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Section 3 **Wood Modification**

Unlocking the Potential of Dairy Coproducts in Wood Modification

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ABSTRACT

The production of dairy coproducts, such as whey (sweet or acid) and whey ultrafiltration permeate, has significantly increased in response to the rising global cheese consumption. Proteins found in by-products are purified for human nutrition. However, upcycling lactose, which is the major compound of cheese by-products, is more challenging due to the high cost of upcycling processes. Furthermore, their high chemical and biological oxygen demands classify them as potential environmental pollutants. One promising approach to maximize the utilization of lactose is the application for wood polyesterification, especially in combination with biosourced acid. This study investigates the modification of Scots pine sapwood through an *in-situ* polyesterification reaction using dairy-derived products. The results indicate a solution absorption exceeding 200%, ensuring deep penetration into the wood structure. After curing, the treated samples exhibited significant weight gain, which persists after water leaching. The cell wall bulking remained below 3% across all treatments, contributing to an anti-swelling efficiency of around 40% after leaching. These findings highlight the potential of dairy-derived polyesterification as a sustainable and efficient method for improving wood stability and durability.

Keywords: Wood modification, dairy coproducts, citric acid, wood durability, dimensional stability.

1. INTRODUCTION

The increasing global demand for dairy products has led to a significant rise in the production of dairy byproducts, particularly whey (sweet and acid) and whey ultrafiltration permeate. These coproducts are commonly used as animal feed or, to a lesser extent, in human nutrition. However, these conventional valorization pathways fail to fully exploit their economic potential and often contribute to environmental concerns due to their high chemical and oxygen demands (Smithers 2008). Consequently, there is a growing interest in identifying innovative and sustainable applications for dairy coproducts. Among the key constituents of dairy byproducts, lactose, a disaccharide primarily derived from whey, represents an abundant and underutilized resource. Recent studies have explored its potential in industrial applications, including bio-based polymer synthesis and material modifications (Panesar *et al.* 2007, Ramos *et al.* 2016, 2021). One promising approach involves the use of lactose in wood modification via polyesterification (Cadieux-Lynch *et al.* 2024), particularly in combination with citric acid, a well-known bio-based crosslinking agent (Lee *et al.* 2020). Polyesterification reactions can effectively alter the hygroscopicity of wood, thereby improving its dimensional stability and durability (Hill 2006, Berube *et al.* 2018, Kurkowiak *et al.* 2022, Hötte and Militz 2024).

This study investigates the *in-situ* polyesterification of Scots pine sapwood using dairy-derived lactose and citric acid. The objective is to assess the use of dairy coproducts in wood modification.

2. MATERIALS AND METHODS

2.1 Materials

Citric acid monohydrate (CA, approx. 97% purity) was purchased from PanReac AppliChem (Darmstadt, Germany) and used as received. Whey ultrafiltration permeate powder (WUP), sweet whey powder (SW), and galactose-oligosaccharides (GOS) were provided by DMK Group (Zeven, Germany). Their composition is presented in Table 1. Scots pine (*Pinus sylvestris* L) sapwood samples were prepared and cut according to the specifications in Table 2.

Table 1: Composition of the dairy coproducts used

	Carbohydrate(%)	Proteins (%)	Non-protein nitrogen (%)	Ash (%)	Moisture (%)
SW	75.7	11.0-15.0	-	8.5	4.5
WUP	86.0	-	3.0	8.0	2.0
GOS	98.2		-	-	1.8

Table 2: Wood sample dimensions and sampling for the different tests performed.

Sample size (L × T × R) mm³	Tests or analysis performed	Number of samples per treatment
10 × 25 × 25	Physical properties, chemical characterisation	15

2.2 Solution preparation

Six different formulations were prepared for the treatment of Scots pine sapwood, each with a solid content of 30% (w/w) in water. Different formulation compositions are presented in Table 3, where 30%WUP, 30%SW, and 30%GOS are an aqueous solution of WUP, SW, and GOS, respectively. WUPCA is an aqueous mixture of WUP (whey ultrafiltration permeate powder) and CA, SWCA is an aqueous mixture of SW (sweet whey powder) and CA and finally, GOSCA is an aqueous mixture of GOS (galactose-oligosaccharide powder) and CA.

Table 3: Composition of different formulations used to treat wood samples.

Formulation	CA (g)	WUP (g)	SW (g)	GOS (g)	Water (g)	Solid content % (w/w)	pH
30%WUP	-	90.0	-	-	210.0	30	6.0
30%SW	-	-	120.0	-	280.0		6.2
30%GOS	-	-	-	120.0	280.0		5.0
30%WUPCA	51.2	38.8	-	-	210.0		1.8
30%SWCA	63.7	-	56.3	-	280.0		1.9
30%GOSCA	72.0	-	-	48.0	280.0		1.4

2.3 Wood treatment and physical properties assessment

Wood samples were impregnated in an autoclave. First, a vacuum of 100 mbar was applied for 1 h, followed by a pressure of 10 bar for 2 h. The samples were taken out, and the excess solution was wiped off. They were then weighed, and dimensions were measured. Afterwards, the samples were dried at room temperature for seven days to avoid crack formation during curing (160 °C for 24 h). The solution uptake, weight gain percentage, cell wall bulking, and anti-swelling efficiency before leaching were determined as described in Table 3. Afterwards, samples were submitted to a water leaching test according to EN84 (2020), and all the previous physical properties were again determined. For all of these properties, the mean and standard deviation are visualized in this study.

Table 4 presents the equations used for the calculation.

Table 4: The equations used to determine physical properties

Measured properties	**Equation used for the calculation**	**Assignments of parameters**
Solution uptake (SU)	$SU = \frac{m_1 - m_0}{m_0} \times 100$	m_0: oven-dry weight before treatment m_1: weight after impregnation
Weight gain percentage (WGP)	$WGP = \frac{m_2 - m_0}{m_0} \times 100$	m_2: oven-dry weight after curing at 160 °C
Weight gain percentage after EN84 (WGP')	$WGP' = \frac{m_3 - m_0}{m_0} \times 100$	m_3: oven-dry weight after EN84
Cell wall bulking before EN84 (CWB)	$CWB = \frac{A_1 - A_0}{A_0} \times 100$	A_0: oven-dry cross-sectional area before treatment A_1: oven-dry cross-sectional area after curing at 160 °C.
Cell wall bulking after EN84 (CWB')	$CWB' = \frac{A_2 - A_0}{A_0} \times 100$	A_2: oven-dry cross-sectional area after EN84
Volumetric swelling (S)	$S = \frac{V_1 - V_0}{V_0} \times 100$	V_0: oven-dry volume V_1: volume after soaking
Anti-swelling efficiency before EN84 (ASE)	$ASE = \frac{S_0 - S_1}{S_0} \times 100$	S_0: volumetric swelling before treatment S_1: volumetric swelling after curing at 160 °C
Anti-swelling efficiency after EN84 (ASE')	$ASE' = \frac{S_0 - S_2}{S_0} \times 100$	S_2: volumetric swelling after EN84

2.5 Chemical composition assessment

Fourier Transform Infrared (FTIR) spectroscopy was conducted on an Alpha FTIR Spectrophotometer (Bruker Optik GmbH, Bremen, Germany) in the attenuated total reflection (ATR) mode to analyze the chemical changes during the wood modification. The measurements were performed at room temperature between wavenumbers of 4000 and 400 cm^{-1} with a resolution of 4 cm^{-1}. A total of 64 scans was recorded. Baseline correction of the spectra was automatically performed by the baseline correction tool integrated into the software. The peaks were normalized by max-min using the normalization correction tool integrated into the software. Pieces of wood samples were cut using a clean blade and put on the diamond crystal for recording. For each kind of sample, two repetitions were recorded.

3. RESULTS AND DISCUSSION

After the impregnation process, the formulation uptake was above 200% for all the formulations and higher than water uptake (Fig. 1).

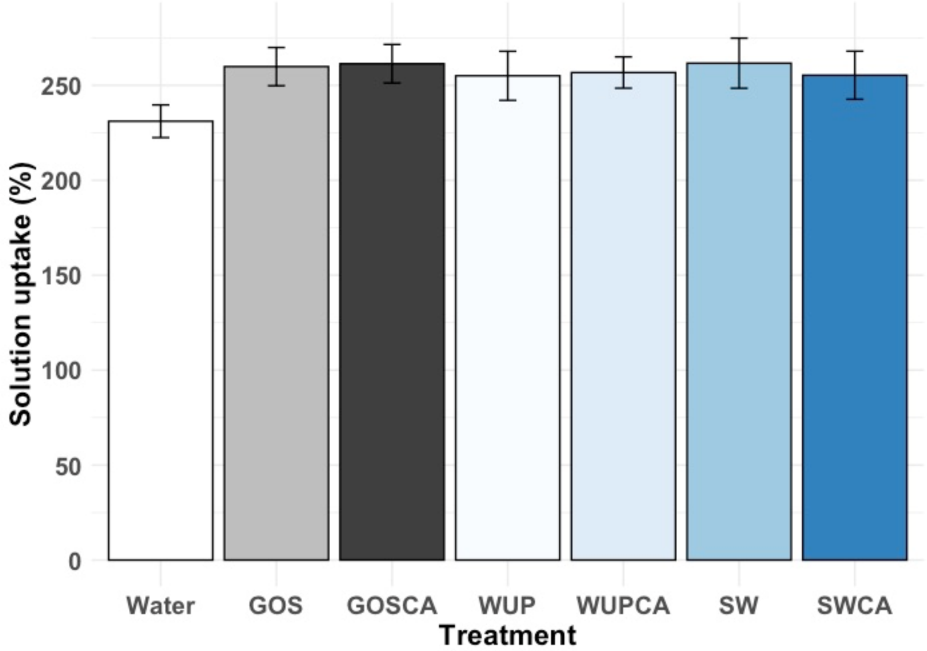

Figure 1: Comparison of the uptake of different formulations by wood samples

The higher uptake of formulations means that there is a good penetration of the formulations within the wood. However, the difference with water uptake is explained by the difference of water density and the densities of the formulations. The formulations were denser than water. This is important because, for this kind of wood modification, the penetration and location of this formulation in wood have a significant impact on wood properties.

After impregnation and drying at ambient air, samples were cured at 160 °C for 24 h. Before leaching (Fig. 2, left), all samples exhibited a WGP exceeding 45%. Interestingly, after leaching (Fig. 2, right), only the formulations including CA kept a WGP above 40%.

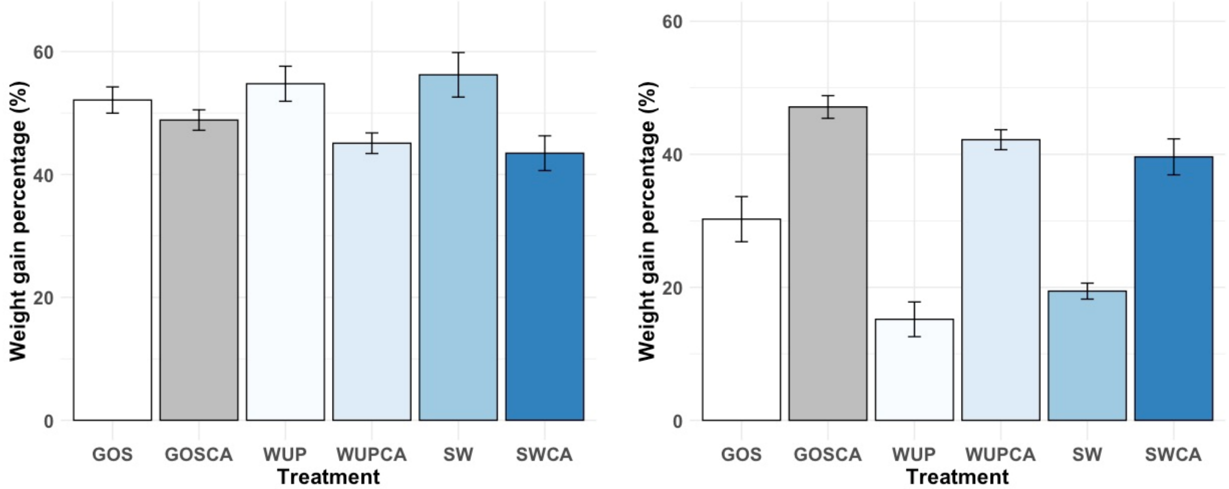

Figure 2: Comparison of weight gain percentage wood of treated with different formulations before (left) and after (right) the leaching test

These results suggest that for the formulations containing CA, a reaction occurs between CA and lactose in dairy coproducts, leading to macromolecules that react or entangle with wood cell wall matrix capable of resisting to water leaching. Unfortunately, for the formulations without CA, no reaction is expected between the dairy coproduct components and wood cell wall matrix at this temperature, so a large part of the retained compounds after curing were leached out. There is still a retention of some chemicals in these compounds, which is due to pressure impregnation.

The cell wall bulking (CWB) before the leaching test was above 2% for all formulations (Fig. 3, left). However, the CWB after the leaching was below 0 for the formulations without CA, and always above 2% for those containing CA (Fig 3, right).

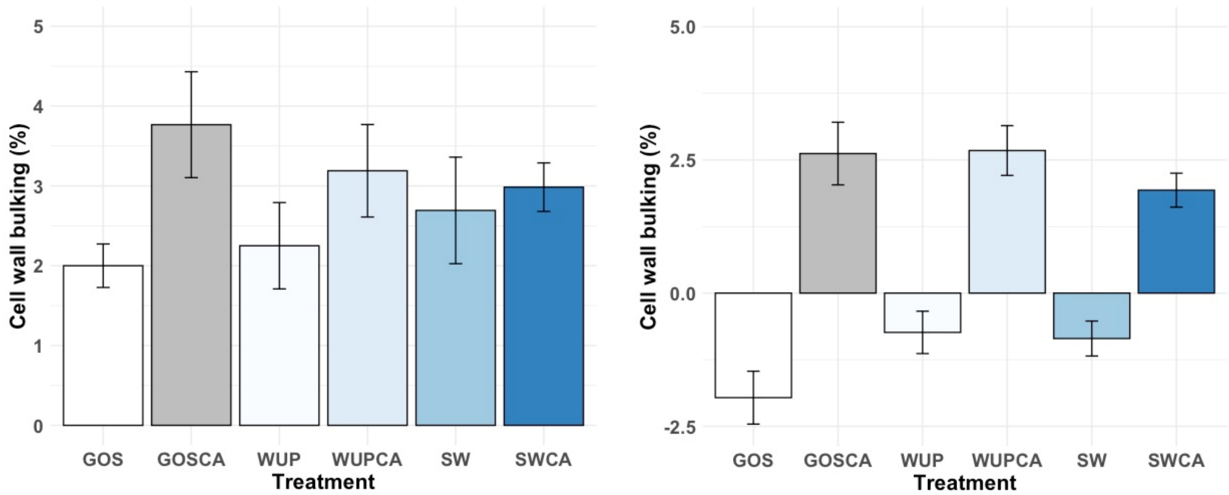

Figure 3: Comparison of cell wall bulking of wood treated with different formulations before (left) and after (right) the leaching test

The CWB after treatment is explained by the diffusion of monomers into the wood cell wall followed by their *in-situ* reaction, leading to a swelling of the cell wall. In this study, the maximum CWB is 4% before the leaching (Fig. 3, left). This value is lower than the CWB of Scots pine sapwood treated with a different modification process (Hötte and Militz 2024). Nevertheless, after the leaching (Fig. 3, right), all the samples that were treated with a formulation containing CA conserved part of the CWB. Those treated with a formulation without CA lost the CWB. The loss of the CWB, in this case, was attributed to the heat, which removed part of the wood cell wall components.

Fig. 4 compares the anti-swelling efficiency (ASE) of modified wood samples before and after water leaching. The left side represents the ASE before water leaching, while the right side shows the ASE after the leaching process. Before water leaching, all the treated wood samples exhibit a high ASE, indicating significant resistance to dimensional swelling for all the treatments. However, After water leaching, wood samples treated with formulations without CA underwent a deep decrease of ASE acquired before water leaching compared to those treated formulations containing CA.

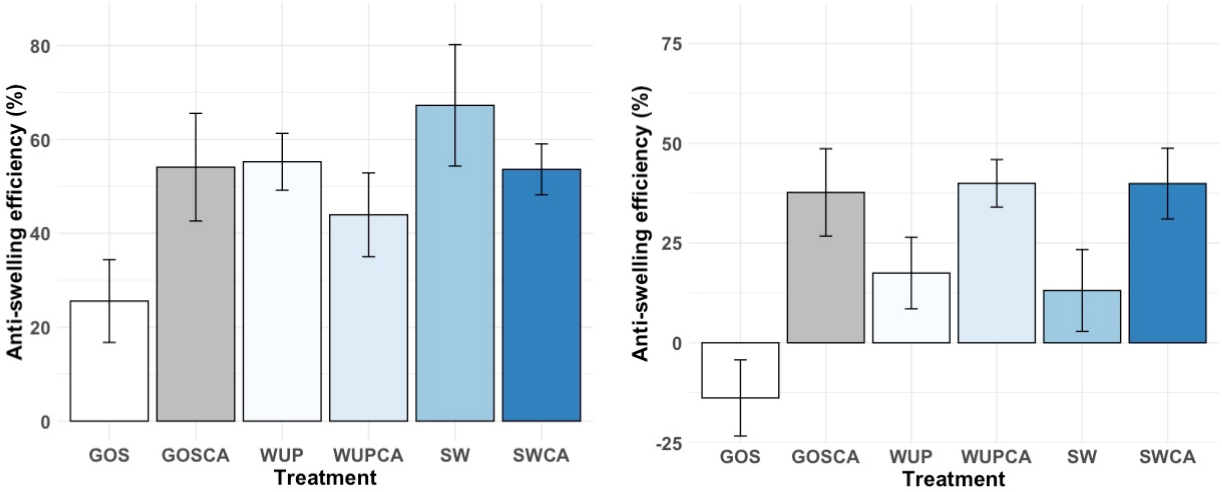

Figure 4: Comparison of anti-swelling efficiency of wood treated with different formulations before (left) and after (right) the leaching test

For the formulations including CA, this suggests that the in-situ polyesterification treatment effectively reduces the hygroscopicity of the wood, limiting its ability to absorb water and swell. For formulations without CA, the calculated ASE could be explained by a physical phenomenon between chemicals and the cell wall matrix, because, after water leaching, a noticeable reduction in ASE is observed, suggesting the removal of the impregnated chemicals. For formulations containing CA, the reduction in ASE is lower than that of formulations without CA. This suggests the leaching of unreacted or weakly bonded chemicals. In fact, some chemicals might not be fully cross-linked within the wood matrix, leading to their removal during water exposure. One possible reason would be the hydrolysis of ester bonds. Since polyesterification involves ester linkages, prolonged water contact could hydrolyze these bonds, reducing treatment effectiveness.

The chemical change during the treatment was analyzed by FTIR (Fig. 5). There was an intensity increase of the band at 1726 cm^{-1} (previously at 1733 cm^{-1} for untreated wood) and the band at 1160 cm^{-1} in samples treated with formulations containing CA, while samples treated with whey ultrafiltration permeate (WUP) and sweet whey (SW) showed a band appearance at 1600 cm^{-1}. However, none of these changes were observed for the samples treated with oligo-saccharide (GOS).

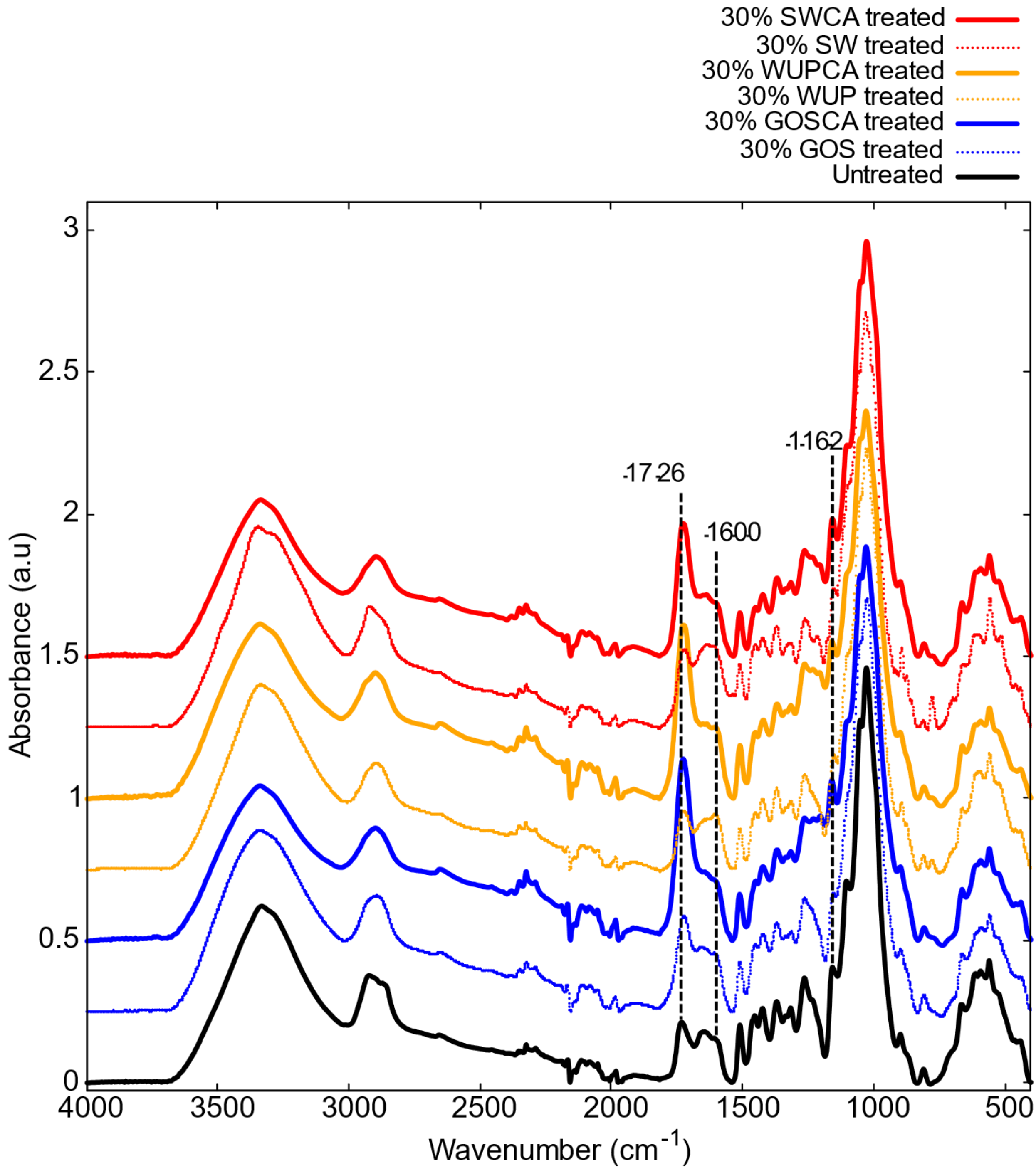

Figure 5: Comparison of FTIR spectra of untreated and Scots pine sapwood treated with different formulations

The intensity increase of bands at 1726 cm^{-1} and 1160 cm^{-1} in samples treated with formulations containing CA suggest the effectiveness of the esterification reaction between CA and the lactose in dairy coproducts (Kurkowiak et al. 2023). However, such changes were not observed for the samples treated with formulations that do not contain CA. Interestingly, for WUP and SW formulations, the appearance of a band at 1600 cm^{-1} (band is absent in GOS formulation) in wood treated samples would indicate a reaction between the components present in WUP and SW. In fact, WUP contains non-protein nitrogen, and SW contains some residual hydrolysable proteins (see Table 1) which can react with lactose through the Maillard reaction (Nielsen et al. 2022). The band at 1600 cm^{-1} is the result of this reaction.

4. EXPECTED PROPERTIES

In general, wood mechanical properties are proportional to its density (Niklas and Spatz 2010). An increase in density leads to an increase in mechanical properties. Scots pine treated with different formulations in this study acquired a significant gain in density, as presented in Fig. 6. However, the wood treatment with CA alone is well-known to make wood brittle (loss in modulus of rupture and impact strength) (Feng et al. 2014). Hence, it is expected that the combination of CA does not negatively affect wood intrinsic mechanical properties, or at least not as strongly as CA alone does. To confirm this hypothesis, tests on the mechanical properties of the modified wood will be carried out.

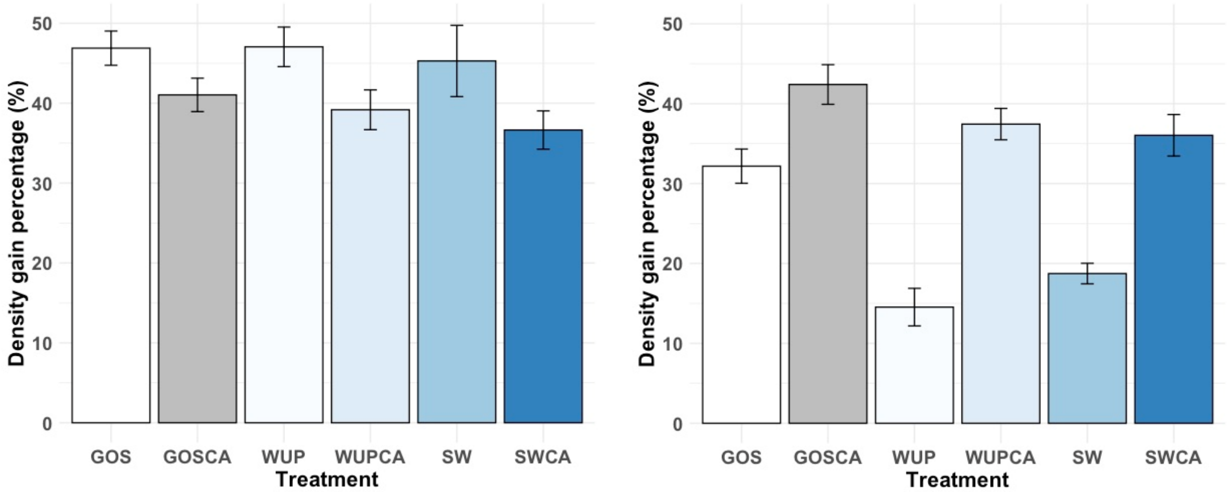

Fig. 6: Comparison of the density gain percentage of wood samples treated with different formulations before (left) and after (right) the leaching test

With regards to the resistance to microorganisms, wood polyesterification is known to reduce the number of moisture absorption sites (Larnøy et al. 2018), thereby lowering the moisture content in the wood. Since many microorganisms require high moisture levels to thrive, this reduction in moisture can help limit their colonization and slow down the onset of decay. The wood will no longer support the colonization of microorganisms as the diffusion of the microorganism enzymes in the wood cell wall is limited due to the decrease of the cell wall moisture uptake (Thybring 2013). This effect could also occure for the wood treated with the methodology described in this study. To confirm, fungal decay tests will be carried out in the near future.

5. CONCLUSION

The goal of this study was to provide a new high-value application for dairy coproducts. The results demonstrate the potential of dairy coproducts, particularly sweet whey, whey ultrafiltration permeate and galactose-oligosaccharides, in combination with CA, for the polyesterification-based modification of Scots pine sapwood. The formulations containing CA penetrated well into the wood leading to a weight gain percentage of around 40% after the water leaching test. The treatment significantly enhances wood dimensional stability, as the anti-swelling efficiency (ASE) remained above 40%, with minimal cell wall bulking loss for CA-based formulations. FTIR analyses confirmed esterification reactions, supporting the formation of durable chemical bonds. These findings highlight the potential of dairy coproducts in wood modification as an environmentally friendly and sustainable alternative for enhancing wood stability, with promising applications in construction and wood-based industries. Future research should focus on optimizing formulation parameters and further evaluate long-term performance in industrial conditions. Ongoing tests will assess if the treatment impacts mechanical properties and contributes to improved fungal resistance due to enhanced density and reduced hygroscopicity. Further research should be carried out to clarify the role of CA in this wood modification technique based on dairy co-products.

6. ACKNOWLEDGEMENT

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