Role of Hornification of Cellulose Rich Biomass for Pellet Production

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Hornification of cellulose-rich materials, particularly wood pulps, occurs when chemical bonds form between cellulose surfaces, along with intermolecular forces created during dewatering and drying, preventing the material from reswelling in water to its original structure. Hornification of pulps results in a reduced ability to form effective fiber networks and therefore weaker paper products. The objective of this work was to investigate the role of hornification in pelletized cellulosic biomass and materials in general to provide more information than can be obtained by measuring standard wet state properties, such as water retention. Pellets were produced from chemical pulps with different degrees of hornification, as indicated by the water retention value (WRV), and their mechanical performance was evaluated. The chemical pulps served as a model material for investigating hornification. Pulps with higher hornification produced pellets with inferior mechanical properties, which has not been shown before by such a test. This effect is attributed to increased fiber stiffness and reduced surface flexibility, which limits fiber-fiber bonding. In addition, high drying temperatures prior to pelletizing, and thus higher hornification, will increase compression energy and friction in the pelletizing process. A novel connection was observed between WRV and mechanical performance, highlighting the impact of hornification on the surface interactions of cellulose-based materials.

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INTRODUCTION

Wet forms of cellulose-rich biomass, such as chemical pulps, that are dried, become to varying degrees *hornified*, meaning that the drying induce strong semi-irreversible bonds between cellulose surfaces, both within and between fibers and by closure of small pores in the fiber wall. This results in particle collapse of pores within the cell walls of the fibers, and particularly resistance of the fibers to reswell to the original shape and behaviour in water after the hornification has happened (Jayme 1944; Laivins and Scallan 1993; Kato and Cameron 1999; Tze and Gardner 2001; Newman 2004; Fernandes Diniz *et al.* 2004; Salmén and Stevanic 2018; Wohlert *et al.* 2022; Mo *et al.* 2022; Sjöstrand *et al.* 2023; Hashemzehi *et al.* 2024; Sjöstrand *et al.* 2024; Hubbe *et al.* 2024; von Schreeb *et al.* 2025; Moser and Sjöstrand 2025). In the application of wood pulp fibers for papermaking, hornification gives decreased fiber wall swelling, decreased

internal and external fibrillation, and decreased flexibility compared with never dried pulp, which leads to a decreased ability to form effective fiber networks with many fiber-fiber bonds, thereby resulting in lower paper strength and higher bulk (Laivins and Scallan 1993; Oksanen *et al.* 1997; Ferreira *et al.* 2023).

In essence, hornification makes the fiber material stiff and hard, and it is also an obstacle for rewetting. Research about this phenomenon has been hampered by the lack of simple and effective methods for quantification; *water retention value* (WRV), a simple and robust method for measuring how much water a biomass (chemical pulp etc.) sample can hold, have been the method of choice for many resent publications, all since it was introduced by Jayme (1944), but this method only measures one aspect of the hornification – the decrease in water adsorption. Adsorption methods measuring the accessible surfaces of fibers have been shown to correlate well with WRV, although they are fairly similar and are also measured in a wet environment (Moser and Sjöstrand 2025).

The mechanistic background for hornification is under debate (Mo *et al.* 2022; Wohlert *et al.* 2022; Sellman *et al.* 2023), with several different suggested mechanisms and alternative chemical bonds responsible. Recent investigations based on WRV have shown that hornification is much stronger when cellulose-rich material is dried from water, than from organic solvents, and that a higher polarity of the organic solvent gives stronger hornification (Sjöstrand *et al.* 2023; Hashemzehi *et al.* 2024). A more swollen and less ordered cellulose hornifies much more strongly than a highly crystalline cellulosic materials (von Schreeb *et al.* 2025). Higher drying temperature gives stronger hornification, and this factor becomes stronger at temperatures above 100 °C (Sjöstrand *et al.* 2024). The strong hornification at temperatures over 100 °C is related to a yellowing of the material (Sjöstrand *et al.* 2024).

These observations have led to a suggestion of a model for hornification of cellulose rich material, where the hydrogen bonding ability of water play a key role (Sjöstrand *et al.* 2023; 2024; von Schreeb *et al*. 2025). Water molecules in its liquid state form more or less stable hydrogen bond networks, "hydrogen bond chains" (Clark 1985; Fellers and Norman 1998; Israelachvili 2011). The idea is that cellulose alcohols are included in such networks and that water to some extent crosslink cellulose surfaces, which is manifested as capillary forces (Sjöstrand *et al.* 2023). When water is gradually removed during drying, cellulose surfaces will be directed towards each other and furthermore guide cellulose alcohols so they form multiple hydrogen bonds. Moreover, also hydrophobic surfaces on the biomass will come closer to each other and can form hydrophobic interaction and when close enough also van der Waals forces. These interactions are not easily broken by water. If cellulose is swollen, more hydrophobic surfaces might be exposed and hornification therefore may be intensified (von Schreeb *et al.* 2025). A similar phenomenon might occur at higher temperature, since increased mobility (Salmén and Stevanic 2018) of the cellulose chains might stimulate hydrophobic interactions. At the highest temperatures dehydrations of the sugar residues might lead to both covalent crosslinking and yellowing (Sjöstrand *et al.* 2024). In summary, it was suggested that hydrogen bonds are the driving force behind hornification, but that hydrophobic interactions, van der Waals bonds, and covalent crosslinking also play roles. Other researchers emphasize additional bonding mechanisms beyond hydrogen bonds, claiming they are more important in hornification (Fernandes Diniz *et al.* 2004; Newman 2004; Wohlert *et al.* 2022; Sellman *et al.* 2023).

Hornification plays a dual role in papermaking; papers made from dried pulp have generally fewer fiber-fiber interactions than never dried pulps (Laivins and Scallan 1993). However, hornified fibers are usually stiffer than non-hornified ones, which can be beneficial for certain applications by creating higher bulk, such as central layer in boards where bending stiffness is important (Kajanto *et al.* 1998). Less is known of the role of hornification in other processes.

Pelletization is a process in which biomass of different kind is pressed together, water free (the biomass can of course contain various degrees of moisture content), to form pellets, as cylinders around 1 cm in diameter. Common subjects for pelletization are saw dust and milled wood residues, but many different kinds of industrial residues in the form of biomass of different kinds are pelletized. Cellulose and other polysaccharides are often a major constituent, and often they are subjected to drying prior to their pelletization. Produced pellets are often used as solid fuel, but they can also be a way of transporting and packaging biomass for other applications (Anukan *et al*. 2021). Important technical problems in the pellet industries include in addition to production costs, uneven and sometimes poor durability (that case dust formation) (Frodeson *et al.* 2019), and that heat and toxic and burnable gases sometimes are formed during storage (Siwale *et al*. 2024).

Unlike kraft pulp fibers, which undergo significant structural and bonding changes upon drying due to hornification, lignified woody materials with most of the wood components still present in the material, such as sawdust and wood residues, are expected to be less susceptible to this phenomenon (Oksanen *et al.* 1997). One possible explanation of this might be that lignin and hemicelluloses are acting as spacers between the cellulose surfaces and thereby facilitating reswelling in water, thus limiting hornification. This distinction is important when evaluating the properties of once-dried versus hornified feedstocks in pelletization processes.

Drying temperature plays a crucial role in determining the quality of wood pellets, particularly in terms of their density and mechanical strength. Research has shown that increasing the drying temperature significantly influences the specific density and durability of pellets; however, elevated moisture content in the raw material can offset these effects (Wróbel *et al*. 2020). Excessive drying temperatures may also result in lower bulk density, which complicates storage and transportation. Studies further indicate that drying temperature affects the grindability of biomass, which indirectly impacts the density and strength of the final pellets. Higher drying temperatures can alter the material's properties, negatively influencing the grinding process and ultimately reducing pellet quality. To what degree these phenomena are related to hornification of cellulose important for the paper industry is not known. To the authors' knowledge, this study is significant for its connection of the fields of biomass pelletizing and cellulosic hornification.

To study the role of hornification in pelleting and materials in general, pellets were made from chemical pulps (*i.e.*, mainly cellulose and hemicelluloses) hornified to varying degrees, and the properties of the pellets were characterized. The pulps used in this study served as a model material for investigating hornification in a dry process. The authors are not suggesting that pelletizing is an appropriate application for chemical papermaking pulps. The results will provide a base for a discussion about hornification phenomena in general, and of the importance of drying conditions in the pellet manufacture.

EXPERIMENTAL

Pulp Fibers

Never-dried, fully bleached chemical softwood kraft pulp was obtained from Gruvön Mill, Billerud AB (Grums, Sweden). The pulp, a blend of Norway spruce (*Picea abies*) and Scots pine (*Pinus sylvestris*), had a consistency of 7.2%, a brightness of 89% (ISO 2470-1 2018) and an intrinsic viscosity of 760 dm³/kg (ISO 5351 2010).

Drying Procedures and Water Retention Values

The pulp was dried at a temperature of 125 °C in a laboratory oven, in up to five drying cycles with disintegration and rewetting between each cycle. To be able to show reference values of the same pulp, one portion of never-dried pulp were kept and measured in parallel to the high temperature drying cycles, as well as one once-dried variant of the pulp, dried at room temperature (approximately 20 °C). The drying process was started by manually squeezing out the free water to achieve a dryness of 20 to 30%; then the pulps were spread on blotting paper and placed in the oven (125 °C). The 20 °C reference pulp was dried at room temperature in a fume hood at 20 °C. During drying, the temperature inside the pulp pieces was monitored using a Mitec AT40 datalogger to ensure stable temperatures with no deviations from the set temperature. All samples were left in the ovens and fume hoods until completely dry, which took at least 24 hours. To rewet the samples to be subjected to further drying cycles, disintegration was performed at 30000 revolutions in an L&W Pulp Disintegrator (App 03) prior to pressing and distribution on new blotting papers. Before and after each drying cycle, the WRV was measured according to ISO 23714 (2014). WRV ranges were calculated to show the variation of the results, defined as half the distance between the maximum and minimum values, displayed as error bars in the results section. An overview of the drying temperatures and cycles, along with sample names, is shown in Table 1.

Table 1. Overview of Drying Procedure of Softwood Kraft Pulps

Sample	Number of Drying Cycles	Drying temperature (°C)
Never-dried	0	-
Once-dried 20 °C	1	20
Once-dried 125 °C	1	125
Dried two times 125 °C	2	125
Dried three times 125 °C	3	125
Dried four times 125 °C	4	125
Dried five 125 °C	5	125

Single Pellet Press

Pellets were produced in a single pellet press unit (SPP), described previously (Frodeson *et al.* 2018; Anukam *et al.* 2019; Ståhl *et al.* 2024), located at the Environmental and Energy Systems at Karlstad University, Sweden. With one difference from the references, a new modern testing machine was used, namely MTS Exceed E43 instead of 505/60B, Riedlingen. An electro-mechanic press mechanism moved the piston downward toward the cylinder die (also known as the press channel), exerting pressure on the material within the die. The cylinder die is connected to an electric heating element, allowing precise temperature regulation during the process. Additionally, a bottom plate is employed to create back pressure during pressing (Fig. 1). The experimental conditions of the single pellet press are shown in Table 2.

Table 2. Experimental Conditions of the Single Pellet Press

Die temperature: 100 °C
Die diameter 8.2 mm
Press speed: 30 mm/min
Hold time: 10 seconds
Material loaded: 1 gram
Max compression force 15kN

After loading the channel die with 1.0±0.05 gram of material and initiating the press system, the piston descends at a constant speed until the compression force reaches 1 kN. Subsequently, the speed is adjusted based on the specific test series being conducted. The press bar continues to apply pressure until it reaches the maximum compression force. The pressure and duration of the press depend on the material being pressed and the specific test conditions. At this point, the press bar holds its position for a specified duration, depending on the hold time. The pellet is then formed, and the press bar descends again to extract the pellet from the press channel. Once the required pressure is applied, the friction phase commences, and the pellet is ejected from the die.

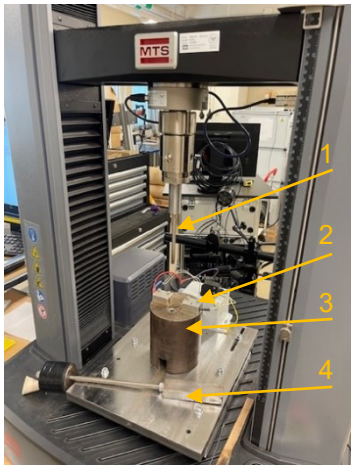

Fig. 1. Single pellet press 1) movable module with piston 8.0 mm 2) heating element 3) cylinder die with a press channel 8.2 mm 4) bottom plate

The samples were pelletized at two moisture content levels: 4.2 ± 0.5% and bone-dry (0% moisture content). The solid density of the pellets was determined by measuring the average length, diameter and weight of ten pellets.

Energy usage in single pellet press

The conversion of raw materials into pellets involves both the compression process and the friction process. These processes require energy to overcome the friction between the raw materials and the die hole. During the compression and friction stages of feed pellet production, force-displacement curves are documented. The energy use for compression (W_{comp}) and friction (W_{fric}) can be determined by integrating the force-displacement data using Eqs. 1 and 2,

$$w_{comp} = \frac{\int_{s_1}^{s_2} F_{comp}(x)dx}{m} \text{ [J/g]} \quad (1)$$

$$w_{fric} = \frac{\int_{s_1}^{s_2} F_{fric}(x)dx}{m} \text{ [J/g]} \quad (2)$$

where w_{comp} is compression energy use and w_{fric} is the friction energy use [J/g], *F(x)* is the compression pressure force [N], *m* is the weight of the loaded material which is 1 g, and s_1 and s_2 are the initial and the final position of the press bar [mm].

The maximum force F_{max} (kN) achieved by the piston pressing out the pellet from the cylinder, was created by the rest friction between the surface of the pellet and the die, representing the maximum potential backpressure level created.

RESULTS AND DISCUSSION

Bleached softwood kraft pulp was subjected to drying in several cycles and different temperatures so that different degrees of hornification were obtained. The WRV data for the different drying conditions are shown in Fig. 2. There was a clear trend where never dried pulp had the highest WRV, and each drying cycle decreased the reswelling capacity of the pulp materials. The first drying cycle gave the largest drop in WRV and the temperature 20 °C versus 125 °C also gave a significant difference. For consecutive drying cycles of 125 °C, the loss in water retaining capacity was almost linear with each cycle, which was similar to the findings of Moser and Sjöstrand (2025). The results of decreasing WRV are also consistent with previous literature such as Lavins and Scallan (1993) and Sjöstrand *et al.* (2024). The absolute values of the present study are of course not exactly matching literature values, even for the very similar pulp types included in Sjöstrand *et al.* (2024). This is showing that even within pulp types, there will be variations between seasons, batches, and operators for the absolute values. Particularly, the linear decrease with increasing number of drying cycles have not been shown in older literature, where they instead have indicated a plateau value of the WRV reached after a certain number of cycles (Laivins and Scallan 1993). For the studied pulps, there might be a minimum plateau WRV after several additional drying cycles, which had not been reached in the present study.

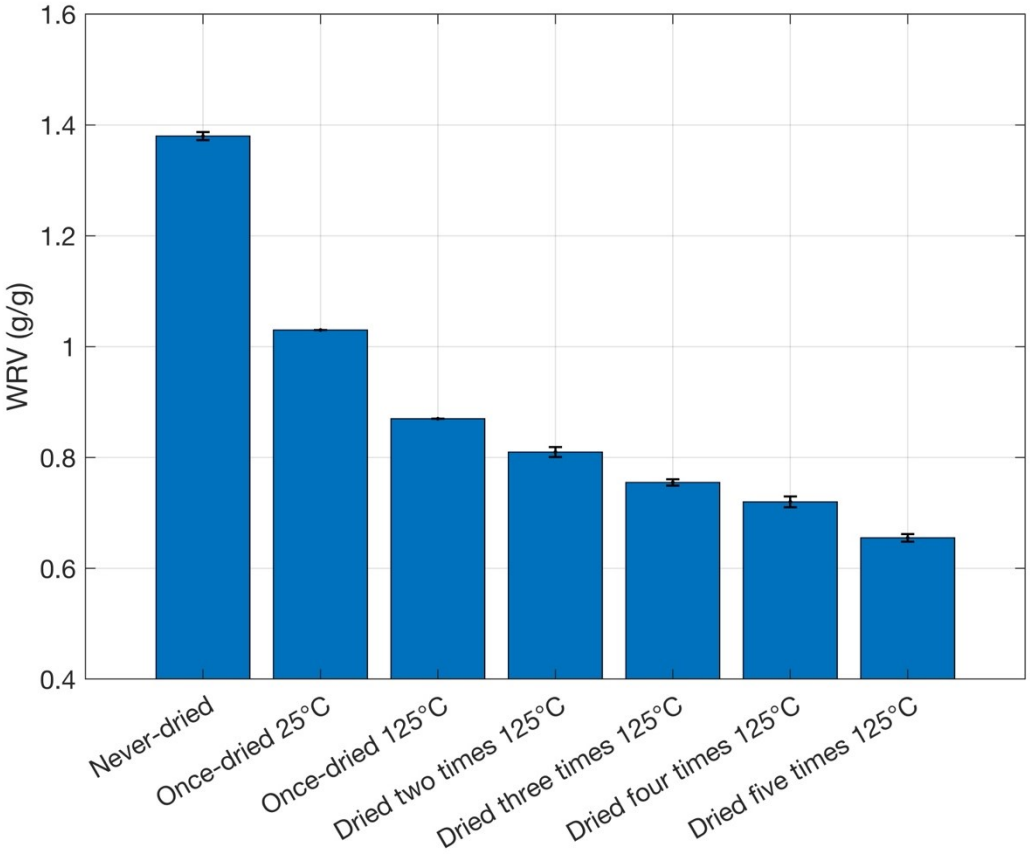

Fig. 2. WRV for the softwood pulps, never-dried, once dried at room temperature, and five drying cycles of 125 °C drying temperature. Error bars represent a range of half the distance between max and min values.

Figure 3 demonstrates that for a moisture content of 4%, the compression energy (W_{comp}) used during SPP production decreased with increasing WRV. The increased W_{comp} is attributed to the hornified fibers' increased stiffness and reduced surface flexibility. This is due to the cellulosic surfaces in and between the fibers being pulled together by chains of multiple hydrogen bonds during the drying cycles. For a moisture content of 0%, the results indicated that both the specimens dried once at 25 °C and those dried five times at 125 °C exhibited a W_{comp} of 51 J/g. This observation suggests that the fibers and the pellets become more resistant to compression at lower moisture content, leading to higher compression energy, regardless of their degree of hornification.

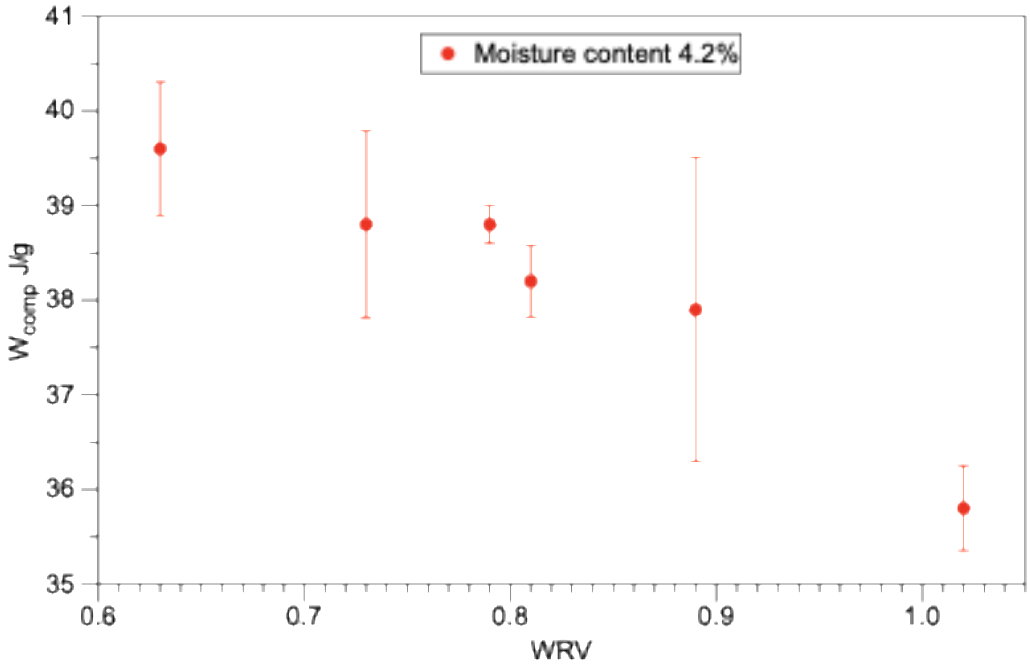

Fig. 3. Energy for compression for different drying conditions of the pulps, where error bars are representing standard deviation

Figure 4 shows the results for the W_{fric}. A slight decrease in W_{fric} with increasing WRV was observed, along with a more significant drop between those dried at 25 °C and those dried at 125 °C. This can be explained by the fact that stiffer fibers contribute to a higher kinetic friction coefficient between the fiber and the channel due to their increased stiffness.

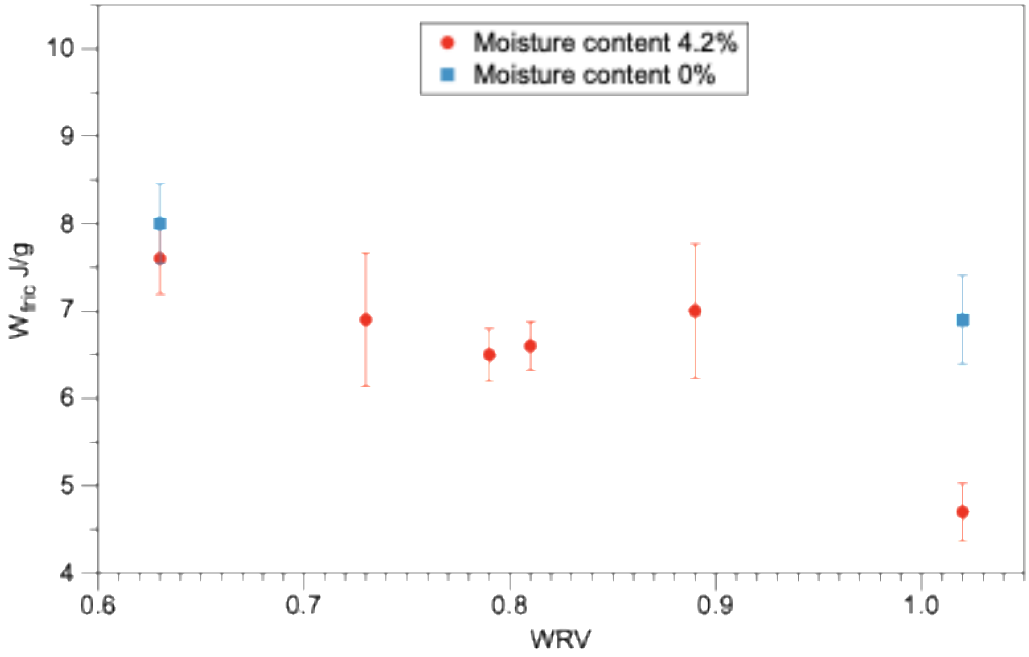

Fig. 4. Friction energy for different drying conditions of the pulps, error bars are representing standard deviation

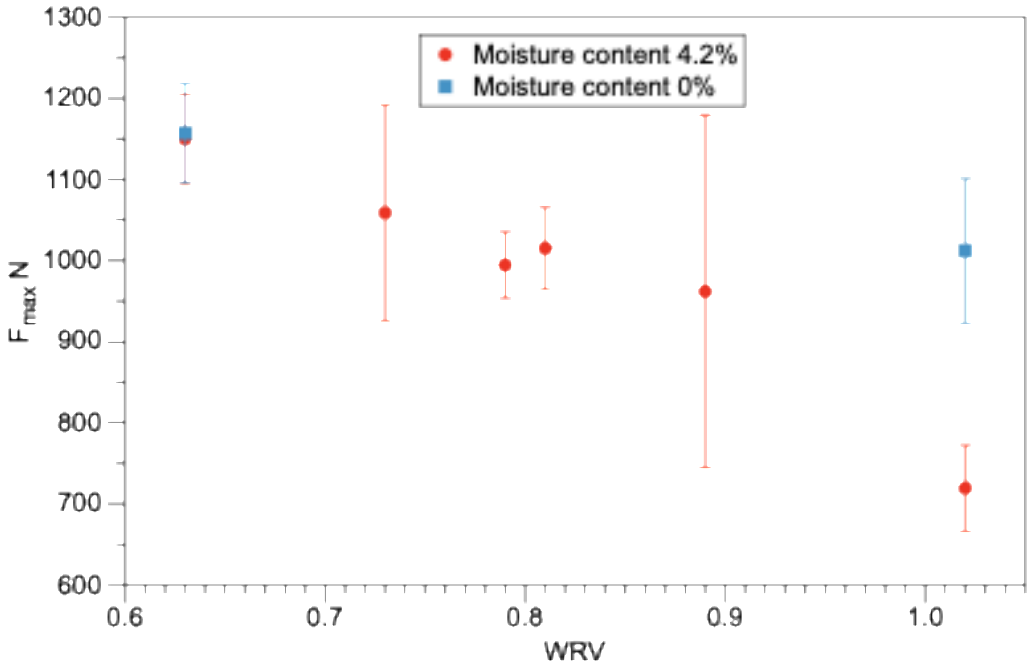

Fig. 5. Maximum friction force for different drying conditions of the pulps, error bars are representing standard deviation

The maximum force required for the piston to extrude the pellet was recorded as the highest force generated (Fig. 5). This value, referred to as F_{max} in N, represents the maximum potential backpressure that the pellets can create due to friction between the pellet surfaces and the die, which influences the press length of the die (Frodeson *et al.* 2018). Lower F_{max} values were observed for higher WRV values (Fig. 5). The increase in F_{max} has the same explanation as for W_{fric}, namely that there was an increasing static friction coefficient between the fiber and the channel due to the increased stiffness.

The quality parameter, solid pellet density was unaffected by the WRV and decreased as moisture content decreases (Fig. 6). The observed decrease in solid pellet density at 0% moisture content was anticipated, as moisture plays a crucial role in the binding process during pellet formation. There exists an optimal range of moisture content that enhances pellet quality, which varies based on the type of biomass and the specific processing conditions used.

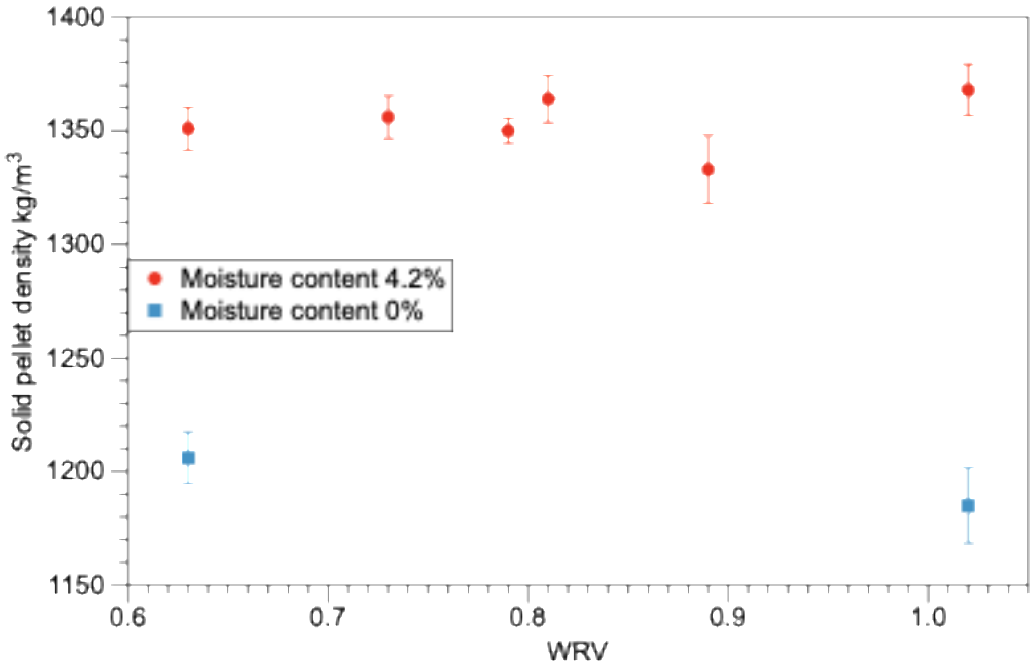

Fig. 6. Solid pellet density for different drying conditions of the pulps, where error bars are representing standard deviation

Technical significance

The parameters that were measured in this study for the pelletization process, namely the compression energy, friction energy, and maximum friction force, were all affected by hornification of polysaccharides, as indicated by a decrease in water retention value (WRV). These process properties are connected to the mechanical strengths of pellets indirectly, mainly by the different strengths and densities that can be achieved. However, it is important to note that the pulps used in this study consisted primarily of polysaccharides and contained only small amounts of lignin. In contrast, the predominant raw material in the Scandinavian pellet industry is sawdust—mainly from softwood—which contains significantly higher amounts of lignin. This difference limits the ability to draw definitive conclusions from this study, as the role of lignin in hornification remains poorly understood.

Nevertheless, it is worth noting that this work shows that materials with low lignin content can still be pelletized effectively, and previous studies have shown that even completely lignin-free polysaccharides are capable of forming high-quality pellets. Thus, the findings suggest that high drying temperatures may lead to differences in the pelletizing process, and perhaps lower pellet quality, due to increased hornification. A bonding agent such as starch can compensate for lower pellet quality. Starch content influences pellet durability, number of fines, and stability by enhancing binding properties during pelletizing (Larsson *et al.* 2015).

From a broader materials science perspective, the results of this study highlight that hornification is not merely a phenomenon related to water absorption; it also negatively impacts surface interactions. This could present challenges in applications where cellulose is used in products such as paper, boards, and composites.

CONCLUSIONS

1. When pellets are produced from chemical pulps with varying degrees of hornification, as indicated by water retention value (WRV), pulps with higher hornification are more difficult to process. This phenomenon is likely due to increased stiffness and reduced surface flexibility of the hornified fibers, leading to weaker fiber–fiber interactions.
2. High drying temperatures of biomass prior to pelletizing may increase hornification of the material, resulting in difficulties in pellets processes and potentially lower mechanical quality.
3. Hornification of cellulose is expected to reduce surface interaction in various types of cellulose-based materials.

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