DE GRUYTER

Nord. Pulp Paper Res. J. 2026; aop

Paper Technology

Björn Sjöstrand*, Sandra Mellroth and Gunnar Henriksson

Paper strength correlates with hornification for kraft pulps dried at different temperatures

https://doi.org/10.1515/npprj-2025-0073
Received October 30, 2025; accepted January 9, 2026;

Abstract: This study examines the impact of hornification on the strength of paper made from chemical pulps. Two key aspects are examined: how varying degrees of hornification influence the mechanical properties of paper sheets, and how temperature affects hornification in papermaking pulps when water remains within the fibre structure. Experiments were conducted on hardwood and softwood kraft pulps that were dried and heated under controlled conditions. The results revealed that hornification reduces the strength of paper made from three different types of pulp. A local minimum of hornification and local maxima of tensile strength occurred around 40–60 °C. A strong linear correlation was observed between decreased tensile strength, decreased water retention, and the hornification ratio. Temperature treatments applied without water removal did not affect the water retention value (WRV) or the strength of the paper. This confirms that water removal is essential for hornification and strength loss to occur. These findings refine our understanding of hornification, suggesting that careful process control during drying can exploit the positive effects of moderate drying while minimizing hornification, and thus, the risk of excessive fibre closure. Such control strategies could improve strength optimization in industrial pulp and paper manufacturing.

Keywords: hornification; mechanical strength; water removal; kraft pulp; cellulose

1 Introduction

Paper is one of the most significant innovations in human history. It is a material that has shaped history by enabling the spread of knowledge, culture and trade (Shenoy and Aithal 2016). However, the role of paper as both an information carrier and a packaging material has recently come under increasing challenge (Olsson and Salmén 2001; Shenoy and Aithal 2016). Therefore, to support the continued development of this versatile material, a deeper understanding of paper's fundamental structure, performance, and behaviour in humid environments is required (Belle and Odermatt 2016). A key factor in the mechanical properties of paper lies in its basic building blocks: individual lignocellulosic fibres and their interactions within the fibre network. The structure, surface chemistry and bonding properties of these fibres directly influence the material's strength (Asta et al. 2024; Hirn and Schennach 2015, 2017; Wågberg 2022).

Strong paper formation involves several key processes. First, the fibres must come into contact with each other during the drying process, in which capillary forces are believed to play a significant role (Hirn and Schennach 2015, 2017). These strong capillary forces rely on hydrogen bonds forming networks of water molecules (Przybysz et al. 2016), which may also interact with hydroxyl groups on the pulp fibres (Sjöstrand et al. 2023). Additionally, ionic interactions, such as salt bridges and ion–dipole interactions, may contribute to fibre bonding as they can act over relatively long distances, potentially 'pulling' fibres closer together (Dodson 1970; Hirn and Schennach 2015, 2017; Zhao and Kwon 2011). The actual fibre-to-fibre bonds depend on the morphology and rigidity of the fibres, which affect the contact area, as well as the nature of the bonds formed at the joints (e.g. hydrogen bonds, London dispersion forces, ionic interactions and aromatic interactions).

The significance of these bonds remains under debate. In particular, scientists are divided on the role of hydrogen bonds: while some regard them as essential, others question their influence (Hirn and Schennach 2015, 2017; Wågberg 2022). Paper typically exhibits lower strength under wet or

---

***Corresponding author: Björn Sjöstrand**, Department of Engineering and Chemical Sciences, Karlstad University, SE-651 88, Karlstad, Sweden, E-mail: bjorn.sjostrand@kau.se. https://orcid.org/0000-0003-4023-594X
Sandra Mellroth and Gunnar Henriksson, Department of Engineering and Chemical Sciences, Karlstad University, SE-651 88, Karlstad, Sweden. https://orcid.org/0000-0001-8817-2031 (G. Henriksson)

humid conditions (Belle and Odermatt 2016), which suggests that water may disrupt critical interactions between fibres. Various dry and wet strength resins are employed to enhance bond strength, with recent developments focusing on adhesive and cohesive failure mechanisms (Lindström et al. 2005). These resins may, of course, contribute other types of interaction to those of the fibres themselves. For example, when pure fibres are binding each other, other non-covalent interactions may be involved than when strength resins are present. Electrostatic interactions during sheet formation influence fibre–fibre bonding and overall paper strength (Torgnysdotter and Wågberg 2004), partly due to the long-distance effect of electrostatic interactions. Conversely, even uncharged wet-end additives can enhance paper strength (Christiernin et al. 2003). Fibre entanglement and mechanical friction also contribute to wet web strength, particularly above the fibre saturation point (de Oliveira et al. 2008). A deeper understanding of molecular interactions is essential for developing stronger paper products and optimising manufacturing processes.

One phenomenon that significantly impacts the properties of pulp fibres, and consequently the mechanical properties of paper, is hornification. When pulps are dried – which is common when pulping and papermaking occur in different locations – the fibres harden and stiffen, and the pores in the fibre wall close. This can lead to fibre collapse, resulting in lower chemical reactivity, reduced fibre–fibre interactions and decreased recyclability due to strength loss over multiple drying cycles (Fernandes Diniz et al. 2004; Hashemzehi and Sjöstrand 2024; Kato and Cameron 1999; Laivins and Scallan 1993; Mo et al. 2022; Newman 2004; Salmén and Stevanic 2018; Sjöstrand et al. 2023; Sjöstrand et al. 2024; Tze and Gardner 2001; von Schreeb et al. 2025; Wohlert et al. 2022).

Hornification occurs when chemical bonds form between the surfaces of cellulose during dewatering and drying. This prevents the material from swelling back to its original structure when it comes into contact with water. While it is often claimed to be an irreversible process, it is not known with certainty whether it is completely or partially irreversible. In papermaking, hornification results in reduced fibre wall swelling, decreased internal and external fibrillation, and lower fibre flexibility in dried pulps compared to undried pulp. These changes hinder the formation of effective fibre networks comprising numerous fibre-to-fibre bonds, ultimately resulting in reduced paper strength (Ferreira et al. 2023; Laivins and Scallan 1993; Oksanen et al. 1997). Conversely, hornification may increase fibre stiffness, and potentially fibre strength, under certain conditions (Çiçekler and Tutuş 2025).

Measuring and quantifying hornification in pulp is challenging because the phenomenon affects several properties, including decreased water adsorption, fibre stiffening and reduced fibre-fibre interaction. This leads to diminished paper strength. In this study, we have chosen to use the decrease in water interaction, as measured by the water retention value (WRV), as the standard method, defining a corresponding ‘hornification ratio’ (HR) based on this metric (Moser and Sjöstrand 2025). WRV was selected due to its robustness and relative simplicity, despite only capturing one aspect of hornification. In this study, we examine the impact of hornification on sheet strength by investigating pulps dried at various temperatures, including samples that have undergone heat treatment without drying. We then relate the sheet strength to the hornification ratio. As a result of this experimental setup, we present the first systematic demonstration of a linear relationship between the WRV-based hornification ratio and the tensile index across various kraft pulps and drying temperatures. We also demonstrate that the previously observed local minimum in hornification at 40–60 °C coincides with a consistent tensile maximum across different pulps. Furthermore, by explicitly decoupling heat treatment from water removal within a single experimental design, we clarify the respective contributions of each factor to strength development.

2 Materials and methods

2.1 Pulps

This study used three never-dried commercial papermaking pulps from Gruvön Mill, Billerud AB, Grums, Sweden: One bleached and one unbleached softwood kraft pulp from a 70/30 mix of Norway spruce (*Picea abies*) and Scots pine (*Pinus sylvestris*), and one bleached hardwood kraft pulp from birch (*Betula pendula*/*Betula pubescens*). The solids content and pH of the three pulps were determined in accordance with ISO 638-1:2022 and ISO 6588-1:2022, respectively. Both the solids content and pH measurements were performed twice. The solids content of all pulps was approximately 4 % at delivery; the pH was 6.5 for bleached softwood, 7.2 for unbleached softwood, and 7.0 for bleached hardwood. The kappa number of the unbleached pulp was 30–31 and the brightness of the bleached pulps was 88 % for hardwood and 89 % for softwood. Typical fibre lengths for these kraft pulps are usually 1.8-2.0 mm for softwood and 0.8-1.0 mm for hardwood, although actual measurements of fiber lengths were not included in the present study.

2.2 Heat treatment

The heat treatment and drying of the pulp were carried out in parallel at temperatures of 23 °C, 40 °C, 60 °C, 80 °C, 100 °C, 120 °C and 140 °C. The pulp was weighed out into two aluminium trays: one was left uncovered, while the other was covered with a lid and several layers of aluminium foil (see Figure 1). Both trays were placed in a drying cabinet overnight for around 18 h. Monitoring of air circulation or humidity in the cabinet was not performed due to previous studies (Sjöstrand et al. 2024) showing consistent drying at temperatures of 40 °C and above. However, the cabinet struggled to maintain consistent drying at lower temperatures. To ensure consistency, the lowest temperature of 23 °C was dried in a controlled climate of 23 °C and 50 % relative humidity, in accordance with ISO 187. All the dried pulps had a solids content of 92–98 %, whereas the heat-treated samples (i.e. those covered with aluminium foil) remained fairly wet, with a solids content ranging between 4 and 20 %. The relatively large range of solids was due to the increasing difficulty of hindering evaporation by covering the trays. Thus, samples with the highest temperatures showed the highest solids content. No correlation was observed between WRV and tensile strength at these solids contents.

2.2.1 Water retention value (WRV)

Hornification was assessed by measuring the water retention value (WRV) in accordance with ISO 23714:2014. Pulp suspensions of 0.2 % solids were prepared in deionised water for each condition (never-dried, dried, and heat-treated pulps). The suspensions were thoroughly dispersed according to ISP 5263-1:2004 and allowed to equilibrate prior to testing. Pads weighing 1700 g/m^2 were formed and dewatered using a 30-min centrifugation at 23 °C with a g-force of 3,000 g. Immediately after centrifugation, the wet mass was recorded. The pads were then oven-dried to constant mass (as prescribed by the standard) to obtain the dry mass. At this level of centrifugal acceleration, the majority of free and loosely bound water is expelled from the fiber network, while a significant fraction of intra-fiber water remains within the cell wall. All measurements were performed four times per condition. Lower WRV values indicated increased hornification, i.e. reduced swelling capacity. For each sample, the hornification ratio (HR), Eq. (1), was calculated by dividing the never-dried WRV by the WRV of the dried materials (Moser and Sjöstrand 2025). In previous work (Moser and Sjöstrand 2025), HR was defined in terms of WRV due to its connection with fibre swelling, accessible pore volume, and the material's bonding capacity. An HR value of 1 corresponds to an undried, non-hornified reference state, while an HR value greater than 1 indicates increased hornification.

$$\text{Hornification Ratio} = \frac{\text{WRV}_{\text{never-dried}}}{\text{WRV}_{\text{dried}}} \quad [1]$$

Figure 1: The drying procedure and heat treatment for pulps: the covered tray hindered water removal during heat treatment, whereas the open tray enabled drying.

2.3 Sheet preparation and tensile testing

Laboratory sheets were formed from both dried and heat-treated pulps, as well as from pulps that had never been dried, in order to evaluate the effects on sheet-level strength. The pulp slurries were disintegrated and diluted to the consistency typically used for preparing handsheets, and then formed into sheets using a standard laboratory sheet former (ISO 5269-2). After formation, the sheets were couched, pressed and dried under controlled conditions identical for all samples within the study. Prior to mechanical testing, all sheets were conditioned at 23 ± 1 °C and 50 ± 2 % relative humidity according to ISO 187 to ensure moisture equilibrium.

Tensile properties were determined using a method in accordance with ISO 1924-3:2008, with a constant rate of elongation of 100 mm/min. Test strips were cut to the dimensions specified in the standard and their width and basis weight were measured to enable the tensile index (kNm/kg) to be reported. Ten test strips were evaluated for each sheet set, and the average value and 95 % confidence intervals were reported. Basis weight was determined according to ISO 536 to enable calculation of the tensile index from the tensile strength, which was performed automatically by the testing software. All measurements from never-dried, dried and heat-treated pulps were performed under identical conditioning and testing settings in order to isolate the effects of drying and thermal history.

3 Results and discussion

Softwood kraft pulps (bleached and unbleached) and hardwood kraft pulp were dried at different temperatures. In parallel, pulps were treated at the same temperature and wrapped in aluminium foil to prevent drying.

3.1 WRV

Figures 2–4 show the results of the WRV measurements for the three pulps, with the reference values for the never-dried pulps indicated by the dashed horizontal lines and the dotted lines showing the 95 % confidence intervals. Heat-treated and dried masses are shown as separate bars for each temperature.

The most significant result from Figures 2–4 is that water removal is crucial for reducing the water retention capacity and hornification of pulps. All heat-treated pulps with solids content of up to 20 % show an unchanged water retention value (WRV) compared with the never-dried references, while all dried samples at corresponding temperatures show a decrease in WRV. Thus, the covalent cross-linking believed to play a role in hornification at higher drying temperatures (Sjöstrand et al. 2024) depends on water removal in line with condensation mechanisms.

The results show that the hardwood and unbleached softwood pulps have a higher water retention value (WRV) for the undried samples than the bleached softwood pulp when treated in the same way. This can be explained by the more abundant surface charges of hardwood fibres, which are due to a higher xylan content. In the unbleached softwood pulp, the charges are due to the remaining lignin (Hubbe et al. 2024). Furthermore, the cells in hardwood are smaller and the hemicellulose content is probably lowest in bleached softwood pulp (Sjöström 1993).

Figure 3 and Figure 4 also show increased hornification for bleached hardwood compared to bleached softwood. The difference between never-dried and once-dried WRV at 23 °C

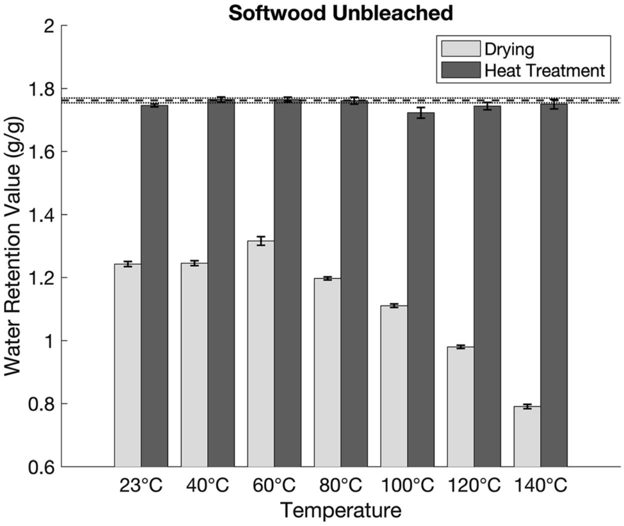

Figure 2: Water retention values for heat treated and dried unbleached softwood pulps in different temperatures, including never dried pulp as reference material indicated by a dashed line. Error bars represent 95 % confidence intervals, based on four repetitions of measurements.

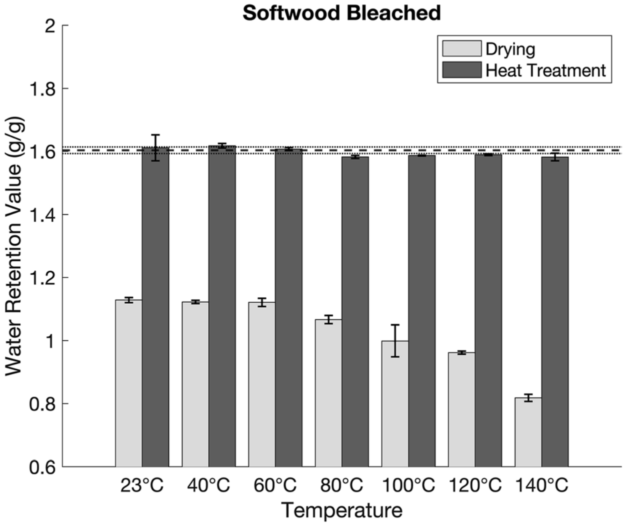

Figure 3: Water retention values for heat treated and dried bleached softwood pulps in different temperatures, including never dried pulp as reference material indicated by a dashed line. Error bars represent 95 % confidence intervals, based on four repetitions of measurements.

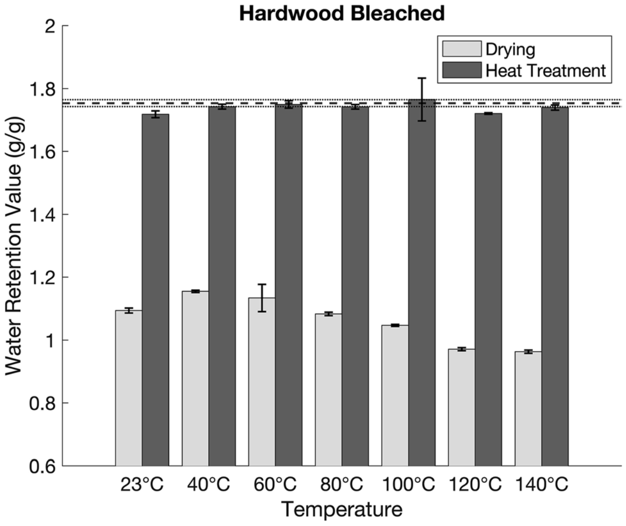

Figure 4: Water retention values for heat treated and dried bleached hardwood pulps in different temperatures, including never dried pulp as reference material indicated by a dashed line. Error bars represent 95 % confidence intervals, based on four repetitions of measurements.

is larger for hardwood. This is also shown in Figure 8, where the hornification ratio increases to 1.5 for hardwood. This may seem counterintuitive, but it is related to the increased capability of intense swelling prior to drying. The swelling of cellulose fibres depends on the balance between expansion forces, primarily osmotic pressure, and the fibres'/fibrils' structural counterforces (Hubbe et al. 2024). Charged side groups, such as uronic acid groups, increase the negative charge in the cell wall. This enhances the osmotic pressure and, consequently, the swelling capacity upon contact with water (Hubbe et al. 2024). Swelling prior to drying has a positive effect on the WRV of never-dried pulps and can lead to more extensive collapse after drying, thus resulting in higher hornification (von Schreeb et al. 2025). As the most hydrophilic polymer in wood, hemicellulose is fully amorphous and probably contributes to a more swollen fibre material itself (Hubbe et al. 2024). Additionally, the charges create electrostatic repulsion between the cell wall polymers, making it easier for water to penetrate (Berglund et al. 2020). The thinner cell walls of short-fibre pulp also offer less resistance to swelling pressure. Together, these properties contribute to hardwood pulp generally exhibiting a higher WRV than softwood pulp (Risén et al. 2004) and, as demonstrated here, achieving a higher degree of hornification.

Figures 2–4 also show that hornification of fibre materials is evident during drying at room temperature, as observed in previous studies (Salmén and Stevanic 2018; Sjöstrand et al. 2023, 2024). The water retention value (WRV) decreases significantly compared to never-dried samples, indicating irreversible changes such as reduced swelling, increased stiffness and collapse of the cell wall structure occur during drying at low temperatures. Furthermore, a gradual decline in WRV is observed as the drying temperature increases. Minor changes are seen for temperatures below 60 °C, but a clear decline in WRV is observed from 60 °C up to 140 °C. Previous studies have demonstrated that hornification is a temperature-dependent phenomenon that increases with temperature (Luo and Zhu 2011; Salmén and Stevanic 2018; Sellman et al. 2023; Sjöstrand et al. 2024). These results are consistent with previous studies: pre-shrinking is a temperature-dependent phenomenon for both short- and long-fibre materials, whether bleached or unbleached. Interestingly, it is important to note that even the highest temperatures do not induce any hornification when the pulps are heat treated, which contradicts previous beliefs (Sjöstrand et al. 2024).

An interesting phenomenon that was observed was that hornification was more severe at lower and higher drying temperatures, with a local minimum occurring around 40–60 °C. This trend was particularly evident in unbleached softwood pulp (Figure 3) and bleached hardwood pulp (Figure 4). One possible explanation is that the ability of water to form hydrogen-bonded chains, which effectively 'pull' cellulose surfaces together (Sjöstrand et al. 2023), decreases with increasing temperature. However, at higher temperatures, the increased thermal motion of polysaccharide chains enhances surface-to-surface interactions during drying (Sjöstrand et al. 2024), which again leads to increased hornification. These two opposing, temperature-dependent mechanisms may explain the local minimum in hornification. Selecting drying temperatures within this optimal range could potentially minimise hornification.

Therefore, the previous explanation that hornification is a consequence of hydrogen bonds forming between cellulose chains in the absence of water (Barrios et al. 2024; Hashemzehi and Sjöstrand 2024; Sjöstrand et al. 2023) still stands. However, they do not explain why the degree of hornification increases with drying temperature. Molecular dynamics simulations have demonstrated that water can manipulate the hydroxyl groups on cellulose surfaces to form hydrogen bonds (Barrios et al. 2024). However, at higher temperatures, these interactions weaken due to increased molecular mobility. Salmén and Stevanic (2018) have presented a hypothesis that suggests fibrils also become more mobile at higher temperatures. This could expose a larger contact surface between the fibrils, enabling direct interactions through hydrophobic forces and van der Waals bonds. These interactions have also been suggested to contribute to pregelatinisation (Hashemzehi and Sjöstrand 2024; von Schreeb et al. 2025). Another suggestion is that chemical changes in the material contribute to increased bonding at temperatures above 100 °C. The results from Sjöstrand et al. (2024) show a correlation between increased yellowness in the fibre material and lower WRV, indicating that pyrolytic reactions have occurred. These colour changes can be attributed to caramelisation and dehydration reactions that occur in carbohydrates when heated in the absence of free water. Such reactions generate reactive intermediates (Yao et al. 2021), which can form covalent cross-links between cellulose chains. These cross-links are presumably ether bonds, resulting in a chemically locked structure that is more permanent (Sjöstrand et al. 2024). This study demonstrates that these mechanisms only occur in conjunction with water removal. The results indirectly contribute to the discussion on the influence of hydrogen bonds by showing that the strongest hornification effects emerge under drying conditions, where hydrogen-bond formation is thermodynamically favoured. This supports the view that hydrogen bonding plays a central role in the early consolidation of the fibre network. At the same time, the reduced temperature dependence observed during heat treatment is consistent with a shift toward interactions less sensitive to water structuring, such as hydrophobic attraction and van der Waals forces. In addition, the effect of the viscosity of water and its surface tension contributes to the capillary forces and viscous drag that influence fiber consolidation and bonding during drying. This interpretation aligns with the idea that multiple intermolecular forces act concurrently, with hydrogen bonds dominating at lower temperatures and other interactions, including hydrophobic, van der Waals, and capillary forces, increasing in relative importance as thermal motion, polymer mobility, and water-mediated effects rise. For all pulps in this study, the heat-treated samples exhibited very similar water retention values to the never-dried reference sample. The insignificant difference between the heat-treated samples and the never-dried reference sample suggests that an increase in temperature alone does not activate any hornification mechanism. This indicates that changes in the structure or water retention capacity of the pulp are not caused by the temperature itself, but rather in combination with the removal of water. As long as the fibre is saturated with water, increasing temperatures do not appear to affect its ability to retain flexibility and water-binding capacity. This suggests that hornification is not only a temperature-controlled mechanism in water-involving conditions, but is instead related to the degree of drying and loss of moisture from the fibre walls. Of course, this does not exclude covalent crosslinking.

3.2 Tensile strength of sheets

The strength properties of paper essentially depend on the interaction between the strength of the individual fibres and their ability to form a network (Asta et al. 2024; Kaplan et al. 2025). These parameters, together with others, are crucial for paper to carry loads. Therefore, in addition to fibre strength, fibre-to-fibre bonds and the number of bonds are also important for paper strength. If the fibre-fibre bonds are weaker than the fibres themselves, the fibres will pull away from each other before they break. Conversely, if the fibre network is stronger, the strength of the paper will be determined by the strength of the fibres themselves, since their breakage constitutes the weakest link (Asta et al. 2024; Kaplan et al. 2025). Traditionally, drying and hornification are associated with a reduction in paper sheet strength (Ferreira et al. 2023; Laivins and Scallan 1993; Oksanen et al. 1997), primarily due to the fibres becoming stiffer and their ability to form fibre-fibre bonds decreasing. Figures 5–7 show the tensile strength indexes of sheets from the three pulps, both dried and heat treated.

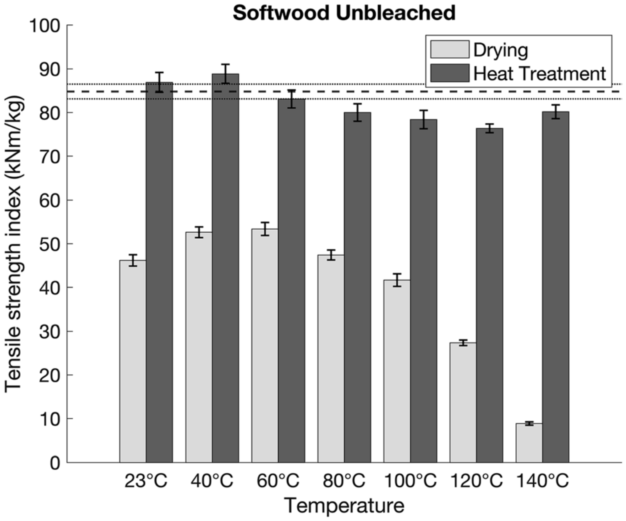

Figure 5: Tensile strength index (kNm/kg) for heat treated and dried unbleached softwood pulps in different temperatures, including never dried pulp as reference material indicated by a dashed line. Error bars represent 95 % confidence intervals, based on ten repetitions of measurements.

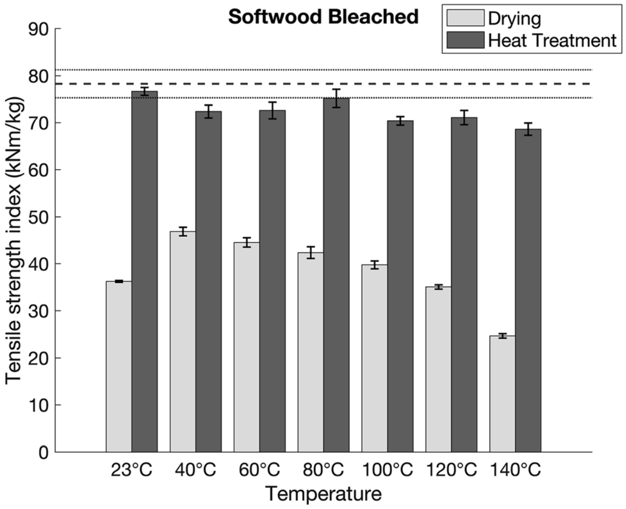

Figure 6: Tensile strength index (kNm/kg) for heat treated and dried bleached softwood pulps in different temperatures, including never dried pulp as reference material indicated by a dashed line. Error bars represent 95 % confidence intervals, based on ten repetitions of measurements.

The observations presented in Figures 5–7 empirically demonstrate that, despite clear differences tied to water removal and thermal exposure, all pulp types exhibit broadly similar behaviours under varied heat treatment conditions. Specifically, pulps that underwent heat treatment without water removal retained their tensile strength, whereas pulps dried at different temperatures showed reductions in strength.

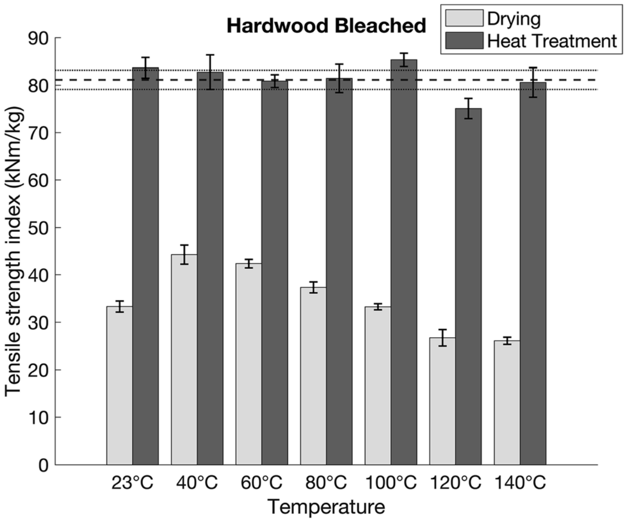

Figure 7: Tensile strength index (kNm/kg) for heat treated and dried bleached hardwood pulps in different temperatures, including never dried pulp as reference material indicated by a dashed line. Error bars represent 95 % confidence intervals, based on ten repetitions of measurements.

As discussed above, a local minimum in hornification was observed at a drying temperature of 40–60 °C. Conversely, a local maximum in tensile index occurred within the same temperature range (see Figures 5–7). These two phenomena are most likely directly connected, i.e. variations in tensile strength appear to be linked to variations in hornification. Figure 8 shows a linear relationship between tensile strength and the hornification ratio, as defined by Moser and Sjöstrand (2025). These results support the conclusion that the hornification ratio is a suitable metric for quantifying hornification and that decreased sheet strength and rewetting resistance are interconnected aspects of the hornification process.

The intact tensile strength in pulp samples that were heat-treated while retaining moisture suggests that water plays a protective or buffering role during thermal exposure. In keeping the pulp moist, the internal structure likely avoids interfibrillar collapse, hindering hornification. Conversely, pulp dried at the highest temperatures shows significant tensile strength loss, elevated drying temperatures effectively removes water within fibre walls, diminish fibre reswelling capacity and by stiffening the fibres, reduce the number of possible fibre–fibre bonds. In addition, irreversible hornification contributes to loss of network cohesion: high-temperature drying reduces the availability of pore volume within fibres, restricts conformability, and diminishes bond formation during sheet consolidation (Sjöstrand et al. 2024). The net effect manifests as a pronounced drop in tensile performance, emphasizing that excessive thermal treatment is detrimental to strength retention. For all dried pulps, there is a clear correlation between tensile strength index and hornification ratio (Figure 8), while no correlation is observed for heat treated pulps. Linear regression analysis gives r-squared values above 0.8 for dried pulps, and below 0.3 for heat treated pulps. The trends are shown in more detail in Table 1, where the dried samples exhibit strong, statistically significant negative slopes, indicating a consistent decrease in tensile strength as the hornification ratio increases. This relationship is supported by high R^2values, which show that the linear model explains most of the variability in tensile strength for the dried materials. By contrast, the heat-treated samples show non-significant slopes and low R^2values, suggesting that there is no clear or reliable linear relationship between tensile strength and hornification ratio after heat treatment.

In practical drying operations, the mechanisms underlying the observed minimum in hornification at temperatures of 40–60 °C can be mapped directly onto several key process variables, including hood temperature, dwell time and the evolving moisture profile of the web. Although industrial dryer air temperatures are much higher, the effective fibre temperature in the earliest stages of drying – in or immediately after the press section and before substantial evaporative cooling – could briefly fall within this range. During this period, water-mediated structuring facilitates close but reversible interfibre interaction, while polysaccharide mobility and chemical reactivity remain comparatively limited. Therefore, our results suggest that moderating exposure to higher temperatures during this initial, highly hydrated stage may help preserve interfibre swelling capacity and reduce premature hornification. It should be noted that this interpretation applies specifically to early drying conditions and is not intended as a target temperature for the full drying process, which involves complex heat and mass transfer regimes that cannot be replicated in our laboratory setup.

4 Conclusions

- Hornification shows a complex temperature dependence, with a consistent local minimum at 40–60 °C that aligns with a tensile maximum. This systematic cross-pulp confirmation supports earlier isolated observations.
- Water removal is confirmed as the primary driver of hornification. High temperature alone, when decoupled from drying, produces negligible structural change. This refines existing models by isolating the respective roles of heat and moisture.

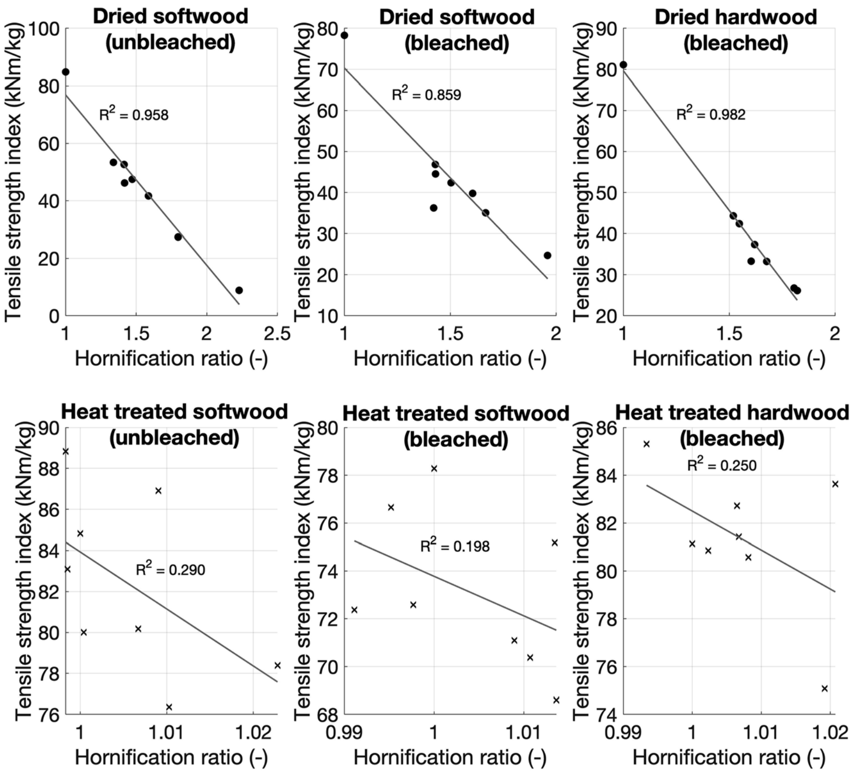

Figure 8: Tensile strength index plotted against hornification ratio for all three pulps, dried and heat treated, linear fit and R^2 values are shown. Linear regression was performed separately for each of the six conditions, drying and heat treatment, hardwood and softwood pulps.

Table 1: Results of linear regressions relating hornification ratio to tensile strength index for dried and heat-treated samples, including regression coefficients, 95 % confidence intervals, and coefficients of determination (R^2).

	Intercept	95 % CI (intercept)	Slope	95 % CI (slope)	R^2
Dried softwood unbleached	136.24	[116.75, 155.73]	−59.36	[−71.78, −46.94]	0.958
Dried softwood bleached	123.76	[90.86, 156.67]	−53.46	[−75.06, −31.86]	0.859
Dried hardwood bleached	147.75	[133.06, 162.44]	−68.09	[−77.32, −58.86]	0.982
Heat treated softwood unbleached	362.32	[−74.90, 799.53]	−278.39	[−713.08, 156.31]	0.290
Heat treated softwood bleached	239.55	[−94.91, 574.01]	−165.77	[−498.94, 167.40]	0.198
Heat treated hardwood bleached	245.28	[−38.58, 529.14]	−162.78	[−444.62, 119.06]	0.250

- Hardwood pulps exhibit stronger hornification than softwood, reinforcing prior trends, but this is now demonstrated across a unified experimental framework.
- A linear, cross-pulp proportionality has been established between tensile loss and the hornification ratio, providing a quantitative link between structural consolidation and strength reduction that had not previously been demonstrated.
- The combined results strengthen hydrogen-bond–dominated models of hornification while clarifying how temperature, moisture removal and fibre type jointly govern the process.

Acknowledgments: The authors acknowledge funding from the Åforsk Foundation. Carl-Anton Karlsson is also acknowledged for experimental support. Billerud AB is acknowledged for the supply of pulp.

Research ethics: Not applicable.

Informed consent: Not applicable.

Author contributions: SM and BS planned the experiments together, SM wrote a master thesis on the subject with BS as supervisor. BS has reworked chosen parts of the master thesis into this manuscript with assistance from GH. All authors read and approved the final manuscript.

Use of Large Language Models, AI and Machine Learning Tools: The authors have used the AI tool DeepL for language check, and the LLM ChatGPT for translating between Swedish and English.

Conflict of interest: The authors have no relevant financial or non-financial interest to disclose.

Research funding: The research was funded by the Åforsk foundation, grant number 24-372.

Data availability: All data available on request.

References

Asta, N., Kaplan, M., Kulachenko, A., Östlund, S., and Wågberg, L. (2024). Influence of density and chemical additives on paper mechanical properties. *Cellulose* 31: 5809–5822, https://doi.org/10.1007/s10570-024-05917-6.

Barrios, N., Parra, J.G., Venditti, R.A., and Pal, L. (2024). Elucidation of temperature-induced water structuring on cellulose surfaces for environmental and energy sustainability. *Carbohydr. Polym.* 329: 121799, https://doi.org/10.1016/j.carbpol.2024.121799.

Belle, J. and Odermatt, J. (2016). Initial wet web strength of paper. *Cellulose* 23: 2249–2272, https://doi.org/10.1007/s10570-016-0961-7.

Berglund, J., Mikkelsen, D., Flanagan, B.M., Dhital, S., Gaunitz, S., Henriksson, G., Lindström, M.E., Yakubov, G.E., Gidley, M.J., and Vilaplana, F. (2020). Wood hemicelluloses exert distinct biomechanical contributions to cellulose fibrillar networks. *Nat. Commun.* 11: 1–16, https://doi.org/10.1038/s41467-020-18390-z.

Christiernin, M., Henriksson, G., Lindström, M.E., Brumer, H., Teeri, T.T., Lindström, T., and Laine, J. (2003). The effects of xyloglucan on the properties of paper made from bleached kraft pulp. *Nord. Pulp Pap Res. J.* 18: 182–187.

Çiçekler, M. and Tutuş, A. (2025). Hornification in recycled fibers and its impacts on quality and recycling efficiency. In: *2nd international conference on recent and innovative results in engineering and technology (ICRIRET 2025)*, pp. 94–104.

Dodson, C. (1970). The nature of bonds in paper and the behaviour of paper under mechanical strain. *Rep. Prog. Phys.* 33: 1–43, https://doi.org/10.1088/0034-4885/33/1/301.

Fernandes Diniz, J.M.B., Gil, M.H., and Castro, J.A.A.M. (2004). Hornification – its origin and interpretation in wood pulps. *Wood Sci. Technol.* 37: 489–494, https://doi.org/10.1007/s00226-003-0216-2.

Ferreira, C.I.A., Pires, F., Martins, M., Ramos, A.N.A., Pinto, P.C.R., and Jorge, R. (2023). Recycling performance of softwood and hardwood unbleached kraft pulps for packaging papers. *Tappi J.* 22: 73–86, https://doi.org/10.32964/tj22.2.73.

Hashemzehi, M., Sjöstrand, B., Håkansson, H., and Henriksson, G. (2024). Degrees of hornification in softwood and hardwood kraft pulp during drying from different solvents. *Cellulose* 31: 1813–1825, https://doi.org/10.1007/s10570-023-05657-z.

Hirn, U. and Schennach, R. (2015). Comprehensive analysis of individual pulp fiber bonds quantifies the mechanisms of fiber bonding in paper. *Sci. Rep.* 5: 1–9, https://doi.org/10.1038/srep10503.

Hirn, U. and Schennach, R. (2017). Fiber-fiber bond formation and failure: Mechanisms and analytical techniques. In: *Advances in pulp and paper research, trans. of the XVIth fund. Res. Symp.*, pp. 839–863.

Hubbe, M.A., Sjöstrand, B., Lestelius, M., Håkansson, H., Swerin, A., and Henriksson, G. (2024). Swelling of cellulosic fibers in aqueous systems: a review of chemical and mechanistic factors. *BioResources* 19: 6859–6945, https://doi.org/10.15376/biores.19.3.Hubbe.

Kaplan, M., Johansson, A., and Östlund, S. (2025). Characterising the mechanical behaviour of dry-formed cellulose fibre materials. *Nord. Pulp Pap Res. J.* 40: 173–189, https://doi.org/10.1515/npprj-2024-0076.

Kato, K.L. and Cameron, R.E. (1999). A review of the relationship between thermally-accelerated ageing of paper and hornification. *Cellulose* 6: 23–40, https://doi.org/10.1023/A:1009292120151.

Laivins, G.V. and Scallan, A.M. (1993). The mechanism of hornification of wood pulps. In: *Transactions of the Xth fundamental research symposium Oxford*, pp. 1235–1260.

Lindström, T., Wågberg, L., and Larsson, T. (2005). On the nature of joint strength in paper – a review of dry and wet strength resins used in paper manufacturing. In S.J. I'Anson (Ed.), Advances in paper science and technology, trans. of the XIIIth fund. Res. Symp. Cambridge, pp. 457–562.

Luo, X. and Zhu, J.Y. (2011). Effects of drying-induced fiber hornification on enzymatic saccharification of lignocelluloses. *Enzyme Microb. Technol.* 48: 92–99, https://doi.org/10.1016/j.enzmictec.2010.09.014.

Mo, W., Chen, K., Yang, X., Kong, F., Liu, J., and Li, B. (2022). Elucidating the hornification mechanism of cellulosic fibers during the process of thermal drying. *Carbohydr. Polym.* 289: 119434, https://doi.org/10.1016/j.carbpol.2022.119434.

Moser, C. and Sjöstrand, B. (2025). Hornification of softwood and hardwood pulps correlating with a decrease in accessible surfaces. *ACS Omega* 10: 26164–26171, https://doi.org/10.1021/acsomega.5c03470.

Newman, R.H. (2004). Carbon-13 NMR evidence for cocrystallization of cellulose as a mechanism for hornification of bleached kraft pulp. *Cellulose* 11: 45–52, https://doi.org/10.1023/B:CELL.0000014768.28924.0c.

Oksanen, T., Buchert, J., and Viikari, L. (1997). The role of hemicelluloses in the hornification of bleached kraft pulps. *Holzforschung* 51: 355–360, https://doi.org/10.1515/hfsg.1997.51.4.355.

de Oliveira, M.H., Maric, M., and van de Ven, T.G.M. (2008). The role of fiber entanglement in the strength of wet papers. *Nord. Pulp Pap Res. J.* 23: 426–431, https://doi.org/10.3183/npprj-2008-23-04-p426-431.

Olsson, A.M. and Salmén, L. (2001). Molecular mechanisms involved in creep phenomena of paper. *J. Appl. Polym. Sci.* 79: 1590–1595, https://doi.org/10.1002/1097-4628(20010228)79:9<1590::aid-app70>3.0.co;2-5.

Przybysz, P., Dubowik, M., Kucner, M.A., Przybysz, K., and Przybysz Buzała, K. (2016). Contribution of hydrogen bonds to paper strength properties. *PLOS One* 11: e0155809, https://doi.org/10.1371/journal.pone.0155809.

Risén, J., Hultén, A.H., and Paulsson, M. (2004). Influence of fiber properties on the network strength of softwood and hardwood kraft pulp fibers from different stages of a bleaching sequence. *J. Wood Chem. Technol.* 24: 289–306, https://doi.org/10.1081/WCT-200046238.

Salmén, L. and Stevanic, J.S. (2018). Effect of drying conditions on cellulose microfibril aggregation and "hornification.". *Cellulose* 25: 6333–6344, https://doi.org/10.1007/s10570-018-2039-1.

Tze, W.T. and Gardner, D.J. (2001). Swelling of recycled wood pulp fibers: effect on hydroxyl availability and surface chemistry. *Wood Fiber Sci.* 33: 364–376.

von Schreeb, A., Sjöstrand, B., Ek, M., and Henriksson, G. (2025). Drying and hornification of swollen cellulose. *Cellulose* 32: 5179–5189, https://doi.org/10.1007/s10570-024-06352-3.

Sellman, F.A., Benselfelt, T., Larsson, P.T., and Wågberg, L. (2023). Hornification of cellulose-rich materials – a kinetically trapped state. *Carbohydr. Polym.* 318: 121132, https://doi.org/10.1016/j.carbpol.2023.121132.

Shenoy, P. and Aithal, P.S. (2016). A study on history of paper and possible paper free world. *Int. J. Manag. IT Eng.* 6: 337–355.

Sjöstrand, B., Karlsson, C.-A., Barbier, C., and Henriksson, G. (2023). Hornification in commercial chemical pulps: dependence on water removal and hornification mechanisms. *BioResources* 18: 3856–3869, https://doi.org/10.15376/biores.18.2.3856-3869.

Sjöstrand, B., Karlsson, C.A., Barbier, C., and Henriksson, G. (2024). Hornification in commercial chemical pulps: dependence on drying temperature. *BioResources* 19: 7042–7056, https://doi.org/10.15376/biores.19.4.7042-7056.

Sjöström, E. (1993). *Wood chemisty fundamental and applications*. Academic Press.

Torgnysdotter, A. and Wågberg, L. (2004). Influence of electrostatic interactions on fibre/fibre joint and paper strength. *Nord. Pulp Pap Res. J.* 19: 440–447, https://doi.org/10.3183/npprj-2004-19-04-p440-447.

Wågberg, L. (2022). Fundamentals of interactions between cellulose-rich surfaces. In: *Fundamentals of papermaking, trans. Xth fund. Res. Symp*.

Wohlert, M., Benselfelt, T., Wågberg, L., Furó, I., Berglund, L.A., and Wohlert, J. (2022). Cellulose and the role of hydrogen bonds: not in charge of everything. *Cellulose* 29: 1–23, https://doi.org/10.1007/s10570-021-04325-4.

Yao, Y.G., Wang, W.Y., Chen, L.Y., Liu, H.M., Yan, R.Z., Li, S., and Wang, X. De. (2021). Structural changes of cellulosic polysaccharides in sesame hull during roasting at various temperatures. *Qual. Assur. Saf. Crop Foods* 13: 13–24, https://doi.org/10.15586/qas.v13i2.876.

Zhao, B. and Kwon, H.J. (2011). Adhesion of polymers in paper products from the macroscopic to molecular level – an overview. *J. Adhes. Sci. Technol.* 25: 557–579, https://doi.org/10.1163/016942410x525821.