	20.4	46.1	15.7
Q1 (EDTA)	34.3	0.1	17.3	38.6	0.2	15.2	55.6	35.6	41.0	20.0
Q2 (DTPA)	33.1	0.1	16.0	37.1	0.2	13.7	52.8	31.7	43.8	16.3
A1 (H_2OS_4)	29.8	0.3	12.7	36.8	0.3	13.4	58.9	29.0	44.9	18.5
A2 (HCl)	29.9	0.3	12.9	40.1	0.1	16.7	59.3	26.3	39.5	21.0
Deink	29.5	0.6	12.5	34.9	0.6	11.5	60.6	27.2	40.1	17.1

Notes:
All standard deviations (± SD) were calculated from samples tested in triplicate.

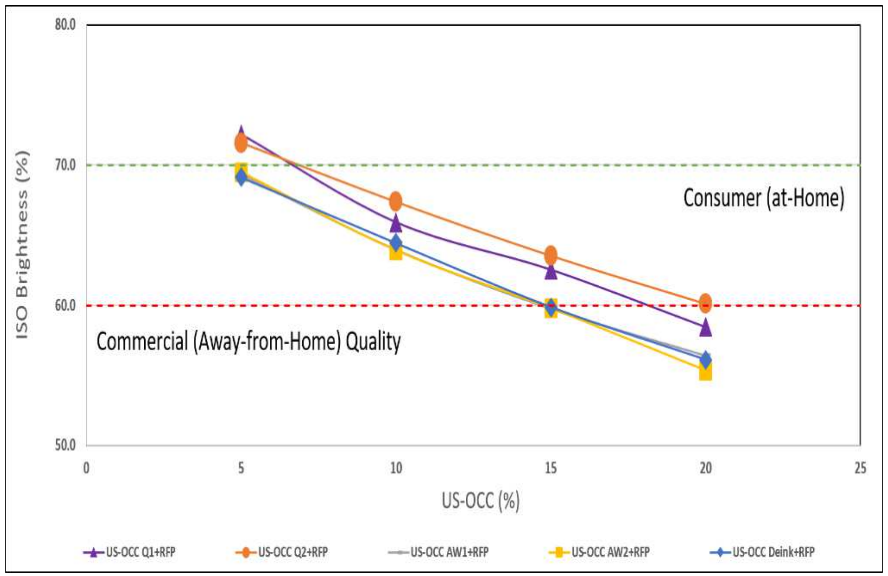


Fig. 9.a RFP:US-OCC blends ISO Brightness results.

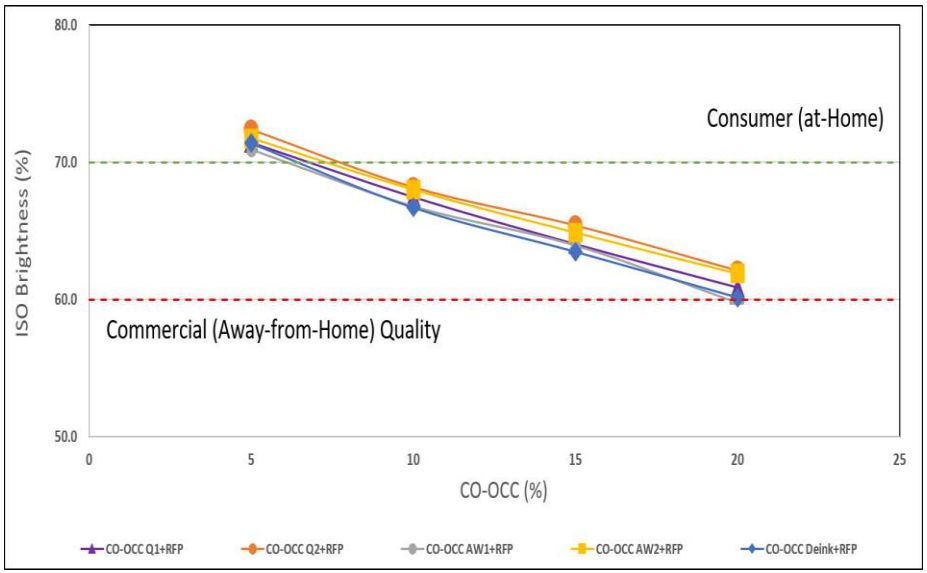


Fig. 9.b RFP:CO-OCC blends ISO Brightness results.

Fig 9. Effect of CO-OCC Content on ISO Brightness of OCC: RFP Blended Pulps Compared to Commercial and Consumer Tissue Standards.

Bleached OCC & RFP blend tissue properties

Table 6 presents the effect of incorporating 20% bleached OCC fibers into recycled fiber pulp (RFP) on key tissue properties, including softness (TS7), tensile index, caliper, bulk, water absorbency, and freeness. The results compare the performance of 100% OCC, 100% RFP, and 80:20 RFP: OCC blends after different pretreatments followed by peroxide bleaching (Q1P, Q2P, AW1P, AW2P, and DeinkP).

The RFP handsheet exhibits an exceptionally high TS7 value (61.4 dB), indicating a significantly lower "real softness" compared to OCC fibers. This is primarily due to the inherent stiffness of its deinked recovered fibers (40% SOW, 40% LPBK, 20% WL), which are typically hornified and contain a higher proportion of fines from prior processing. These characteristics generate high-frequency acoustic vibrations (~6.5 kHz) that are interpreted by human mechanoreceptors as a harsher surface feel (Pawlak et al. 2022).

Furthermore, RFP displays a markedly higher tensile index (33.1 Nm/g) and lower freeness (435 mL CSF) than pure US-OCC or CO-OCC, suggesting extensive prior refining and fibrillation. This mechanical treatment increases fiber conformability and creates secondary fines that fill the sheet structure, leading to enhanced inter-fiber bonding and a denser, lower-bulk sheet (3.7 cm^3/g) (De Assis et al. 2018). In blends, these fibers maintain necessary tensile levels, allowing manufacturers to optimize the classic strength–softness–freeness trade-off by targeting softness improvements while utilizing the high bonding potential of chemically bleached pulps.

Table 6 also details benchmarks for non-creped tissue handsheets (~30 g/m^2) made from deinked recovered fibers, such as SOP or similar DIP furnishes. These metrics provide a baseline from lab and pilot studies to evaluate the OCC/RFP blends against industry norms.

After blending 20% treated OCC with 80% RFP, the resulting tissue properties consistently fell between pure OCC and RFP extremes, aligning well with literature benchmarks for deinked recovered fiber (DIP/SOP) handsheets. TS7 values (34–54 dB) increased relative to OCC baselines (31–34 dB) but stayed below RFP's 61.4 dB and within typical DIP ranges (45–65 dB), reflecting RFP's stiffer fibers dominating micro-surface softness. Tensile index (20.6–24.9 Nm/g), caliper (113–146.7 μm), and bulk (3.7–7.4 cc/g) were intermediate—matching benchmark targets (tensile 15–25 Nm/g, caliper 120–180 μm, bulk 4.0–6.0 cc/g)—while freeness (430–504 mL CSF) and water absorbency (3.7–5.1 g/g) balanced OCC's more open structure against RFP's denser, lower-CSF (435 mL) network, enabling viable tissue performance.

Table 6. Effect of Bleached OCC Incorporation on tissue properties in RFP Blends[1].

Sample ID	Softness (TS7) (dB)	± SD (dB)	Tensile Index (KNm/Kg)	± SD (KNm/Kg)	Caliper (µm)	± SD (µm)	Bulk (cc/g)	Water Absorbancy (g/g)	Freeness (CSF)
Benchmark Range (DIP/SOP-based)[2]									
Initial US-OCC 100%	31.0	1.3	21.6	1.1	158.8	3.8	4.9	6.1	523
Initial CO-OCC 100%	34.4	5.9	20.8	1.1	122.4	5.5	4.8	5.4	481
RFP 100%	61.4	18.2	33.1	3.1	106.5	4.8	3.7	5.0	435
20% US-OCC Untreated: 80% RFP	46.9	6.5	21.3	1.3	104.4	8.6	3.3	5.0	449
20% CO-OCC Untreated: 80% RFP	38.5	4.8	20.6	1.4	118.5	4.2	3.8	5.7	453
20% US-OCC Q1P: 80% RFP	34.3	6.0	22.8	0.9	116.0	3.6	3.8	5.9	473
20% CO-OCC Q1P: 80% RFP	35.4	6.4	23.9	1.2	113.0	5.1	3.7	5.2	450
20% US-OCC Q2P: 80% RFP	44.4	8.6	20.6	1.5	146.7	4.8	5.1	5.9	470
20% CO-OCC Q2P: 80% RFP	47.7	11.5	24.3	0.5	131.4	5.7	4.4	5.9	470
20% US-OCC AW1P: 80% RFP	43.6	9.9	21.3	1.6	119.8	5.9	4.3	5.7	498
20% CO-OCC AW1P: 80% RFP	48.3	11.4	24.9	2.0	116.3	7.4	4.2	5.4	470
20% US-OCC AW2P: 80% RFP	46.3	7.7	24	2.0	118.4	5.2	4.2	5.4	504
20% CO-OCC AW2P: 80% RFP	54.0	17.5	23.4	1.5	134.1	4.8	4.6	5.5	430
20% US-OCC DeinkP: 80% RFP	43.5	6.9	22.2	1.5	134.9	4.2	4.7	5.7	501
20% CO-OCC DeinkP: 80% RFP	37.8	6.2	22.4	2.4	120.6	6.3	4.3	5.7	476

Notes[1]: All standard deviations (± SD) were calculated from samples tested in quintuplicate.
Notes[2]: Benchmark properties for non-creped deinked tissue sheets (typically ~30 g/m² basis weight)(Urdaneta et al., 2025).

Conclusions

Properly selected metal-ion removal pretreatments, including chelation, acid washing, and deinking, substantially reduce the ash and transition-metal content of OCC, improving hydrogen peroxide stability and enabling more effective delignification and brightness development at a fixed peroxide charge. Chelation with EDTA or DTPA and hydrochloric-acid washing emerge as the most effective strategies for mitigating Fe, Cu, and Mn catalysis, yielding the largest kappa number reductions and the highest post-bleaching brightness while preserving or even increasing pulp viscosity. The two OCC sources exhibit distinct responses to pretreatment and bleaching, indicating that furnish origin—encompassing virgin/recycled ratio, ash level, metal profile, and fiber morphology—is a critical design parameter when upgrading OCC for tissue applications. Incorporating up to 20% peroxide-bleached OCC into a high-brightness RFP furnish decreases ISO brightness but consistently maintains tensile indices within acceptable ranges for hygiene tissue grades. Overall, alkaline hydrogen peroxide bleaching combined with appropriate metal-ion control enables OCC to function as a complementary fiber source that enhances tactile properties and supports greater circularity in tissue manufacturing without unacceptable penalties in strength or absorbency.

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Data Availability The authors are committed to supporting research transparency. The datasets and materials presented in this study may be available by the corresponding author upon reasonable request.

Declarations

Conflict of interest I would like to submit our manuscript entitled '*Old Corrugated Containerboard (OCC) Bleaching for Tissue Application: Metal Ion Removal Pretreatments and Hydrogen Peroxide-Based Delignification*' to ***Cellulose.*** The authors declare that no conflict of interest exists with respect to the research, authorship, and publication of this article. All authors have read and approved the manuscript. The authors also certify that the manuscript contains original work, has not been published before, and is not being considered for publication by any other journal.

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