



Tuning chemithermomechanical pulps to achieve sustainable packaging materials: study of fines and lignin content

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Abstract

Given the declining demand for newsprint and the rising demand for packaging materials, new applications for high-yield pulps (HYPs), such as sustainable packaging, are being developed. While the traditional use of HYPs as a major component in paperboard is growing alongside this demand, their use in other packaging types with different property demands requires quality modifications or improvements to enhance mechanical strength and/or barrier properties. The research presented here explores the role of lignin and lignin-rich fine content, combined with hot-press technology, in improving the paper produced with chemithermomechanical pulp (CTMP). Critical properties for some packaging materials, as tensile strength (dry and wet) and air permeability were evaluated. Results indicate that moderate delignification (15%) or increased fines content together with hot-pressing improves the evaluated properties. The highest dry tensile strength was achieved through soft delignification, tripling the resistance (from 27 to 83 kN m/kg). Maximum wet strength (28 kN m/kg) was obtained with 35% fines content and 260 °C hot-pressing, which also resulted in the densest sheets. Air permeability was significantly reduced, either through partial delignification or by increasing the fines content, resulting in values decreasing from approximately 2000–20 mL/min. This approach aims to develop more sustainable packaging materials without relying on wet strength additives typically derived from fossil raw materials.

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Introduction

Among the alternatives to plastics, high yield pulps (HYPs) offer a promising solution, as they require less biomass than other pulps due to their high production yield (>85%) (Mboowa 2021). Traditionally, these pulps mainly stone groundwood pulp (SGW) and thermomechanical pulp (TMP) have been used in newsprint and magazine paper grades due to their adequate tensile strength, high light scattering and their excellent printing properties. SGW and TMP have also been used for a long time in packaging papers mainly as middle layer in paperboard and boxboard. During the seventies, chemithermomechanical pulps (CTMPs), were developed from TMP by introducing chemical impregnation with alkaline sodium sulfite before preheating. This chemical pretreatment reduces energy consumption for defibration of woodchips to free fibers in the subsequent mechanical processing of the pulp while maintaining yields of approximately 85–95% (Hoglund and Bodin 1976; Biermann 1996; Roffael et al. 2001). Today CTMP and BCTMP (bleached) made from hardwoods and softwoods are increasingly used as major component (middle layer pulps) in paperboard and boxboard as well as in hygiene products as tissue.

As the demand for printing papers has drastically declined in favor of digital formats, there is an opportunity to repurpose these pulps for more sustainable packaging and hygiene products. With the rise of parcel shipments and e-commerce, the demand for such materials has increased. However, some types of packaging products require high mechanical and barrier properties, making it necessary to enhance and adapt papermaking processes to improve the quality of CTMP papers (Joelsson et al. 2020). Traditionally, quality improvements have been achieved by increasing and/or improving the efficiency of the specific refining energy used in the production process, or by using various additives (Sandberg et al. 2020; Berg et al. 2022). Refining is widely implemented at the industrial level, as it promotes fiber fibrillation, which enhances fiber bonding. Nevertheless, it is highly energy-intensive (Gharehkhani et al. 2015). Strength-enhancing additives include retention aids, wet strength additives and sizing agents. For example, cationic starch can act as a retention aid although also functioning as a dry strength agent and binder (Sharma et al. 2020). Internal sizing agents as rosins, alkyl ketene dimer (AKD), or alkenyl succinic anhydride (ASA) are introduced into cellulosic slurries at the wet-end of the papermaking process to render the final product hydrophobic, allowing it to resist water and other fluids (Ginebreda et al. 2012; Ntifa et al. 2023; Korpela et al. 2021). Wet strength additives reinforce fiber bonding by limiting water absorption (Lindström et al. 2005; Schäfer et al. 2021). Most of these additives are synthetic compounds, like urea–formaldehyde, melamine–formaldehyde and polyamideamine-epichlorohydrin resins (Häggkvist et al. 1998; Schäfer et al. 2021). Additionally, some sizing agents, including AKD, can also enhance wet resistance (Korpela et al. 2021). On a smaller scale, some biopolymers, for example, chitosan or lignin-derived particles, have also been studied for their hydrophobic properties, making them potential wet-strength agents (Hamzeh et al. 2013; Wang et al. 2021; Huang et al. 2023; Sanchez-Salvador et al. 2024a). These hydrophobic biopolymers originate from two of the most abundant biopolymers in the world, lignin and chitin, ensuring their widespread availability.

Over the past decade, numerous studies have demonstrated that hot-pressing technology enhances the mechanical and physical properties of paper with significant improvements in density, air permeability reduction, as well as wet and dry tensile strength (Norgren et al. 2018; Joelsson et al. 2020; Negro et al. 2023; Sanchez-Salvador et al. 2024b). Among the process parameters, hot-pressing temperature has been identified as the most critical factor for lignin-rich pulps (Joelsson 2021), enabling a permanent densification of the paper. In particular, hot-pressing improves significantly wet resistance at the same time that dry strength is also improved. This is attributed to lignin softening, that act as a binder (Cristescu et al. 2015; Norgren et al. 2018; Joelsson et al. 2020).

In this paper, rather than adding new additives to reinforce CTMP handsheet properties, we investigated the potential role of both lignin and fines content in the CTMP on the mechanical paper properties. Lignin, one of the three most abundant biopolymers globally and the most prevalent aromatic compound in nature, reinforces plant cells by embedding cellulose and hemicellulose, providing structural rigidity to the cell walls and protecting against biological stresses (Moreno and Sipponen 2024). Its complex three-dimensional amorphous structure, formed by branched polyphenolic polyethers with diverse functional groups, has a hydrophobic nature (Beisl et al. 2017; ur Rahman et al. 2018), making it useful in particle form for numerous applications, including reinforcement of polymers (PVA, PLA or cellulose) and the production of Pickering emulsions (Nypelö et al. 2015; Yang et al. 2016; Beisl et al. 2017; Zhang et al. 2023).

On the other hand, fines, defined as the non-homogeneous fraction of a fiber suspension that passes through a 200-mesh wire (Luukko 1998), are generated during the pulping process (Lamminen 2020). There are different types of fines, which vary in specific gravity and size, for instance, fibrils, flakes, or ray cells (Sundberg and Holmbom 2004), although their chemical composition and surface charge is also important (Retulainen et al. 2001). This justifies the existing controversy about their effect on some paper properties, since in some studies, fines have been fractionated based solely on size, using for example the Bauer-McNett fraction method (Pettersson et al. 2021). In the case of CTMP, the chemical composition of the various fractions of CTMP fines differs significantly from the initial pulp before passing through the 200-mesh netting. According to Sundberg and Holmbom (2004), after dynamic drainage jar (DDJ) filtration of a spruce CTMP pulp containing 40% cellulose and 29% total lignin, the retained fibers had a slightly higher proportion of cellulose than the initial pulp, while all fine fractions showed lower cellulose and higher lignin content. Based on their properties, fines may play a significant role in the paper properties (Odabas et al. 2016), especially for CTMP products.

This study explores new approaches to enhancing the mechanical properties of CTMP papers by tuning their composition without introducing additional additives beyond those already present in the raw materials. The two strategies investigated focus on understanding the role of lignin and fine content in paper mechanical properties. Furthermore, the synergy between composition and the hot-pressing technology has also been studied to develop more sustainable packaging materials.

Materials and methods

Materials

A CTMP from SCA Östrand mill (Timrå, Sweden) was used as the cellulose raw material. The CTMP was produced from bleached, flash-dried spruce (CSF of 420 mL). For pulp production, spruce chips were first pretreated by impregnation with a 2 wt% sodium sulphite solution to soften the lignin. For the same purpose, the chips were heated with steam at 115–135 °C, simultaneously promoting more energy-efficient fiber separation. The CTMP was then refined in a pressurized system with a total energy of 1200 kWh/bdt, followed by peroxide-bleaching and flash-drying, resulting in a yield of approximately 95%. The chemicals used for CTMP delignification were a 25 wt% solution of NaClO₂, glacial acetic acid, and a 72 wt% H₂SO₄ was used for chemical composition characterization.

Methods

CTMP delignification

The CTMP sample from the mill was gently treated to remove part of the Klason, or insoluble lignin, using sodium chlorite and acetic acid, a method originally known as the Wise method (Le 1946). The Wise method, widely used for delignification is usually performed at 60–70 °C for several hours, up to 8 h, with fresh sodium chlorite and acetic acid added at loadings of 0.3–0.6 g sodium chlorite/g dry biomass and 0.1–0.6 mL acetic acid/g dry substrate (Kumar et al. 2013; Wang et al. 2018).

In this study, three delignification conditions were used, Batches of 80 g of CTMP were used at 6 wt% consistency. In all cases, the delignification process was carried out inside a 70 °C bath with manual kneading in a dark bag. The mildest delignification condition (DL15) was 0.3 g of NaClO₂ and 0.1 mL of glacial acetic acid per gram of CTMP, all added at the beginning for 15 min. The second treatment (DL60) doubled the NaClO₂ and glacial acetic acid doses (0.6 g NaClO₂ and 0.2 mL glacial acetic acid). Half was added at the beginning of the reaction and the other half was added after 30 min, for a total reaction time of 60 min. Finally, the most severe delignification (DL120) followed the same quantities of chemical reagents as DL60, but extended the reaction time to 2 h. After delignification reaction, the CTMP pulp was washed and filtered, first with tap water and then using distilled water until a neutral pH was achieved. Samples were stored at 4 °C with a few drops of biocide for preservation.

Fines production

Fines were extracted and quantified from the reference CTMP by passing a 1 wt% suspension through a Dynamic Drainage Jar (DDJ) tester with a 200-mesh wire screen, according to the SCAN-CM 66:05 standard (SCAN 2005). A total of 13% fines were obtained in the CTMP composition (13F). A CTMP pulp without fines (0F) was obtained by washing the retained pulp that did not pass through the DDJ

four times. A pulp with a low fine content of 5% (5F) was prepared by washing the pulp once. The filtrate containing the fines was sedimented for 12 h and the supernatant water was removed. The fines concentration in the sediment suspension was calculated, and two samples of CTMP enriched with fines were prepared by adding additional fines, resulting in final fine content of 20% (20F) and 35% (35F).

CTMP chemical composition

The chemical composition of the delignified samples and the fines fraction was evaluated in terms of cellulose, hemicellulose, pectin, extractives, ashes, soluble lignin and insoluble lignin. Ash content was determined by calcination at 525 °C according to TAPPI T211. Extractive contents in acetone were quantified using Soxhlet extraction following TAPPI T204. To determine the remaining composition, NREL/TP-510-42618 standard was followed. Samples of 300 mg CTMP without extractives were hydrolyzed with 3 mL of H₂SO₄ (72 wt%) for 1 h at 30 °C. After hydrolysis, 84 g of deionized water was added, and the mixture was autoclaved at 121 °C for 1 h. The samples were then vacuum filtered, with the insoluble lignin or Klason lignin retained on the filters. The soluble lignin fraction was quantified by measuring the filtrate absorbance using a UV–Visible spectrophotometer at 240 nm (AquaMate 8100, Thermoscientific, Waltham, MA, USA), according to the standard. Hemicellulose, cellulose and pectin content were analyzed from the filtrate by HPLC (Jasco LC-4000 Series, Jasco, Spain). Before analysis, the filtrate was neutralized with CaCO₃, and filtered through 0.2 µm filters. All samples were quantified in triplicate.

Handsheets preparation

First, the delignified CTMP samples, or those with varying proportions of fines, were soaked in hot water for 1 h to soften and promote fiber swelling. The pulp was then heated to 85 °C and disintegrated at 2% solids using a pulp disintegrator at 30,000 revolutions (PTI, Austria), following ISO 5263–3. Handsheets of 100 g/m² were prepared using a Rapid Köthen sheet former (PTI, Austria) according to ISO 5269-2. The dried sheets (>96% dry content) were dried in the drying plates of the Rapid Köthen until they reached a constant weight. The wet sheets were dried to a dry content of 65–70 wt% and then sealed in plastic bags to maintain their moisture level and stored at 4 °C before pressing.

Hot-pressing technology

Hot-pressing was performed using automatic planar pressing equipment (Negro et al. 2023). The upper pillar rack is attached to a hydraulic MTS™, which requires MTS™ RPC software to control the compression loads, creating block-programmed load versus time sequences up to 100 kN. The pressing time was set to 3 s, and the pressure was established at 3.5 MPa. The experiments were carried out at two temperatures, 180 and 260 °C, with a moisture content of 30–35% (moist) and 7% (named as dry). The pressure release lasted 0.1 s, followed by a 6 s after hold at 0.1 MPa. The sheets were manually handled during the hot-pressing trials.

Mechanical properties characterization

Sheets after hot-pressing were characterized following conditioning at 23 °C and 50% moisture for at least 24 h (ISO 187). Density was determined by measuring weight to calculate grammage and thickness, using ISO 534 and 536. Tensile strength, tensile stiffness, tensile energy absorption and elongation in dry state were measured according to ISO 1924 using an horizontal tensile machine (ABB / Lorentzen & Wettre, Kista, Sweden). Similarly, wet strengths and elongation were measured on the same equipment according to ISO 3781, with samples soaked in water for one minute. This soaking time was considered sufficient for conducting the wet tests, as previous studies showed that hot-pressed CTMP sheets, after soaking for either one hour or one day, experienced a tensile reduction of approximately 4% and 13%, respectively, in contrast to the wet tensile index after one minute (Joelsson et al. 2020). Figure 1 summarizes the four measurements carried out in the horizontal tensile machine test. Some of these measurements are standardized by dividing by the grammage: (i) Tensile Index, which measures the tensile strength (the maximum load a test specimen can withstand before the rupture per unit width) divided by the grammage; (ii) Tensile Stiffness Index (TSI), which evaluates the stiffness or ability of a test specimen to resist deformation when an external force is applied, allowing it to regain its original shape (elastic region). It is the slope that relates the tensile force per unit width to tensile strain, which can also be defined as elastic modulus or Young modulus. To standardize TSI, the result is divided by the grammage; (iii) Strain at break, defined as maximum elongation at rupture measured as the percentage of stretch the test specimen can reach before failure; and (iv) Tensile Energy Absorption (TEA) Index, which evaluates the previous three measures through the area under the curve of tensile force versus elongation until paper rupture, including both the elastic and plastic regions, and is divided by the grammage.

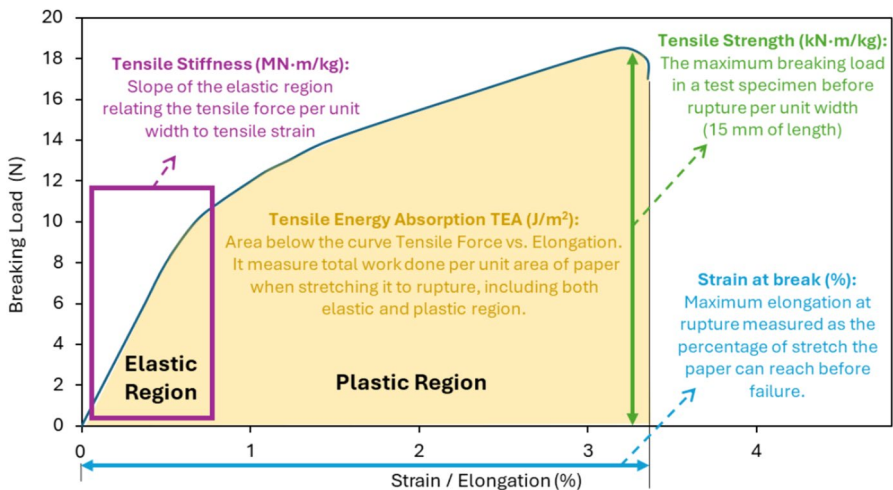


Fig. 1 Example of a tensile machine test that relates breaking load and paper elongation— description of parameters evaluated

The short-span compression test (SCT) was conducted following ISO 9895. Air permeability was determined using the Bendtsen method according to ISO 5636. Both devices are also from ABB / Lorentzen & Wettre. All mechanical tests were carried out at least ten times, and error bars were calculated using the standard deviation. Additionally, the statistical significance of the results was determined using the Multiple Range Test (MRT) with the Least Significant Difference (LSD) and 95% of confidence, utilizing Statgraphics Centurion 19 software with the analysis of variance (ANOVA) tool. Due to the extensive amount of overlapping information, only those samples differing by a single variable will be compared, as detailed in the caption of each figure with MRT.

Results

Chemical composition after CTMP modification

First, the chemical composition of CTMP after the delignification process with sodium chlorite and acetic acid is shown in Fig. 2a. In all cases, the reaction yield exceeds 70%. A discrete amount of total lignin present in the reference CTMP is

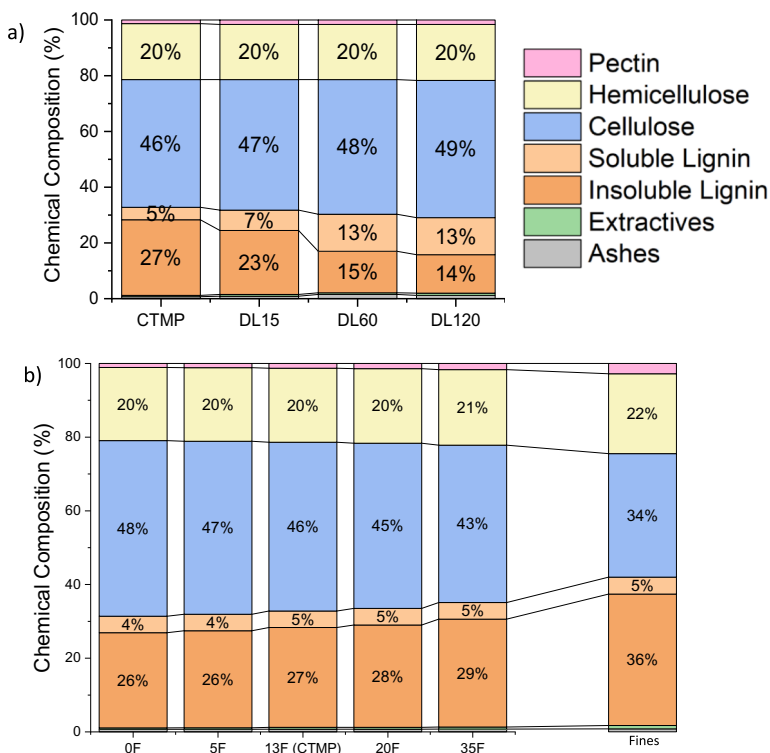


Fig. 2 Chemical composition: **a** After CTMP delignification; **b** With added or removed fines from reference CTMP

removed as the delignification process becomes more severe, decreasing from 31.6 to 27.1% in DL120. When analyzing insoluble and soluble lignin separately, part of the insoluble lignin is converted into acid soluble lignin during delignification. The insoluble lignin content decreases from 27.1% in the CTMP to 13.8% in DL120. Consequently, an increase in soluble lignin is observed, rising from 4.5% in the reference CTMP to 13.3% after delignification (DL120). Regarding the cellulose and hemicellulose content, the results of around 45 wt% and 20 wt%, respectively, are consistent with those reported by other authors for spruce CTMP and for CTMP from other species as Aspen (Wu 2021; Sundberg and Holmbom 2004). The cellulose content slightly increases as delignification becomes more severe, while hemicellulose content remains almost unchanged.

Concerning the variation in fines content in CTMP pulp, Fig. 2b presents the chemical composition of pulps with different fines levels and the fines themselves. Fines contain 40.3% total lignin, making it the predominant compound. This value aligns with Sundberg and Holmbom (2004), who analyzed different CTMP fines fractions (fibrils, flakes and ray cells) with a similar lignin content and lower cellulose content (33.5%) than the raw material. The soluble lignin and hemicellulose content in fines is comparable to that of raw CTMP pulp, meaning that adding or removing fines primarily affects insoluble lignin and the cellulose content. Removing all fines results in a pulp composition of 47.6% cellulose and 25.8% insoluble lignin, whereas increasing fines content to 35% leads to 42.7% cellulose and 29.3% insoluble lignin. Unlike delignification, where soluble lignin increased significantly, varying fines content maintains a stable soluble lignin level (4.5%). Additionally, the extra lignin from fines added in suspension may remain more on the surface rather than acting as a fiber binder.

Tuning chemical composition by delignification of CTMP

Next, the effect of delignification on mechanical properties was evaluated through tensile tests on handsheets subjected to hot-pressing under different conditions. Figure 3 shows these mechanical properties in dry state handsheets. Figure 4 shows the same properties after wetting the samples in water for one minute. Two pressing temperatures were evaluated, along with two initial sheet moisture levels prior hot-pressing, dry (7 wt% moisture) and moist (30–35 wt% moisture).

Regarding tensile strength, not hot-pressed samples (NHP) show that the delignification process increases the Tensile Strength Index (normalized by grammage) by 103% in DL60 (Fig. 3a). Extending the delignification time to 120 min (DL120) and maintaining the chemical dosage to prevent excessive yield loss does not provide further improvements beyond the 60 min treatment. Wang et al. (2018) conducted CTMP delignification at 3% pulp concentration and observed that reducing insoluble lignin content below 13% resulted in a 10% increase in tensile strength. Further lignin removal to 0% under more severe delignification conditions than those used in this study led to a tensile strength increase up to 75%. In our study, at 6 wt% pulp concentration, we found that with 15% of insoluble lignin (DL60), the tensile strength (normalized by thickness, in MPa) increased by over 175%. Thus, complete lignin

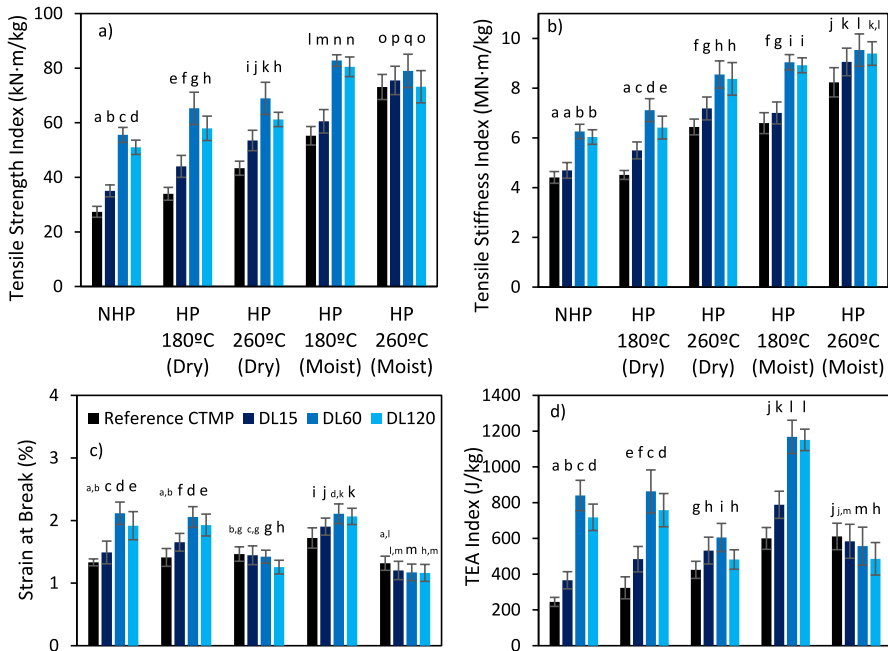


Fig. 3 Dry mechanical properties of CTMP samples after delignification: **a** Tensile Strength Index; **b** Tensile Stiffness Index; **c** Strain at break; and **d** TEA Index. In each Figure, values with the same letter are not significantly different according to the multiple range test (comparison applies only to samples with the same hot-pressing condition or the same pulp)

removal is unnecessary as it would negatively impact pulp yield and lead to increased waste generation.

Besides, the effect of hot-pressing on delignified samples was also studied. At both temperatures (180 and 260 °C), moist-pressed sheets exhibited higher tensile indexes than those pressed without moisture. Previous studies have also shown that a moisture content of around 38% in hot-pressed sheets results in better tensile strength and stiffness compared to fully dried sheets (Mattsson et al. 2021). This is because the softening temperature of disordered cellulose, hemicellulose and lignin decreases with increasing moisture content (Back and Salmén 1982; Joelsson 2021). Additionally, moist lignin becomes tacky with increased temperatures, enhancing the bond strength between lignin and fibers in close contact (Norgren et al. 2018). Comparing DL60 moist handsheets at both temperatures, 180 °C and 260 °C, we observe a tensile strength index increase of 49% and 42% in relation to DL60 NHP, and an increase of 202 and 188% with respect to reference CTMP NHP, respectively. In no case does DL120 outperform DL60 in strength, ruling out the need for more severe delignification. This suggests that the separation of lignin from the cellulose and hemicellulose matrix during delignification facilitates lignin softening during hot-pressing, increasing its fluidity and promoting better distribution around fiber surfaces. Nevertheless, excessive delignification degrades the pulp structure, worsening its mechanical properties. Moreover, when comparing the hot-pressed samples at

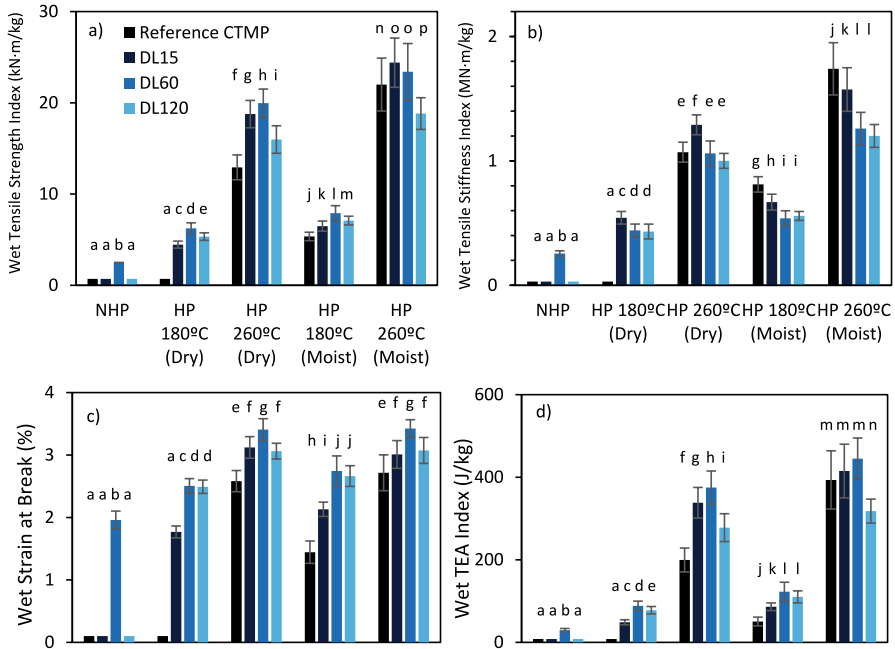


Fig. 4 Wet mechanical properties of CTMP samples after delignification: **a** Tensile Strength index, **b** Tensile Stiffness Index, **c** Strain at break, and **d** TEA Index. In each Figure, values with the same letter are significantly equal according to the multiple range test (only comparing samples with the same hot-pressing condition or the same pulp)

different temperatures but at the same moisture level, DL60 samples exhibit similar tensile strength results, whereas reference CTMP show variations with temperature.

As for TSI, Fig. 3b indicates that not only moisture content of the sheets but also temperature contributes to greater sheet stiffness, in contrast to tensile strength. A higher temperature (260 °C) results in increased CTMP rigidity, as reported by Norgren et al. (2018). Similar to the tensile strength index, the best TSI results were obtained for DL60 using moist sheets and a hot-pressing temperature of 260 °C, reaching 9.5 MN m/kg, an even better result than kraft pulp with 25 mg/g of starch as a retention aid hot-pressed at 200 °C (Norgren et al. 2018). This TSI represents a 15% increase regarding reference CTMP under the same hot-press conditions, and when combining the effects of hot-pressing and delignification, the increase reaches 115%. Thus, a certain amount of lignin acts as a natural glue that binds cellulosic fibers, providing both stiffness and strength (Mili et al. 2022).

In terms of strain at break (Fig. 3c), elongation to failure remains limited in all cases, never exceeding 2.1%. These results align with those obtained in other CTMP tests (Norgren et al. 2018; Pasquier et al. 2023) and are significantly lower than those of bleached kraft pulps, which exhibit higher elasticity and plasticity. Alternative approaches such as refining or the addition of bacterial nanocellulose, have been shown to improve tensile strength (Campano et al. 2018; Kouko et al. 2019). The presence of lignin in the pulp restricts elongation, particularly at high temperatures (260 °C), where strain at break does not exceed 1.5%, regardless of sheet moisture

content. As a result, CTMPs are not suitable for applications requiring plasticity, especially after hot-pressing at 260 °C. Regarding delignification, DL60 shows a slight improvement in elongation versus other pulps, and this effect is maintained when hot-pressed at 180 °C.

Finally, Fig. 3d shows the TEA index, which combines elongation at break and tensile strength into a sigule parameter, with the plastic region prevailing over the elastic, the latter already measured in stiffness. As noted in the low strain at break of hot-pressed samples at 260 °C, the plasticity of these samples is reduced compared to the sheets pressed at 180 °C and NHP, with the highest TEA index in moist sheets pressed at 180 °C. In the elastic region, the high stiffness was already evident from the steep slope between strength and strain, indicating limited strain at break. Therefore, despite the tensile strength of the sheets, they remain rigid with restricted elongation. DL60 shows the highest TEA index except at 260 °C in moist sheets, where non-delignified sheets achieve the best results due to their higher strain at break, which influences the TEA index. In comparison with other CTMP modifications, TEA index of DL60, whether NHP or hot-press under any condition, outperforms obtained by adding a mixture of anionic and cationic microfibrils or anionic and cationic starches using a wet-press (Ankerfors et al. 2016).

Figure 4 shows the wet tensile properties of the delignified samples. It is observed that hot-press temperature plays a crucial role in achieving waterproof papers with the highest temperature tested (260 °C being essential for optimizing all four parameters evaluated. This occurs because lignin softens under these conditions, behaving like a plastic material, increasing its fluidity, improving lignin penetration, ensuring better distribution on fiber surfaces, and forming stronger inter-fiber bonds (Li et al. 2015; Wang et al. 2024).

Figure 4a shows Tensile Strength Index in wet conditions. Even before hot-pressing, DL60 exhibits slight wet strength, whereas the other delignified materials show no wet strength without hot-pressing. The highest results are observed in moist sheets at 260 °C, with DL15 reaching 24.4 kN m/kg, although DL60 achieves a comparable value (23.4 kN m/kg) according to MRT. This indicates that severe delignification process is unnecessary, as 15% of insoluble lignin is sufficient. The wet tensile strength of DL15 represents approximately 32% of its dry tensile index, making these papers highly water-resistant (Lindström et al. 2005), with DL60 showing a similar ratio. These wet strength results align with findings by Joelsson (2021), who studied pulps hot-pressed up to 200 °C and found the highest wet tensile index in pulps containing 12% insoluble lignin, compared to samples with 7 and 26%, respectively.

With regard to rigidity, elongation and TEA (Figs. 4b, c and d), the most favorable outcomes are observed at 260 °C as the softening of the lignin facilitates water penetration. In contrast, NHP or hot-pressing at 180 °C causes the samples to break apart without any or with minimal tensile strength. In the moist sheets at 260 °C, which perform best except in elongation with similar strain at break in moist and dry sheets at 260 °C, CTMP delignification results in a lower TSI, as the delignified pulps became more elastic and less rigid with decreasing lignin content. Both elongation and the TEA index show the best wet results in DL60, indicating greater plastic deformation.

To compare the differences in wet and dry resistance of the best samples (DL60 and reference), Fig. 5 presents the tensile strength index vs strain at break in a single graph. Each color represents two results, with the start of the arrow indicating dry results and the end of the arrow representing wet strength results. Additionally, the density of sheets prepared under different conditions is shown in the graph. An increase in sheet densification is observed with hot-pressing. Pressing, drying, and dewatering contribute to fiber–fiber bond development as water is removed, increasing the contact area between fibers and enhancing higher bond strength, resulting in improved strength (Joelsson 2021). These densification results align with Pasquier et al. (2023), where temperature and pressure significantly increased CTMP density up to 1200 kg/m^3 when 100 MPa and 200°C were applied, whereas in our case, the applied pressure is 3.5 MPa.

Comparing dry and wet strength, the effect of lignin at high temperature (260°C) is particularly evident in wet conditions, where it increases resistance and elasticity. Regarding the latter, strain at break when wetted in water exhibits greater elongation than in the dry state, reaching up to 3.4%, though still lower than pulps with less lignin content, which are more elastic. On the contrary, for dry properties, the hot-pressing effect in the 180 to 260°C range is counterproductive, reducing elasticity (which was maintained transitioning from NHP sheets to hot-pressing them at 180°C) although slightly decreasing tensile strength and density. Thus, moderate delignification (DL60) sufficiently enhances CTMP mechanical properties and, at the same time, maintains high pulp yield. Further investigation is needed into hot-pressing temperatures between 180 and 260°C in DL60 sheets to optimize both dry and wet resistance simultaneously.

Other complementary properties evaluated in Fig. 6 include air permeability measured according to the Bendtsen method, density, and compressive strength measured by the SCT test. Air permeability (Fig. 6a) decreases with hot-pressing. Moderate delignification (DL60) yields the highest results under all hot-press conditions, reducing permeability by more than four times with respect to the moist reference CTMP sheets. In terms of moisture, for delignified moist sheets with 30–35% moisture content, those pressed at 260°C exhibit similar permeability with no significant differences according to MRT. In dry sheets, higher temperatures further decrease

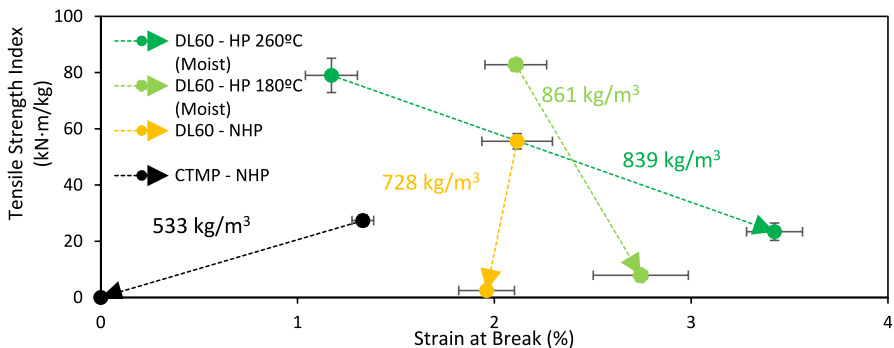


Fig. 5 Tensile strength Index vs. Strain at break in dry conditions (arrow start) and wet conditions (arrow end). Density is shown as numerical data

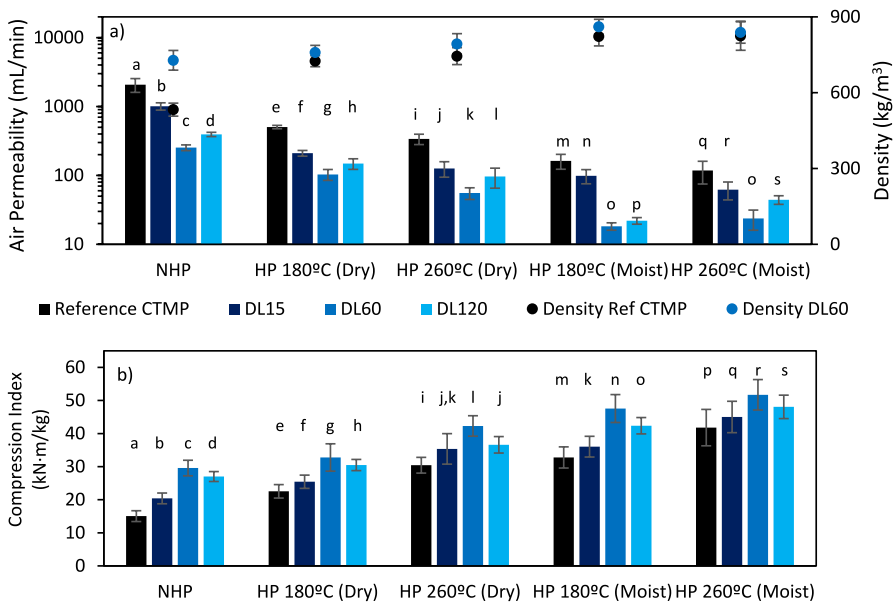


Fig. 6 Additional mechanical and physical properties: **a** Air permeability, and **b** Compression Index. In each Figure, values with the same letter are significantly equal according to the multiple range test (only comparing samples with the same hot-pressing condition or the same pulp)

permeability, as also observed by Pasquier et al. (2023) with 12% moisture content. On the other hand, while delignification increases the densification of NHP samples, the reference CTMP pulp shows increased density with the hot-pressing. However, no synergistic effect between hot-pressing and delignification is observed, as similar results are obtained for reference CTMP and DL60.

Finally, the compression index is shown in Fig. 6b, following the same trend as the dry tensile strength index, with the highest values observed in moist sheets hot-pressed at 260 °C. The delignification of DL60, with 14.7% of Klason lignin, doubles the SCT index of the reference CTMP without hot-pressing, reaching 29.6 kN/m/kg. Comparing DL60 samples with NHP state with those under the best hot-pressing condition, a 75% increase is observed, reaching 51.7 kN/m/kg. Thus, for compression strength, moderate delignification is also sufficient to enable lignin softening during hot-pressing, enhancing this property.

Tuning chemical composition by modifying the content of fines

The effect of fines content in the pulp is evaluated under different hot-pressing conditions. Figure 7 shows the dry tensile properties at two pressing temperatures and two sheet moisture levels.

Figure 7a shows the tensile strength index for different fines contents. Before hot-pressing (NHP samples), an increase in tensile strength index up to 32% is observed as fines are incorporated into the pulp. This is consistent with literature indicating that tensile strength increases with fines content up to more than 50% in the pulp (Odabas

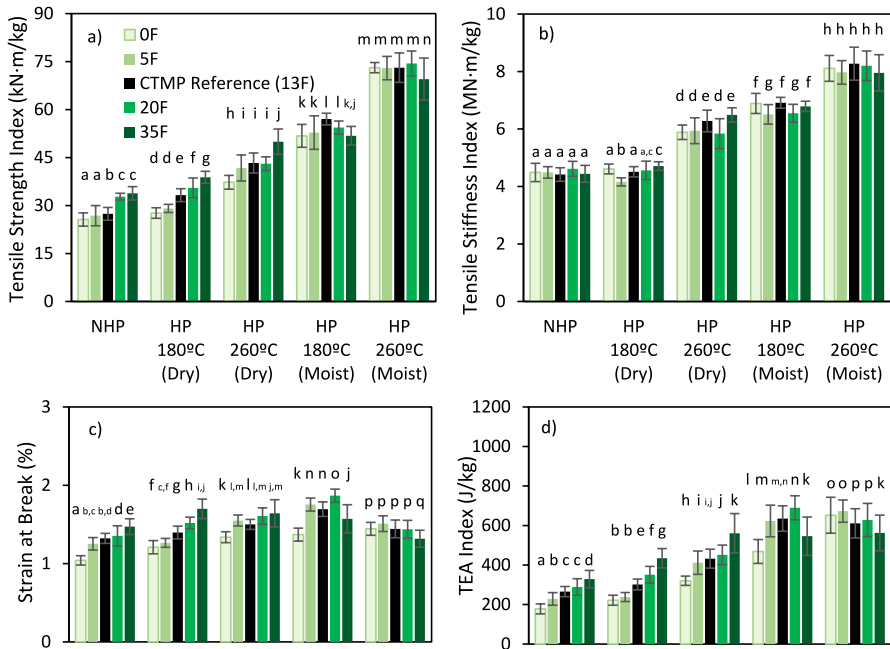


Fig. 7 Dry mechanical properties of CTMP samples with different fines content: **a** Tensile Strength Index, **b** Tensile Stiffness Index, **c** Strain at break, and **d** TEA Index. In each Figure, values with the same letter are significantly equal according to the multiple range test (only comparing samples with the same hot-pressing condition or the same pulp)

et al. 2016), as well as finding that that pulp washing to remove fines reduces tensile strength and increases bulk (Björk 2020). Regarding the effect of hot-pressing at different conditions, it promotes densification and lignin softening, particularly at the highest temperature. At both temperatures, dry-pressed sheets perform better results than NHP samples with the same fines content, with this improvement being more pronounced at 260 °C. Additionally, a linear increase in tensile strength is observed in dry hot-pressed sheets as fines content increases, with strength improving up to 40% (35F) in relation to sheets without fines. The improved tensile strength may be attributed to the higher lignin content in the fines, which softens during hot-pressing. For moist sheets, tensile strength is higher than in dry hot-pressed sheets, especially at 260 °C, similar to the effect observed with delignification. However, fines content is not a determining factor in the tensile index, as results remain similar up to 20% fines, with only a slight (~5%) when reduction at 35% fines. This may be because moisture in the pulp helps reduce the softening temperature of its components, facilitating their dispersion. The available moisture in the moist sheets (30–35%) appears sufficient to reach the softening temperature of cellulose, hemicellulose, and lignin, minimizing its impact on tensile strength (Joelsson 2021; Back and Salmén 1982). Moreover, since the sheets already reached their highest tensile values, the slight reduction at high fines content may be due to the artificial addition of fines, leading to more external deposition rather than full integration into the sheet structure. Comparing sheets with fines with delignified pulps, the latter yields higher values, with

DL60 reaching a tensile strength index above 80 kN m/kg in, making delignification the more effective alternative for enhancing dry tensile index.

The tensile stiffness index (Fig. 7b) shows that not only the increase in temperature but also the moisture content in the handsheets affects the improvements in rigidity. This was demonstrated by Mattsson et al. (2021), who indicated that sheets with a moisture content of around 38% produced the highest stiffness values in thermomechanical pulp. In this study, a pressing temperature of 260 °C and a moisture content of 30–35% produced the highest rigidity of all samples, with an increase of around 80%. Nonetheless, the presence of a higher or lower content of fines results in very small variations in TSI. Concerning delignification, a certain intermediate amount of lignin (around 15%, DL60) produces a good distribution of lignin, acting as a natural glue around the fibrillar structure, providing stiffness. The addition or removal of fines without delignification, leads to much higher lignin levels (above 26% in all cases), resulting in tensile stiffness in the dry state similar to the unligified samples and far from the delignified samples.

In relation to strain at break (Fig. 7c), a slight increase is observed with fines content. This agrees with the literature, which indicates that fine fibers lead to greater strain at break than coarse fibers, as well as better wet-web and dry tensile strength (Seth and Kingsland 1990; Karlsson 2007). This trend is only altered in samples hot-pressed under wet conditions with a high fines content. From 13 to 20% fines, elongation at failure slightly decreases, as was also observed with tensile strength. It should be noted that in this type of pulp, the strain at break is low, with values ranging from 1.2 to 1.8% in all hot-pressed sheets. These values are well below the dry strain at break of the best delignified DL60 samples and significantly lower than those of bleached kraft pulps. Regarding TEA index (Fig. 7d), this parameter shows the same trend as tensile strength in the three variables studied: temperature, moisture, and fines content. In short, the pulps with a higher content of fine particles, which increase the lignin content in the sheets, exhibit less satisfactory results in dry tensile strength than those obtained after delignifying the pulps to 15% insoluble lignin, which is the lignin proportion that yields the most satisfactory results in dry condition.

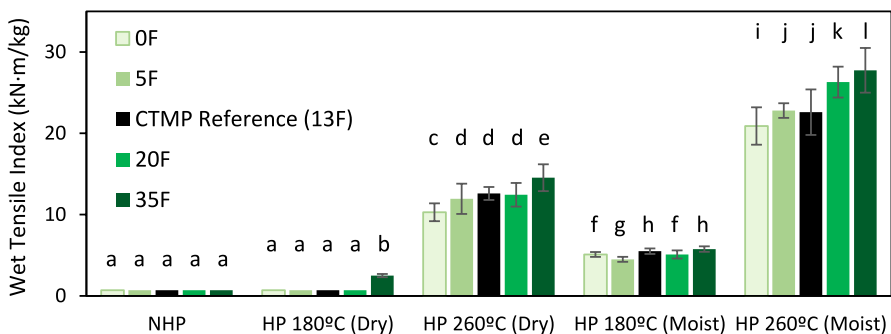


Fig. 8 Wet Tensile Strength Index of CTMP samples with different fines content. Values with the same letter are significantly equal according to the multiple range test (only comparing samples with the same hot-pressing condition or the same fine content)

Nevertheless, analyzing the wet tensile strength index (Fig. 8), the presence of a significant lignin content produced by the addition of fines is favorable, especially in pulps hot-pressed at 260 °C, the temperature at which the softening of the lignin and the other pulp compounds occurs. As with the delignification of the pulp, moist sheets hot-pressed under these conditions achieve better results with more resistant papers. In these conditions, sheets with a fines content of 35% in the pulp composition present a wet tensile index of 28 kN m/kg, which is an increase of about 20% compared to the reference pulp with 13% fines, hot-pressed under the same conditions, and represents 40% of the dry tensile strength index. This is a higher figure than that achieved by delignified pulps, which only reached 24 kN m/kg with an insoluble lignin content of 15%, and worse results were observed as the amount of insoluble lignin in the pulp increased. This demonstrates that not only is the amount of insoluble lignin in the pulp important for improving wet strength, but also the way it is distributed around the fibers. It is necessary either to chemically treat the lignin to remove part of it from around the fibers and facilitate its distribution through hot-pressing of the sheets, or to incorporate lignin-rich fines, which, being smaller than 200 mesh, facilitate distribution around the fibers. These fines then melt and distribute around the fiber bundles during pressing at 260 °C.

On the other hand, the other wet mechanical properties were evaluated only in sheets hot-pressed in moist conditions at 180 and 260 °C (Fig. 9), as sheets that were not hot-pressed exhibited no wet strength. Stiffness does not show significant variations with the amount of fines, but strain at break increases with both the temperature in the press and the amount of fines, similar to the trend observed in dry conditions. This elongation at failure does not exceed the results obtained through delignification, as seen with wet tensile strength. Lastly, the TEA index, which combines tensile strength and elongation at failure into the same parameter, shows the same trend as strain and strength, increasing with the amount of fines and reaching 543 J/kg in wet conditions, an increase of 22% over the best sheets produced with delignified pulp.

Finally, other mechanical and physical properties were measured in Fig. 10. Air permeability shows a decrease with hot-pressing in moist conditions, independent of the temperature, as well as with the amount of fines. The presence of smaller particles

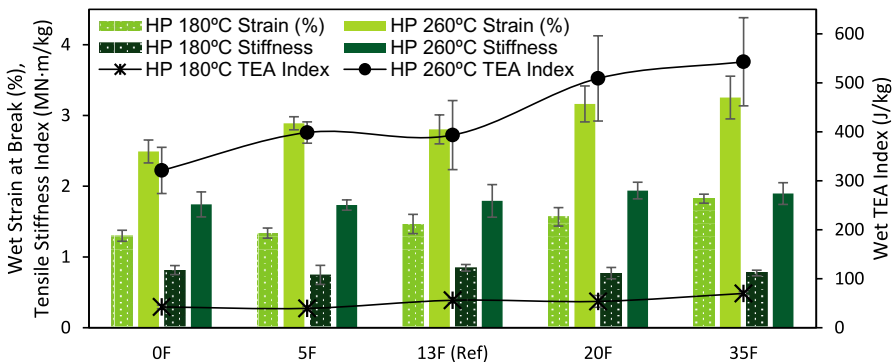


Fig. 9 Other wet mechanical properties of CTMP samples hot-pressed at 180 and 260°C under wet conditions: Wet strain at break (light green bars), wet tensile stiffness index (dark green bars), and TEA index (black dots)

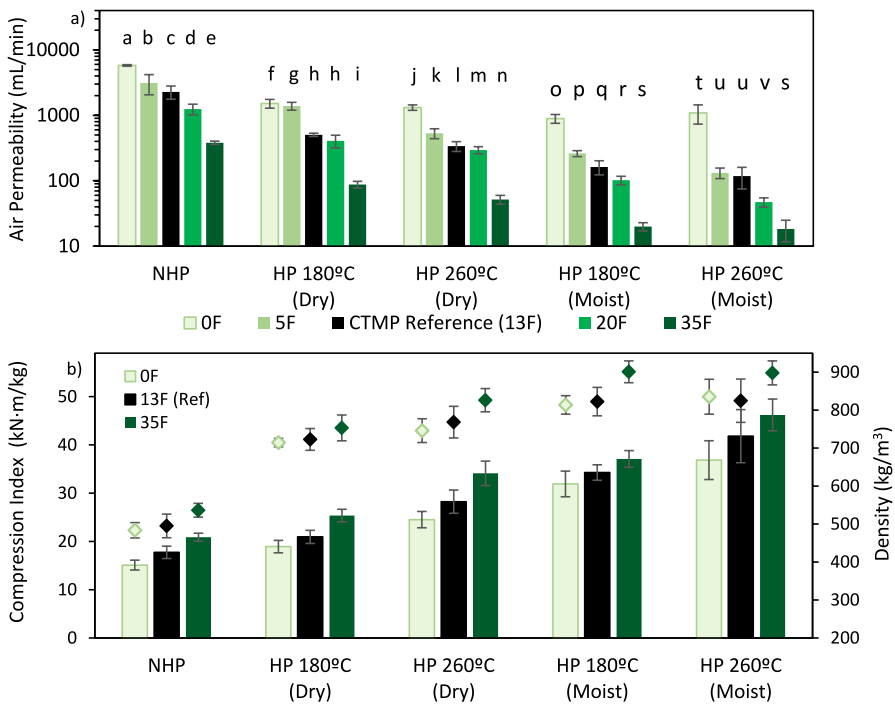


Fig. 10 Other mechanical and physical properties: **a** Air permeability, and **b** compression Index (bars), and density (dots)

blocks more pores in the sheets, reducing this property and favoring compactness (Rasi 2013). In sheets hot-pressed in moist conditions, the use of 35% fines in the pulp reduces air permeability by about 50 times compared to sheets without fines. In contrast to the reference pulp, the reduction is one order of magnitude with the reference pulp under the same hot-pressing conditions and two orders of magnitude in relation to NHP reference pulp. Both strategies, delignification and addition of fines, show similar reductions in air permeability.

On the other hand, sheet compaction due to the effect of fines can be observed in the density, which is also affected by the hot-pressing conditions. The density of sheets hot-pressed using moist sheets, independent of temperature (180 or 260 °C), increases to 900 kg/m³ in sheets with 35% fines, which represents a 9% increase relative to reference CTMP hot-pressed sheets. These densities are higher than those obtained after delignification and hot-pressing, making the strategy of incorporating fines in the pulp the most effective for improving sheet densification.

Regarding compression strength, SCT shows an increase using the hot-press, high temperature, moist conditions, and fines content. However, the maximum compression index does not exceed the results obtained after delignification, which exceed 51 kN m/kg, whereas the maximum compression index reached with fines is 46 kN m/kg.

Conclusion

This research highlights the balance between lignin content and processing conditions using hot-press technology, demonstrating a synergistic effect. By either applying moderate delignification or increasing the fines content, the performance of CTMP handsheets can be optimized for various applications, resulting in stronger and more water-resistant paper products. Moisture content plays a crucial role, with a moisture content of 30–35% enhancing the mechanical properties by facilitating the softening of lignin and other pulp compounds. Each of the evaluated treatments demonstrates optimal conditions depending on the mechanical or physical properties targeted for maximization. Among the key findings, the best dry tensile strength and stiffness are achieved with a soft delignification up to 15% insoluble lignin (DL60) at both 180 °C and 260 °C, particularly at the latter temperature, where an improvement in both wet properties is also observed. These conditions are also ideal for optimizing the SCT index.

To maximize wet strength and wet stiffness, the most effective approach is the use of 35% fines, hot-pressed at 260 °C, which also results in the highest sheet densification. Still, attention must be given to the dry strain at break as it decreases considerably when hot-pressing is applied at 260 °C compared to 180 °C, regardless of delignification or fines addition. Notable reductions in air permeability are observed with both partial delignification and increased fines content, in similar proportions. These findings provide valuable insights for advancing the industrial production of CTMP-based materials, paving the way for more durable, efficient paper products suited for packaging and structural applications.

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Data availability Data is provided within the manuscript.

Declarations

Conflict of interest The authors declare that they do not have conflict of interest.

Ethical approval The authors declare that principles of ethical and professional conduct have been followed.

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