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► **To cite this version:**

Assira Keralta, Jérémy Winninger, Julien Chamberland, Yoshiharu Nishiyama, Véronic Landry. Enhancing moisture exclusion in wood through cell wall bulking via whey ultrafiltration permeate and citric acid modification. *European Journal of Wood and Wood Products*, 2026, 84 (1), pp.4. <10.1007/s00107-025-02344-7>. <hal-05522704>

HAL Id: hal-05522704

<https://hal.science/hal-05522704v1>

Submitted on 22 Feb 2026

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1 **Enhancing Moisture Exclusion in Wood through Cell Wall Bulking via**
2 **Whey Ultrafiltration Permeate and Citric Acid Modification**

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14

15 **Abstract**

16 Wood modification through polyesterification enhances its dimensional stability and
17 reduces its moisture uptake. The efficacy of this modification depends on the placement
18 of the polymeric networks within the wood. When these networks are situated within the
19 cell wall, they cause cell wall bulking, thereby reducing the potential volume for
20 moisture, which improves dimensional stability and water repellence capacity. The anti-
21 swelling efficiency is typically calculated by comparing the dimensions of untreated and
22 treated samples in both saturated and anhydrous states. The definition of the saturated
23 state can influence the anti-swelling efficiency value. While it is established that
24 polyesterification reduces wood moisture uptake, the kinetics of moisture absorption
25 before and after modification have not been extensively studied. In this study, we
26 demonstrate that the modification of four North American wood species with a mixture of
27 whey ultrafiltration permeate and citric acid significantly enhances their physical
28 properties as previously studied, as evidenced by increased anti-swelling efficiency. We
29 also show that while wood modification deeply alters the kinetics of moisture uptake, it
30 effectively reduces overall moisture absorption.

31 **Keywords:** Wood modification; weight gain percentage; cell wall bulking; kinetics of
32 moisture uptake; anti-swelling efficiency; moisture exclusion efficiency.

33

34 Introduction

35 Wood is a hygroscopic material that changes its dimensions in response to moisture con-
36 tent variations driven by ambient relative humidity (Yang et al. 2016). Hemicellulose and
37 lignin, being amorphous, absorb water and swell isotropically, while crystalline cellulose
38 remains dimensionally stable (Niemz et al. 2023; Hill et al. 2024). This results in anisot-
39 ropic dimensional changes upon moisture sorption. Typically, swelling ranges from 6–
40 10% tangentially, 4–5% radially, and only 0.1–0.5% longitudinally (Almeida and
41 Hernández 2006; Patera et al. 2013). Such dimensional changes can cause deformation,
42 cracking, warping, and failure in wood-based products (Glass and Zelinka 2010).

43 Beyond physical deformation, moisture plays a key role in the biological degradation of
44 wood (Hill 2002; Brischke and Alfredsen 2020). Fungal enzymes, typically measuring
45 3.3-7 nm (Hill 2002), cannot penetrate the wood cell wall, which has a pore size of about
46 2 nm in its swollen state (Hill et al. 2005). However, moisture allows some low molecular
47 weight agents to diffuse into the cell wall, initiating decay (Papadopoulos and Hill 2002;
48 Rowell 2006; Rowell et al. 2009). This leads to the breakdown of wood’s structural integ-
49 rity and a loss in mechanical and aesthetic properties. Additionally, water acts as a plasti-
50 cizer, reducing the stiffness and strength of wood as its moisture content increases.

51 Polyesterification involves impregnating wood with reactive chemicals followed by an
52 *in-situ* polymerization to form a network within the cell wall (Hill and Mallon 1998;
53 Berube et al. 2018; Kurkowiak et al. 2022, 2023). Recent studies demonstrated that lac-
54 tose from whey ultrafiltration permeate (whey UF permeate) can react with citric acid
55 inside the wood, improving its physical properties (Cadieux-Lynch et al. 2024). For ef-
56 fectiveness, the impregnated agents must diffuse into the cell wall and form a polymeric
57 network that chemically bonds or entangles with native cell wall polymers (Peeters et al.
58 2018). This network induces bulking, reducing the space available for water sorption.

59 The presence of a fixed polymeric network in the wood cell wall restricts water uptake
60 and impedes the diffusion of degradative agents (Rowell and Banks 1985; Thybring
61 2013). As a result, the wood becomes more dimensionally stable and durable. These im-
62 provements are quantified using Moisture Exclusion Efficiency (MEE), which measures
63 the reduction in water uptake, and Anti-Swelling Efficiency (ASE), which indicates the
64 extent to which dimensional changes are suppressed (Yang 2012; Zelinka et al. 2022;
65 Sargent 2022; Masoumi and Bond 2024a). Both metrics are essential indicators of the
66 efficacy of wood modification.

67 For thermally modified wood, a higher weight loss percentage (WLP) is associated with
68 better MEE and ASE values (Hillis 1984; Kamdem et al. 2002; Bekhta and Niemz 2003;

69 Candelier et al. 2016). In contrast, for acetylated wood, Thybring (2013) observed a lin-
70 ear relationship between weight gain percentage (WGP) and both MEE and ASE. How-
71 ever, polyesterification may not follow this trend. If high molecular weight polymers
72 form outside the cell wall, they may contribute to a high WGP without necessarily im-
73 proving MEE or ASE, highlighting the importance of assessing polymer localization.

74 Numerous studies have explored the moisture uptake kinetics of untreated and chemically
75 modified wood (Droin et al. 1988; Xie et al. 2010; Thybring et al. 2019; Masoumi et al.
76 2024; Hill et al. 2024). For citric acid and whey UF permeate modifications,
77 understanding the mechanisms that reduce moisture sorption rates is essential. Building
78 on the findings of Cadieux-Lynch et al. 2024, this study investigates how a CA/whey UF
79 permeate mixture affects the moisture behavior and physical properties of four wood
80 species. We identify predictors of modification efficiency, assess how ASE is calculated
81 (Sargent 2019; Schorr et al. 2022), and examine the impact of temperature on moisture
82 uptake kinetics.

83 **Materials and methods**

84 **Materials**

85 Citric acid (CA) $\geq 99\%$ was purchased from Sigma Aldrich (Oakville, ON, Canada).
86 Whey ultrafiltration permeate (whey UF permeate) was provided by Agropur (Longueuil,
87 QC, Canada). The whey UF permeate contained 88% lactose, 5.8% ash, 2% non-protein
88 nitrogen on a dry basis, and 4% w/w moisture. Wood samples were sawn to dimensions
89 of $25 \times 25 \times 10 \text{ mm}^3$ (T x L x R) and stored in a conditioning room (41% RH and 21 °C).
90 The wood moisture content equilibrated to around 8%. Four species were treated: two
91 hardwoods, sugar maple (*Acer saccharum* L., SM) and willow (*Salix spp* IBID., W), and
92 two softwoods: black spruce (*Picea mariana* M., BS) and white pine (*Pinus strobus* L.,
93 WP), with ten samples per species. These species were chosen for their anatomical
94 features, their impregnability differences, and their abundance in Canada or their market
95 value. The sampling could contain a mixture of sapwood and heartwood. Two methods
96 were tested to understand their effect on the anti-swelling efficiency and moisture
97 exclusion efficiency. For each method, ten samples per species were used. The samples
98 used for the equilibration in saturated vapour method were separated from those used for
99 the water suction method.

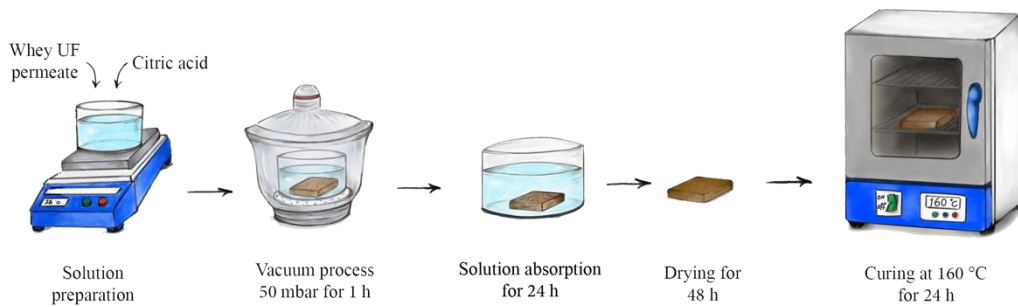
100 **Whey-CA Solution preparation**

101 The aqueous mixture solution of CA and whey UF permeate was prepared as follows:
102 39.6 g of CA was weighed into a beaker, and 100 g of water was added. The solution was
103 stirred at 50 °C. Once the CA was completely dissolved, 20 g of whey UF permeate was
104 added to the solution. This corresponds to the molar ratio of hydroxyl groups to CA
105 carboxyl groups of 1:1. The concentration of the solution was 37% and the solution

106 density was 1.20 ± 0.01 g/ml. The mixture was stirred at 50 °C until the complete
 107 dissolution of whey UF permeate. This solution was named whey-CA. The solution was
 108 stored at 5 °C.

109 **Wood treatment**

110 We immersed ten wood block samples per species in the whey-CA solution, with a
 111 weight on top to keep the samples fully submerged. After leaving under controlled
 112 vacuum of 50 mbar for 1h, we kept the immersion at ambient conditions for 24 hours. We
 113 weighed the impregnated samples after blotting the excess solution with paper and
 114 measured their dimensions. The treatment process is summarized in Fig. 1.



115

116 **Fig. 1** Wood treatment process

117 **Physical characterization**

118 *Solution uptake*

119 The solution uptake, SU, is defined as the amount of whey-CA solution absorbed by a
 120 sample. The SU were compared to the theoretical maximum water uptake (WU_{max}) and to
 121 the theoretical maximum whey-CA uptake (SU_{max}). SU was calculated according to Eq.
 122 1, and WU_{max} and SU_{max} according to Eq. 2 (Glass and Zelinka 2010) and Eq. 3,
 123 respectively.

$$SU = \frac{m_1 - m_0}{m_0} \times 100 \quad (1)$$

124 Where m_1 is the sample's weight after the impregnation and m_0 is the oven-dry weight of
 125 the sample before the impregnation.

126 The maximum water uptake can be estimated as:

$$WU_{max} = \frac{1.54 - G_b}{1.54G_b} \times 100 \quad (2)$$

127 Where 1.54 is the specific gravity of the wood cell wall, and G_b is the wood basic specific
 128 gravity:

129 $G_b = \frac{m_0}{V_1}$

130 Where V_1 is the water-swollen wood volume. Similarly, taking the density of
 131 solution, d_{sol} into account, the maximum whey-CA solution can be estimated as:

$$SU_{max} = \quad (3)$$

132 Here, we assume that the wood swelling by the solution is the same as by water, and the
 133 biomass occupies the portion corresponding to $m_0/1.54$, which is not accessible to the
 134 solution volume. The remaining volume, $V_1 - (m_0/1.54)$, is then assumed to be filled by
 135 the solution that keeps the bulk density d_{sol} .

136 *Weight gain percentage*

137 Weight gain percentage (WGP) is the increase in the total amount of solid compounds
 138 remaining in the wood samples after curing at 160°C.

$$WGP = \frac{m_2 - m_0}{m_0} \times 100 \quad (4)$$

139 Where m_2 is the oven-dry weight of samples after curing at 160 °C.

140 WGP was then compared to the theoretical weight gain percentage (TWGP) (Hötte and
 141 Militz 2024) estimated as:

$$TWGP = \frac{m_{chemicals}}{m_0} \times 100 \quad (5)$$

142 Where $m_{chemicals}$ is the weight of the chemical in the 37% of whey-CA solution, assuming
 143 that only water is volatile at 160°C

144 *Cell wall bulking*

145 The diffusion of whey UF permeate and CA followed by polycondensation in the cell
 146 wall leads to a volume increase characterized by cell wall bulking (CWB) expressed as:

$$CWB = \frac{A_2 - A_0}{A_0} \times 100 \quad (6)$$

147 Where A_2 is the dry cross-sectional area of the sample after curing at 160 °C, and A_0 is
 148 the sample's dry cross-sectional area before the treatment, calculated from the dimensions
 149 in the radial-tangential plane.

150 CWB results were compared to the theoretical cell wall bulking (TCWB) estimated as:

$$TCWB = \frac{A_{swollen} - A_0}{A_0} \times 100 \quad (7)$$

151 Where $A_{swollen}$ is the swollen cross-sectional area of the sample after impregnation with
 152 the whey-CA solution. We assumed that at this state, the wood cell wall was expanded to
 153 its maximum volume.

154 *Moisture exclusion efficiency*

155 The moisture exclusion efficiency (MEE) was evaluated as the change in the water up-
 156 take behaviour due to the treatment. We used two methods to compare the effects of
 157 these methods on the results and assess the mechanism behind wood modification with
 158 the mixture of whey UF permeate and CA. Ten wood samples per species were used for
 159 each method.

160 *a) Equilibration in saturated vapour*

161 The samples were conditioned in a desiccator containing distilled water (100% RH), and
 162 their mass was measured over time. We consider the moisture content to have reached
 163 equilibrium when the mass variation was less than 1% over one week. This was the
 164 equilibration in saturated vapour method.

$$EMC_2 = \frac{m_{t2} - m_2}{m_2} \times 100$$

165 The moisture exclusion efficiency method (MEE) is defined as:

$$MEE = \frac{EMC_0 - EMC_2}{EMC_0} \times 100 \quad (8)$$

166 *b) Water suction*

167 In the water suction method, the equilibrium moisture content prior to treatment (MC'_0)
 168 and post-treatment (MC'_2) were calculated using the same approach as in the
 169 equilibration in saturated vapour method, with the only difference being the saturated
 170 weights m'_{t1} and m'_{t2} . In this case, m'_{t1} and m'_{t2} were obtained through vacuum
 171 impregnation of distilled water. The corresponding MEE' values were then calculated
 172 according to Eq. 9.

$$MEE' = \frac{MC'_0 - MC'_2}{MC'_0} \times 100 \quad (9)$$

173 MEE' (suction method) is used here to establish the relationship between MEE and WGP
 174 or MEE and CWB in comparison with MEE (equilibration in saturated vapour method)

175 *Anti-swelling efficiency*

176 Similar to the MEE calculations, the Anti-Swelling Efficiency (ASE) was determined
177 using two methods of water uptake. This allowed to better understand how the wood
178 treatment with a mixture of citric acid and whey UF permeate alters the wood-moisture
179 relationship and the result on the related physical properties. Ten samples from each
180 species were analyzed using both methods.

181 a) *Equilibration in saturated vapour*

182 ASE measurement using the equilibration in saturated vapour method followed these
183 steps: The volumes of the same samples were recorded in their anhydrous state (V_0) and
184 under vapor saturation (V_{sat0}), where the moisture content (MC) change over time was
185 less than 1% over the course of a week for both untreated and treated samples. ASE
186 values were calculated using Eq. 10.

$$ASE = \frac{S_0 - S_2}{S_0} \times 100 \quad (10)$$

187 Where: S_2 and S_0 were the volumetric swellings after and before treatment, respectively,
188 obtained by vapor absorption.

$$S_i = \frac{V_{sati} - V_i}{V_i} \times 100 (i = 0,2)$$

189 b) *Water suction*

190 In the water suction method, ASE' was calculated similarly to the equilibration in
191 saturated vapour method, with the only difference being that V_{sat2} was obtained by
192 vacuum impregnation of the samples with distilled water. ASE' values were calculated
193 according to Eq. 11.

$$ASE' = \frac{S'_0 - S'_2}{S'_0} \times 100 \quad (11)$$

194 Where: S'_2 and S'_0 were the volumetric swellings after and before treatment, respectively,
195 obtained by vacuum impregnation with water.

$$S'_i = \frac{V'_{sati} - V_i}{V_i} \times 100 (i = 0,2)$$

196 For all these physical properties, the mean values and error bars are presented in the
197 results.

198 *Optical microscopy images*

199 To prepare cross-sectional samples, an automatic rotary microtome (HistoCore AUTO-
200 CUT, Leica Biosystems, Buffalo Grove, USA) was used to cut 20 μm thick sections.
201 These sections were observed using a VHX-7000 digital microscope (Keyence Co. Ltd.),
202 with full ring lighting for dark field observation, which is optimal for heterogeneous
203 samples.

204 **Measurement of the kinetics of the moisture uptake before and after the treatment**

205 The kinetics of moisture uptake were measured as follows: Samples were oven-dried at
206 103 $^{\circ}\text{C}$ for 48 hours and then cooled in a silica gel desiccator. They were weighed using a
207 Sartorius Balance with 1 mg precision, then conditioned in a desiccator over distilled
208 water, and placed in a controlled chamber (20 $^{\circ}\text{C} \pm 1$). The moisture content at a given
209 time (MC_t) was calculated using Eq. 12 and plotted against the square root of time to
210 determine absorption kinetics.

$$MC_t = \frac{m_t - m_0}{m_0} \times 100 \quad (12)$$

211 Where m_t is the sample weight at a given time, and m_0 is the sample weight before
212 conditioning. For untreated samples, m_0 represents the oven-dried weight without
213 treatment, and for water-treated and whey-CA-treated samples, m_0 refers to the oven-
214 dried weight after impregnation and curing at 160 $^{\circ}\text{C}$.

215 Ten samples (25 \times 25 \times 10 mm^3 , T \times L \times R) per species per treatment were used for this
216 test, with results presented as mean values with standard deviations.

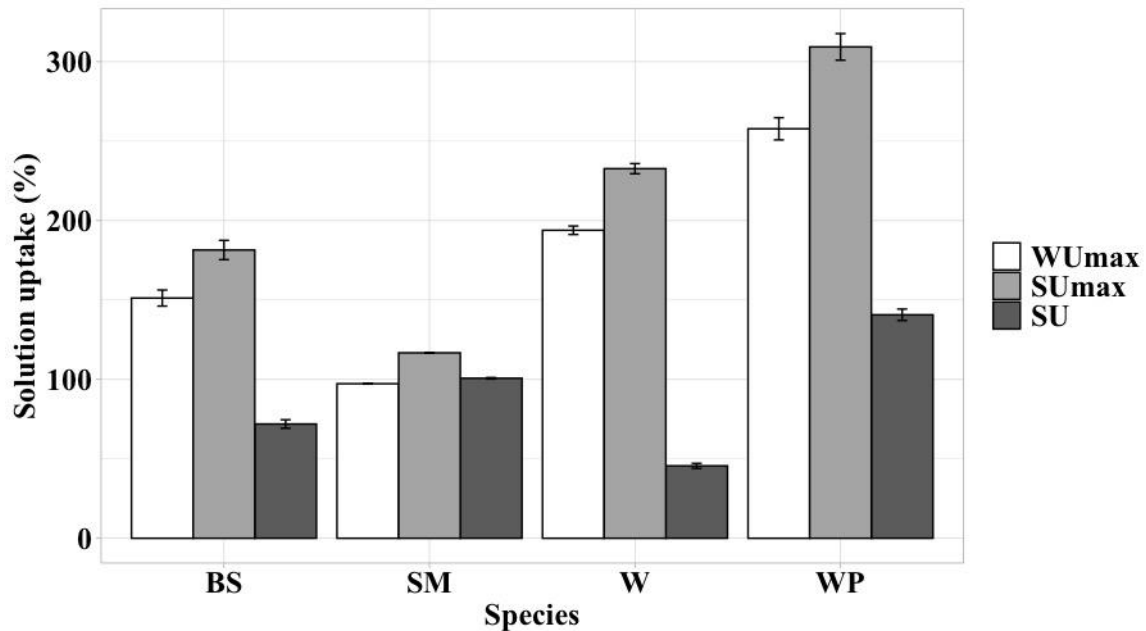
217 **Results and Discussion**

218 **Physical properties**

219 *Solution uptake and microscopic image observation*

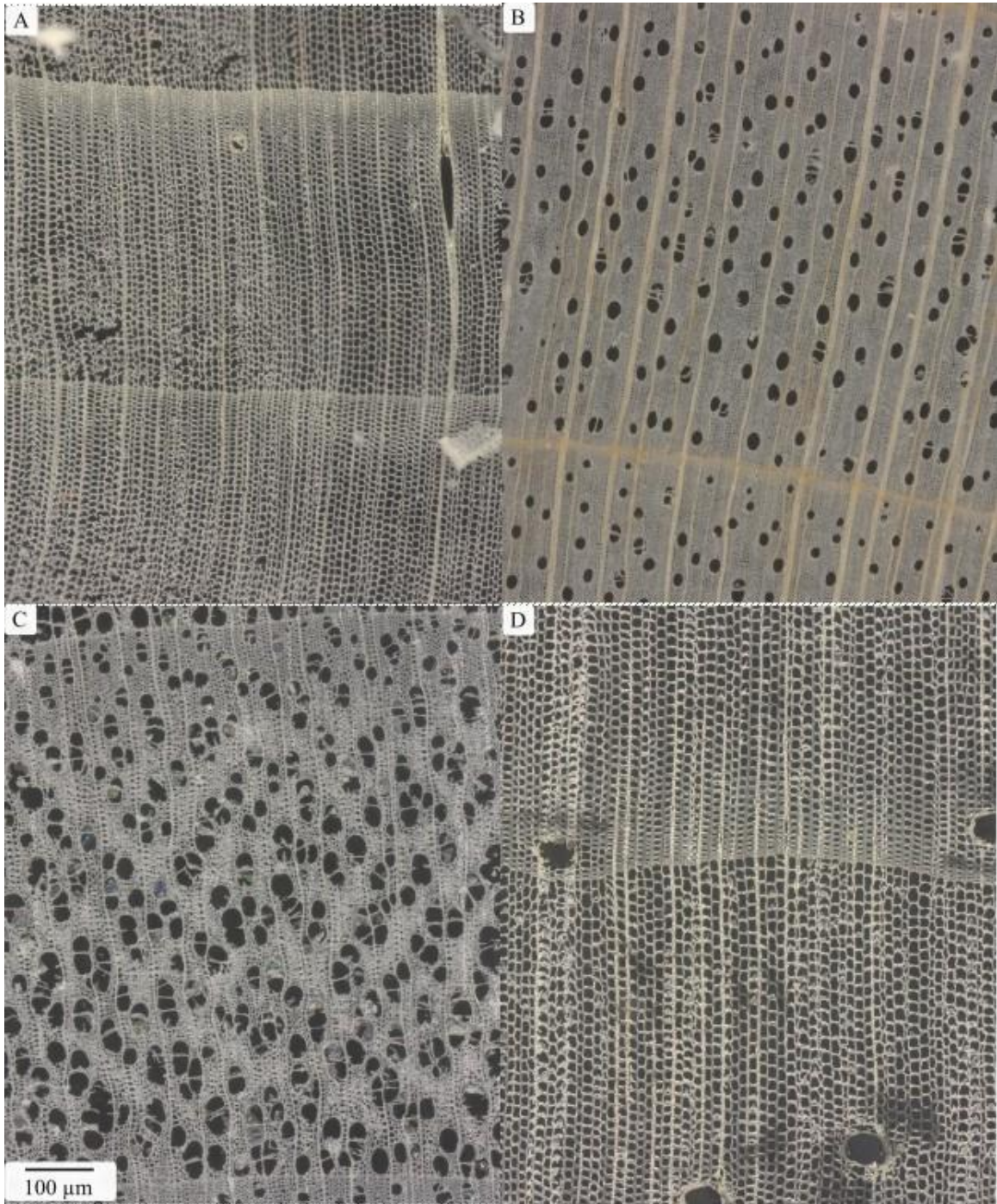
220 Figure 2 shows the solution uptake (SU), theoretical maximum water uptake (WU_{max}),
221 and theoretical maximum whey-CA uptake (SU_{max}) of each species. SU_{max} is lower than
222 the theoretical SU_{max} for all species, but with difference among species reflecting the ana-
223 tomical structure and surface chemistry on the impregnation rate. SM showed a SU close
224 to the SU_{max} . This can be explained by the significant volumetric swelling characteristic
225 of SM (Mantanis et al. 1994; Hernández 2007; Schroeder 2007). The diffuse porous
226 structure of SM cell wall, as seen in Fig. 3, also contributes to easier impregnation of SM.
227 The solution uptake of WP was highest among the four species because of the low den-
228 sity, but the solution occupied only half of the available space in the wood. This same
229 finding was also seen in BS, where the solution occupied just half of the space. Micro-
230 scopic analysis, as shown in Fig. 3, supports this observation. On the other hand, Willow

231 displayed the lowest solution uptake and the lowest proportion of void occupied by solu-
232 tion. Willow wood is known to have good impregnability (Boever et al. 2007), but our
233 sample might have been duramen which less impregnable than the sapwood.



234

235 **Fig. 2** Comparison of maximum water uptake (WUmax), theoretical maximum uptake of
236 whey-CA solution (SUmax) and measured whey-CA solution uptake (SU) of different
237 species.



238

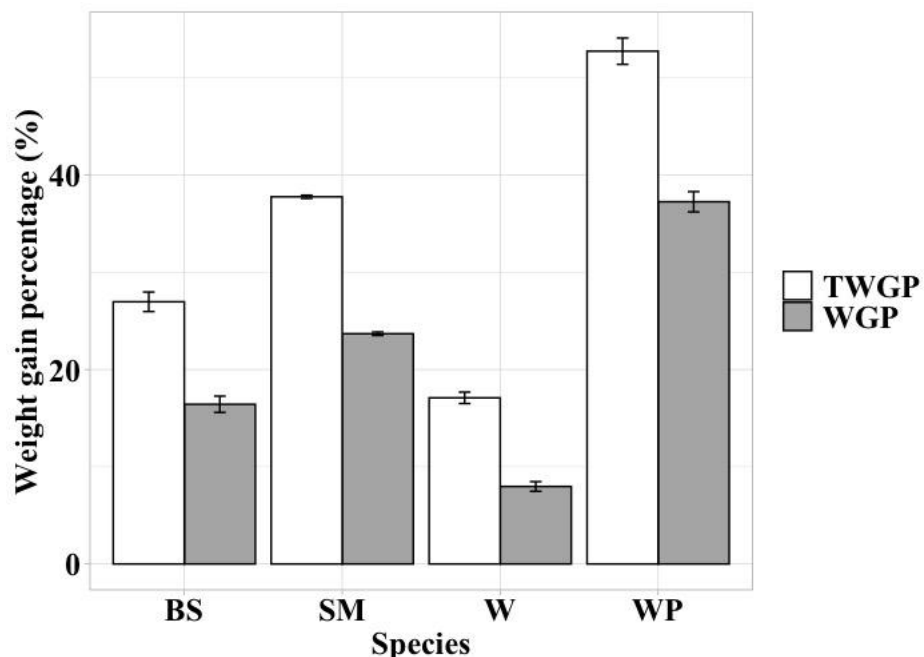
239 **Fig. 3** Optical microscopic images of untreated: A)black spruce (BS), B)sugar maple
240 (SM), C)willow (W), and D)white pine (WP). Magnification: $\times 100$.

241

242 *Weight gain percentage and cell wall bulking*

243 The weight gain percentage (WGP) was lower than expected (TWGP) from the solution
244 uptake, typically in the range of 8 to 38% (Fig. 4). Part of chemicals from the wood as
245 volatile components during curing as observed by (Hötte and Militz 2024). The WGP
246 follows a similar trend to the SU across all species. Species with higher SU also show
247 higher WGP values. WP exhibits the highest WGP, followed by SM, with BS and W dis-
248 playing the lowest values.

249

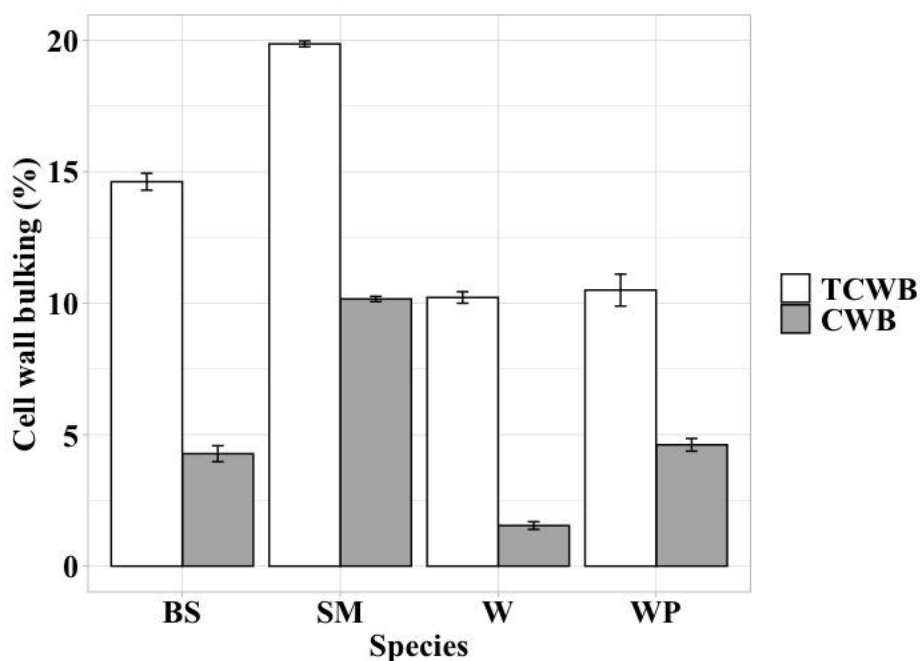


250

251 **Fig. 4** Comparison of theoretical weight gain percentage (TWGP) and measured weight
252 gain percentage (WGP) of different species treated with whey-CA solution.

253 Similarly to WGP, the experimental cell wall bulking (CWB) is lower than the theoretical
254 cell wall bulking (TCWB) (Fig. 5), but SM and WP achieved almost half the TCWB
255 value. The theoretical bulking can be achieved if the water molecules in the cell wall are
256 replaced by the non-volatile component of the solution in the thermal process after im-
257 pregnation. If the weight gain percentage completely contributes to the bulking by mi-
258 grating into the cell wall, the bulking should be the theoretical one at a weight gain of 45
259 % if we consider the water fiber saturation point of 30% and a specific gravity of 1.5 for
260 the non-volatile component of whey solution. The level of impregnation is never
261 achieved, but if we consider the cell wall bulking to be linear against weight gain, SM
262 shows about half the theoretical bulking (20%) with 23 % weight gain, so the non-volatile
263 components should be essentially present in the cell wall. On the contrary, despite the
264 close to 40% weight gain, WP achieved less than half of the maximum cell wall bulking,

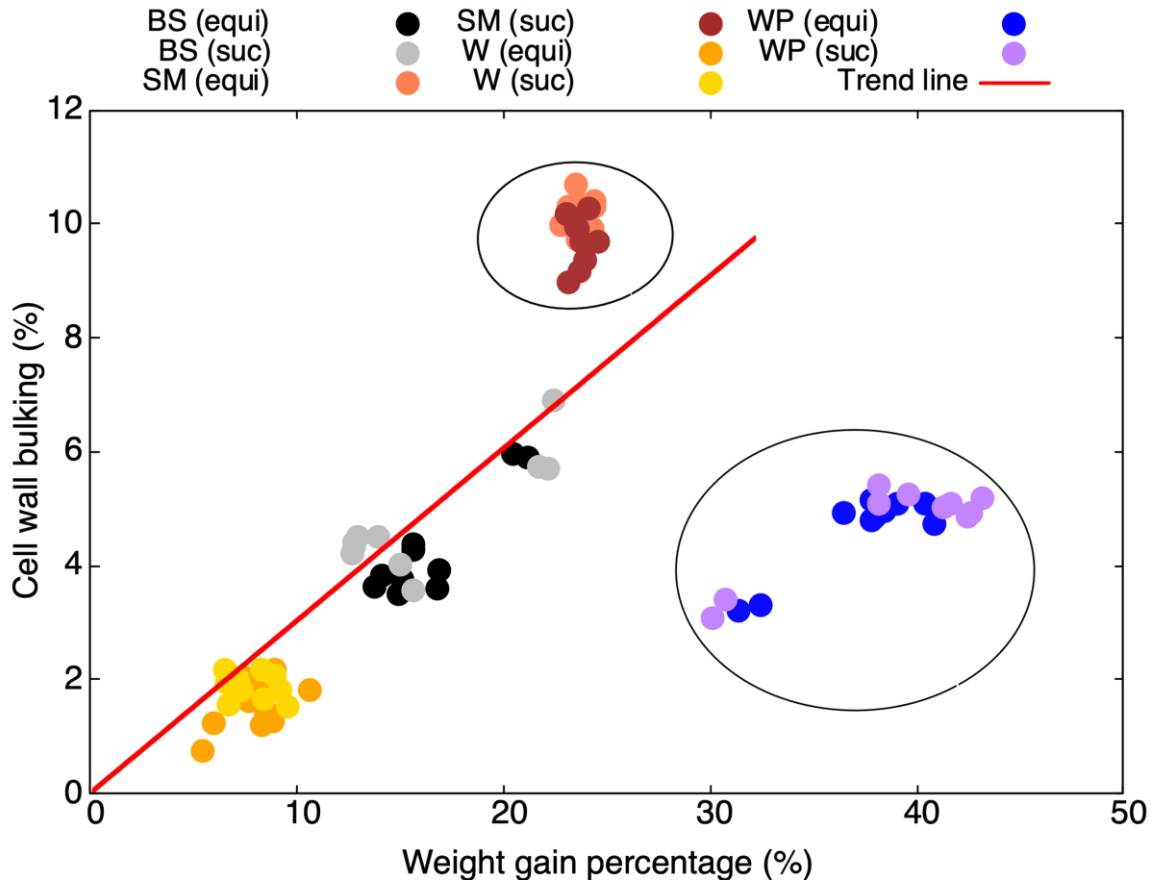
265 suggesting that half of the non-volatile compounds remain inside lumens. This was also
266 observed by Peeters et al. (2018) when they modified pine by the Maillard reaction.



267

268 **Fig. 5** Comparison of theoretical cell wall bulking (TCWB) and measured cell wall
269 bulking (CWB) of different species treated with whey-CA solution.

270 Fig. 6 presents the relationship between WGP and CWB of the two groups of the
271 samples. A linear correlation is observed for SM, BS, and W samples, suggesting that the
272 increase in WGP translates proportionally to CWB for these species. However, WP
273 deviates from this linear trend. This deviation likely indicates that a large portion of the
274 reaction products in WP does not integrate into the cell wall, but rather remains in other
275 structures, such as the lumens or resin canals.

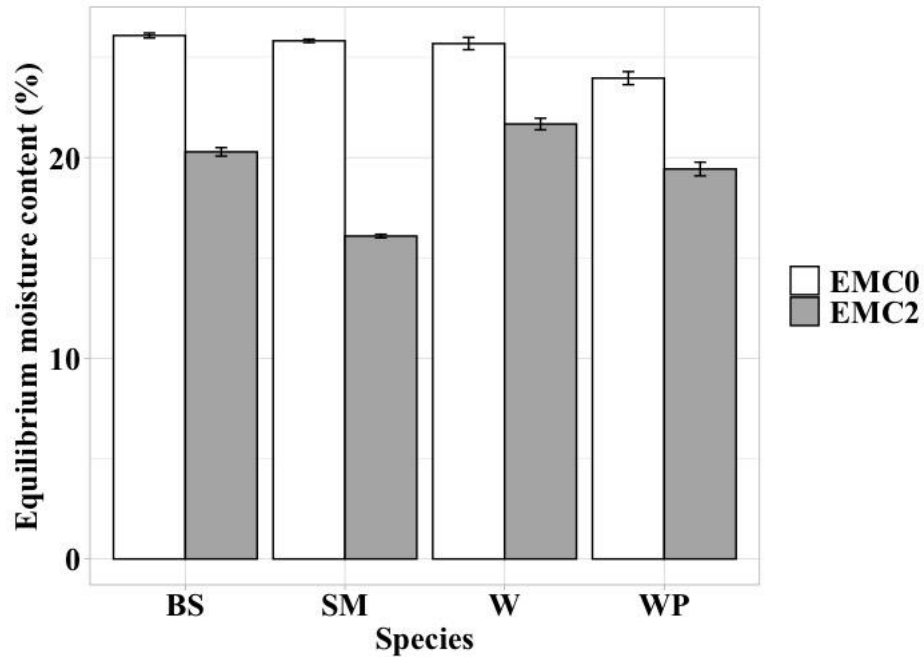


276

277 **Fig. 6** Relationship between WGP and CWB obtained by equilibration in saturated
 278 vapour (equi) and water suction (suc) methods..

279 *Equilibrium moisture content and moisture exclusion efficiency calculated by*
 280 *equilibration in saturated vapour method*

281 The moisture content and the moisture exclusion efficiency (MEE) from the equilibration
 282 in saturated vapour method are presented in Fig.7 and Fig. 8, respectively. This method
 283 was selected to assess these properties here because it (self-vapour absorption) allows us
 284 to understand how the treatment alters the hygroscopic behaviour of the cell wall. At this
 285 condition, moisture would mostly accumulate into the cell wall rather than lumens. The
 286 modification process effectively reduced the moisture uptake for all four species (Fig. 7).
 287 The reduction was most pronounced in SM, followed by BS, WP, and W, following the
 288 same trend observed for the CWB. In this method, moisture tends to accumulate within
 289 the cell wall, and since SM exhibited the highest CWB, it also experienced the most
 290 significant reduction in the available volume for moisture uptake. This trend was
 291 consistently observed across the other species as well.

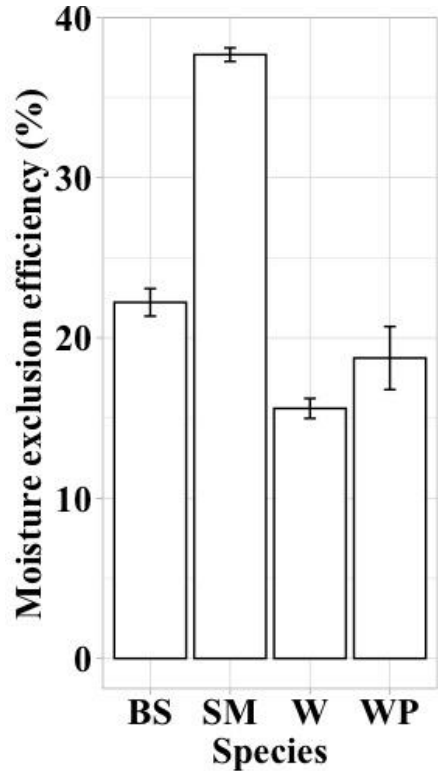


292

293 **Fig. 7** Comparison of equilibrium moisture content before treatment (EMC0) and
 294 equilibrium moisture content after treatment (EMC2) obtained by equilibration in
 295 saturated vapour method of different species treated with whey-CA solution.

296

297 The reduction in moisture uptake after modification resulted in a MEE exceeding 15%
 298 (Fig. 8), even for W, which had the lowest WGP. The highest MEE was observed in SM,
 299 which showed a bulking of about 50% of the maximum value.



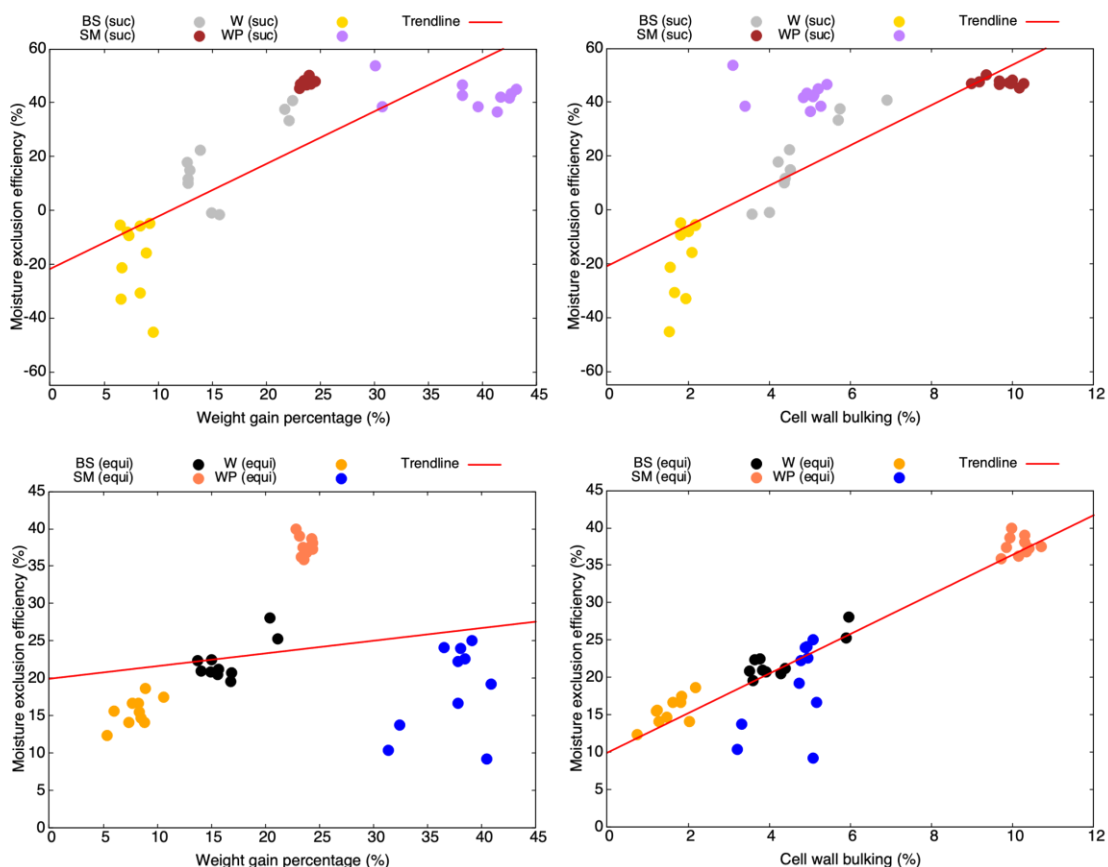
300

301 **Fig. 8** Moisture exclusion efficiency obtained by equilibration in saturated vapour
 302 method of different species treated with whey-CA solution.

303 In many modification processes, WGP correlates with the MEE trend. For example, in
 304 wood acetylation, a higher WGP typically leads to a higher MEE, which is explained by
 305 the esterification reaction with the cell wall free OH groups and the CWB (Awais et al.
 306 2022). This occurred because the small acetic anhydride penetrates into the wood cell
 307 wall. On the other hand, in thermally treated wood, a higher weight loss results in an in-
 308 creased MEE. In this case, it is generally explained by the loss of OH groups (hemicellu-
 309 lose and cellulose) and lignin crosslinking during thermal treatment (Čermák et al. 2015;
 310 Hill et al. 2021; Masoumi and Bond 2024b). However, wood polyesterification might
 311 behave differently since the polyesterification can occur also in the lumen, in which case
 312 it does not affect the cell wall behavior.

313 Fig. 9 illustrates the relationships between WGP, MEE, and CWB for both the equilibra-
 314 tion in saturated vapour and water suction methods of MEE calculation. In both methods,
 315 no clear relationship was found between WGP and MEE. In contrast, a strong linear rela-
 316 tionship was observed between CWB and MEE for both methods. This finding suggests
 317 that, in the case of wood polyesterification, CWB is the key factor for predicting MEE, as
 318 some products formed outside the cell wall do not contribute to enhanced wood proper-
 319 ties. Additionally, the relationship between WGP and MEE in the water suction method

320 was weaker compared to the equilibration in saturated vapour method, raising questions
 321 about the evaluation of MEE based on the suction method for wood polyesterification.



322

323 **Fig. 9** Comparison of the relationship between WGP and MEE for (A): water suction
 324 (suc) method and (C): equilibration in saturated vapour (equi) method, and CWB and
 325 MEE for (B): water suction method and (D): equilibration in saturated vapour method.

326 *Anti-swelling efficiency*

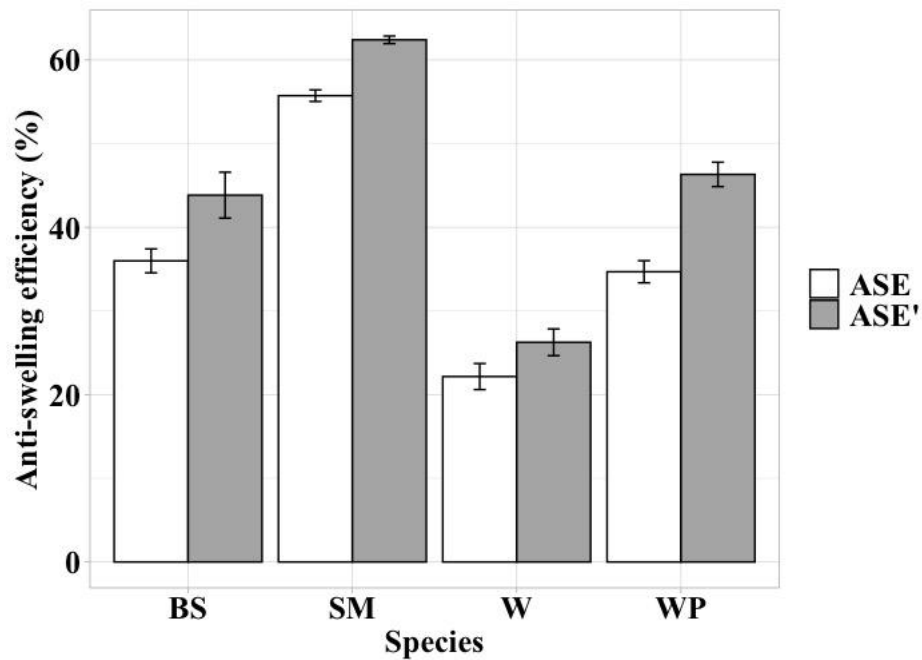
327 Fig. 10 presents the ASE, calculated using the equilibration in saturated vapour method,
 328 and ASE', calculated using the water suction method. A comparison of SU, WGP, and
 329 CWB for the samples used in both ASE calculations is provided in Table 1. The values of
 330 SU, WGP, and CWB are fairly consistent across samples, allowing for a meaningful
 331 comparison between ASE and ASE'.

332 **Table 1** Solution uptake, weight gain percentage, and cell wall bulking of samples used to
 333 calculate anti-swelling efficiency and moisture exclusion efficiency by water suction and
 334 equilibration in saturated vapour methods.

	ASE (equilibration in saturated vapour)			ASE' (water suction)		
	SU (%)	WGP (%)	CWB (%)	SU (%)	WGP (%)	CWB (%)
BS	72 ± 8	16 ± 3	4 ± 1	71 ± 14	16 ± 4	5 ± 1
SM	101 ± 1	24 ± 1	10 ± 0	101 ± 2	24 ± 1	10 ± 0
W	46 ± 5	8 ± 2	2 ± 1	46 ± 4	8 ± 1	2 ± 0
WP	141 ± 10	37 ± 3	5 ± 1	147 ± 15	39 ± 5	5 ± 1

335

336



337

338 **Fig. 10** Comparison of anti-swelling efficiency obtained by equilibration in saturated
 339 vapour method (ASE) and anti-swelling efficiency by water suction method (ASE').

340

341 For all species, ASE was lower than ASE', indicating that the evaluation method has a
 342 significant impact. $ASE = \frac{Su - St}{Su} \rightarrow ASE = 1 - \frac{St}{Su} \wedge Su = \frac{V_{sat} - V_{anh}}{V_{anh}} \rightarrow Su = \frac{V_{sat}}{V_u} - 1$

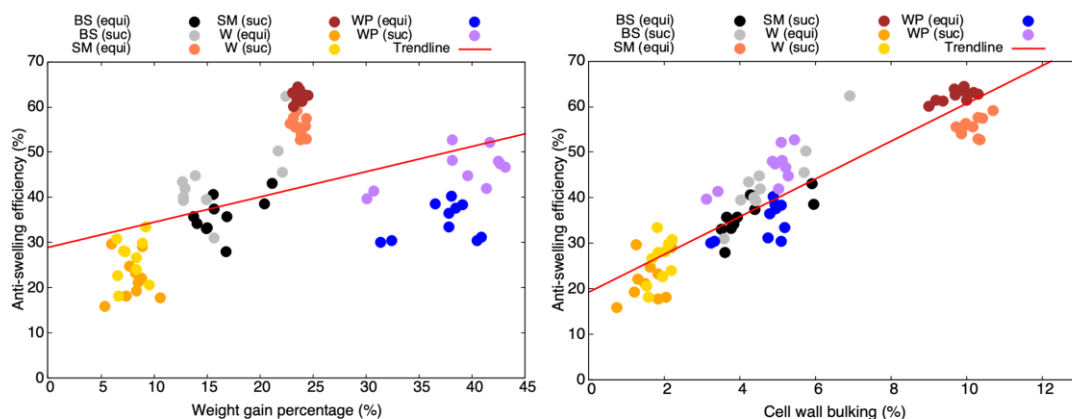
343 The discrepancy between ASE and ASE' is due to the difference in limit moisture content
 344 in the swollen state. For ASE, the moisture content was below the fiber saturation point
 345 (FSP) (<25% for all species), and thus showed lower swelling compared to the suction
 346 method, in which the moisture content exceeds the FSP (>50% for all species, and thus
 347 probably attained the maximum swelling (Table 2).

348 **Table 2** Comparison of the cross-sectional swellings obtained by equilibration in
 349 saturated vapour and water suction methods.

Samples	Cross-sectional swelling by equilibration in saturated vapour method	Cross-sectional swelling by water suction method
BS	12.7 ± 0.6	15.3 ± 1.0
SM	17.1 ± 0.4	20.6 ± 0.3
W	10.6 ± 2.0	11.0 ± 0.7
WP	9.2 ± 1.4	11.1 ± 1.7

350

351 Similar to the trend observed with MEE, CWB proves to be a better predictor of dimen-
 352 sional stability (presented by ASE) compared to WGP, as shown in Fig. 11. For all spe-
 353 cies, a strong linear relationship was found between CWB and ASE. In contrast, no linear
 354 relationship was observed between WGP and ASE.



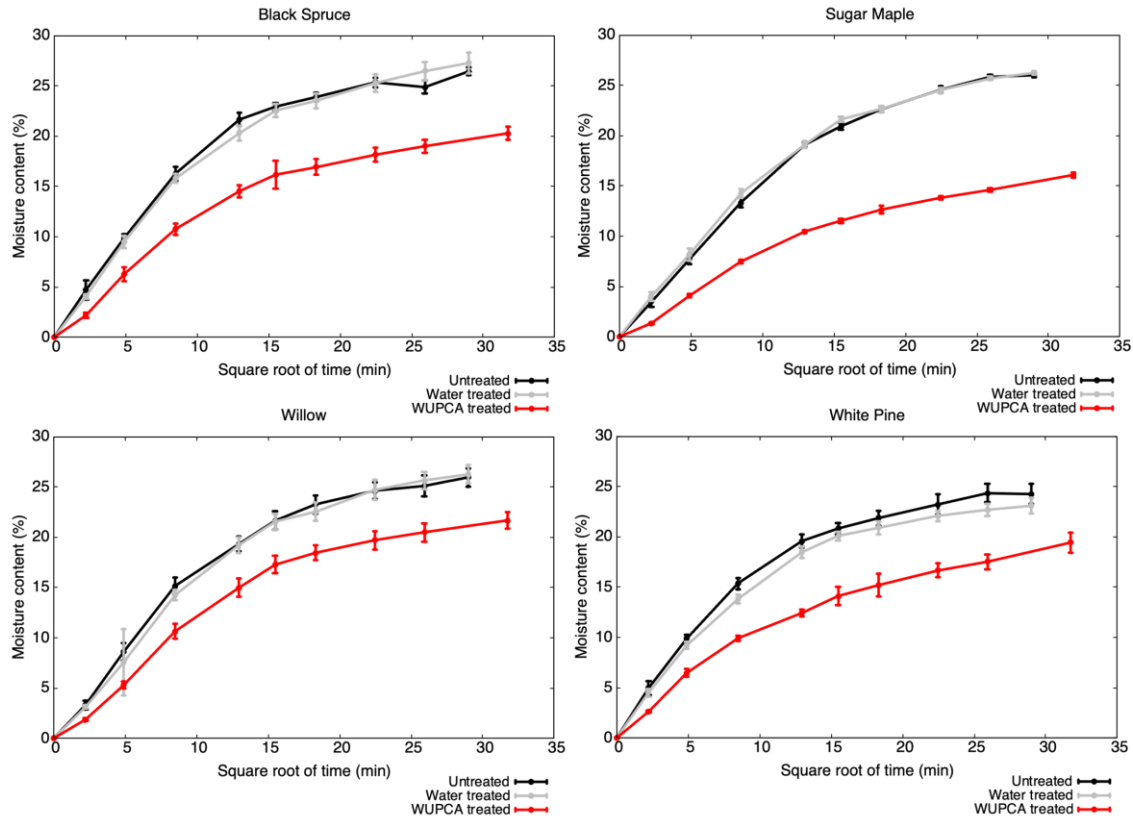
355

356 **Fig. 11** Comparison of the relationship between (A): WGP and ASE and (B): CWB and
 357 ASE obtained by equilibration in saturated vapour (equi) and water suction (suc)
 358 methods.

359 **Kinetics of moisture absorption before and after the treatment**

360 The moisture content of the samples was plotted against the square root of time to ana-
361 lyze the kinetics of moisture uptake, as shown in Fig. 12. The trend was similar to that
362 observed by Masoumi et al. (2024), Thybring et al. (2019), Droin et al. (1988), and Xie
363 et al. (2010). Before treatment, the samples rapidly absorbed moisture, and after ap-
364 proximately 200 hours, the absorption rate decreased over time. The moisture uptake lev-
365 eled off at approximately 25% for all four species. After water treatment, the overall
366 trend remained unchanged, with no significant reduction in total moisture uptake, show-
367 ing that the curing temperature did not (or lightly) affect the moisture absorption sites in
368 wood. Essoua Essoua et al. 2016 did not observe a change in the hygroscopic behaviour
369 of white pine after heating at 180 °C.

370 However, following treatment with whey-CA (whey UF permeate and CA), the moisture
371 uptake kinetics were decreased compared to the untreated samples for all the species. The
372 total moisture content of whey-CA-treated wood was lower than that of untreated and
373 water-treated wood for all species. SM exhibited the greatest reduction in the equilibrium
374 moisture content, followed by WP and BS. The results indicate that the whey-CA treat-
375 ment deeply altered the moisture absorption rate and reduced the total moisture content,
376 suggesting that wood modification using whey UF permeate and CA blocked some mois-
377 ture absorption sites, impacting the moisture uptake kinetics. The location of the poly-
378 meric network formed in the wood appeared to play a key role in post-treatment moisture
379 content. For example, although WP had a higher WGP than SM, the moisture content of
380 SM was lower than that of WP after treatment. This discrepancy suggests that in WP, the
381 impregnated materials did not fully diffuse into the cell wall, whereas in SM, a larger
382 proportion of the materials penetrated the cell wall.



383

384 **Fig. 12** Comparison of the kinetics of moisture uptake before and after the treatment with
 385 whey-CA solution of different species.

386 **Conclusion**

387 Wood modification using whey UF permeate, and citric acid significantly enhanced the
 388 physical properties of all four studied species. Notably, wood density increased consid-
 389 erably across all species. The modification led to improved moisture exclusion efficiency
 390 (MEE) and enhanced dimensional stability. Unlike other wood modification techniques,
 391 such as acetylation or thermal treatment, the weight gain percentage (WGP) was not a
 392 good indicator of MEE and anti-swelling efficiency (ASE). Instead, the results suggest
 393 that cell wall bulking (CWB) is a better predictor of the modification's effectiveness, as
 394 this process results in the formation of bulk compounds, some of which are located out-
 395 side the cell wall. The kinetics of water uptake for the samples treated with water alone
 396 mirrored those of untreated samples, indicating that water treatment did not affect the rate
 397 of moisture diffusion. However, while the moisture uptake trend in whey-CA-treated
 398 samples followed that of untreated samples, their total moisture content was significantly
 399 lower. This suggests that the whey-CA treatment altered the kinetics of moisture uptake
 400 and reduced the limit moisture content, likely due to the presence of whey-CA conden-

401 sates in the cell wall that translates to bulking, which decreased the room for moisture
402 absorption in wood.

403 **Acknowledgments**

404 We would like to express our gratitude to the Fonds de Recherche du Québec – Nature et
405 Technologies (FRQNT) (2023-NOVA-315224) and the Natural Sciences and
406 Engineering Research Council of Canada (NSERC) (ALLRP 571482-21) for their
407 financial support. Our thanks also extend to Luc Germain of the Renewable Materials
408 Research Center for his assistance with sample preparation.

409 **Reference**

- 410 Almeida G, Hernández RE (2006) Changes in physical properties of tropical and
411 temperate hardwoods below and above the fiber saturation point. *Wood Sci Technol*
412 40:599–613. <https://doi.org/10.1007/s00226-006-0083-8>
- 413 Awais M, Altgen M, Mäkelä M, et al (2022) Quantitative prediction of moisture content
414 distribution in acetylated wood using near-infrared hyperspectral imaging. *J Mater Sci*
415 57:3416–3429. <https://doi.org/10.1007/s10853-021-06812-2>
- 416 Bekhta P, Niemz P (2003) Effect of High Temperature on the Change in Color,
417 Dimensional Stability and Mechanical Properties of Spruce Wood. *Holzforschung*
418 57:539–546. <https://doi.org/10.1515/HF.2003.080>
- 419 Berube M-A, Schorr D, Ball RJ, et al (2018) Determination of In Situ Esterification
420 Parameters of Citric Acid-Glycerol Based Polymers for Wood Impregnation. *J Polym*
421 *Environ* 26:970–979. <https://doi.org/10.1007/s10924-017-1011-8>
- 422 Boever LD, Acker JV, Vansteenkiste D, Stevens M (2007) Preservative treatment of
423 willow wood (*Salix alba*): Product retention and spatial distribution. *Wood Research*
424 53:29–42
- 425 Brischke C, Alfredsen G (2020) Wood-water relationships and their role for wood
426 susceptibility to fungal decay. *Appl Microbiol Biotechnol* 104:3781–3795.
427 <https://doi.org/10.1007/s00253-020-10479-1>
- 428 Cadieux-Lynch R, Leroux E, Hermann A, et al (2024) From waste to building material:
429 How whey ultrafiltration permeate can increase wood stability. *Journal of Materials*
430 *Research and Technology* S223878542400228X. <https://doi.org/christ>
- 431 Candelier K, Thevenon M-F, Petriassans A, et al (2016) Control of wood thermal
432 treatment and its effects on decay resistance: a review. *Annals of Forest Science* 73:571–
433 583. <https://doi.org/10.1007/s13595-016-0541-x>
- 434 Čermák P, Rautkari L, Horáček P, et al (2015) Analysis of Dimensional Stability of
435 Thermally Modified Wood Affected by Re-Wetting Cycles. *BioResources* 10:3242–
436 3253. <https://doi.org/10.15376/biores.10.2.3242-3253>
- 437 Droin A, Taverdet JL, Vergnaud JM (1988) Modeling the kinetics of moisture adsorption
438 by wood. *Wood Sci Technol* 22:11–20. <https://doi.org/10.1007/BF00353224>
- 439 Ellis B, Found MS (1983) The effects of water absorption on a polyester/chopped strand
440 mat laminate. *Composites* 14:237–243. [https://doi.org/10.1016/0010-4361\(83\)90010-1](https://doi.org/10.1016/0010-4361(83)90010-1)
- 441 Essoua Essoua GG, Blanchet P, Landry V, Beauregard R (2016) Pine Wood Treated with
442 a Citric Acid and Glycerol Mixture: Biomaterial Performance Improved by a Bio-

443 byproduct. *BioResources* 11:3049–3072. <https://doi.org/10.15376/biores.11.2.3049-3072>

444 Glass S, Zelinka S (2010) Moisture Relations and Physical Properties of Wood. In: *Wood*

445 *handbook--Wood as an engineering material*, United States Department of Agriculture-

446 Forest Service-Forest Products Laboratory. p 40

447 Hernández RE (2007) Swelling Properties of Hardwood as Affected by their Extraneous

448 Substances, Wood Density, and Interlocked Grain. *Wood and Fiber Science* 39:146–158

449 Hill C, Altgen M, Rautkari L (2021) Thermal modification of wood—a review: chemical

450 changes and hygroscopicity. *J Mater Sci* 56:6581–6614. [https://doi.org/10.1007/s10853-](https://doi.org/10.1007/s10853-020-05722-z)

451 [020-05722-z](https://doi.org/10.1007/s10853-020-05722-z)

452 Hill CAS (2002) How does the chemical modification of wood provide protection against

453 decay fungi? *Proceedings of the COST E22 Meeting*

454 Hill CAS, Altgen M, Penttilä P, Rautkari L (2024) Review: interaction of water vapour

455 with wood and other hygro-responsive materials. *J Mater Sci.*

456 <https://doi.org/10.1007/s10853-024-09636-y>

457 Hill CAS, Forster SC, Farahani MRM, et al (2005) An investigation of cell wall

458 micropore blocking as a possible mechanism for the decay resistance of anhydride

459 modified wood. *International Biodeterioration & Biodegradation* 55:69–76.

460 <https://doi.org/10.1016/j.ibiod.2004.07.003>

461 Hill CAS, Mallon S (1998) The Chemical Modification of Scots Pine with Succinic

462 Anhydride or Octenyl Succinic Anhydride. I. Dimensional Stabilisation. *Holzforschung*

463 52:427–433. <https://doi.org/10.1515/hfsg.1998.52.4.427>

464 Hillis WE (1984) High temperature and chemical effects on wood stability: Part 1:

465 General considerations. *Wood Sci Technol* 18:281–293.

466 <https://doi.org/10.1007/BF00353364>

467 Hötte C, Militz H (2024) Esterification of wood with citric acid and sorbitol: effect of the

468 copolymer on the properties of the modified wood. Part 1: macroscopic changes, fixation

469 of chemicals and impact bending properties. *Holzforschung.* [https://doi.org/10.1515/hf-](https://doi.org/10.1515/hf-2024-0070)

470 [2024-0070](https://doi.org/10.1515/hf-2024-0070)

471 Kamdem DP, Pizzi A, Jermannaud A (2002) Durability of heat-treated wood. *Holz als*

472 *Roh- und Werkstoff* 60:1–6. <https://doi.org/10.1007/s00107-001-0261-1>

473 Kurkowiak K, Emmerich L, Militz H (2022) Wood chemical modification based on bio-

474 based polycarboxylic acid and polyols – status quo and future perspectives. *Wood*

475 *Material Science & Engineering* 17:1040–1054.

476 <https://doi.org/10.1080/17480272.2021.1925961>

477 Kurkowiak K, Hentges D, Dumarçay S, et al (2023) Understanding the mode of action of

478 sorbitol and citric acid (SorCA) in wood. *Wood Material Science & Engineering* 18:67–

479 75. <https://doi.org/10.1080/17480272.2022.2125340>

480 Mantanis GI, Young RA, Rowell RM (1994) Swelling of wood: Part 1. Swelling in

481 water. *Wood Sci Technol* 28:. <https://doi.org/10.1007/BF00192691>

482 Masoumi A, Bond B, Sharp AZ (2024) Kinetics of moisture absorption, swelling and

483 shrinkage of thermally modified hardwoods.

484 <https://doi.org/10.13140/RG.2.2.28417.67687>

485 Masoumi A, Bond BH (2024a) Dimensional stability and equilibrium moisture content of

486 thermally modified hardwoods. *BioRes* 19:1218–1228.

487 <https://doi.org/10.15376/biores.19.1.1218-1228>

488 Masoumi A, Bond BH (2024b) Dimensional stability and equilibrium moisture content of
489 thermally modified hardwoods. *BioRes* 19:1218–1228.
490 <https://doi.org/10.15376/biores.19.1.1218-1228>

491 Niemz P, Teischinger A, Sandberg D (eds) (2023) *Springer Handbook of Wood Science*
492 *and Technology*. Springer International Publishing, Cham

493 Papadopoulos AN, Hill CAS (2002) The biological effectiveness of wood modified with
494 linear chain carboxylic acid anhydrides against *Coniophora puteana*. *Holz als Roh- und*
495 *Werkstoff* 60:329–332. <https://doi.org/10.1007/s00107-002-0327-8>

496 Patera A, Derome D, Griffa M, Carmeliet J (2013) Hysteresis in swelling and in sorption
497 of wood tissue. *Journal of Structural Biology* 182:226–234.
498 <https://doi.org/10.1016/j.jsb.2013.03.003>

499 Peeters K, Larnøy E, Kutnar A, Hill CAS (2018) An examination of the potential for the
500 use of the Maillard reaction to modify wood. *International Wood Products Journal* 9:108–
501 114. <https://doi.org/10.1080/20426445.2018.1471840>

502 Rowell RM (2006) Chemical modification of wood: A short review. *Wood Material*
503 *Science & Engineering* 1:29–33. <https://doi.org/10.1080/17480270600670923>

504 Rowell RM, Banks WB (1985) Water repellency and dimensional stability of wood. U.S.
505 Department of Agriculture, Forest Service, Forest Products Laboratory, Madison, WI

506 Rowell RM, Ibach RE, McSweeney J, Nilsson T (2009) Understanding decay resistance,
507 dimensional stability and strength changes in heat-treated and acetylated wood. *Wood*
508 *Material Science & Engineering* 4:14–22. <https://doi.org/10.1080/17480270903261339>

509 Sargent R (2019) Evaluating dimensional stability in solid wood: a review of current
510 practice. *J Wood Sci* 65:36. <https://doi.org/10.1186/s10086-019-1817-1>

511 Sargent R (2022) Evaluating Dimensional Stability in Modified Wood: An Experimental
512 Comparison of Test Methods. *Forests* 13:613. <https://doi.org/10.3390/f13040613>

513 Schorr D, Boivin G, Stirling R (2022) Effect of a Thermal Catalyst on Organosilanes
514 Treatment to Improve Durability and Stability of Canadian Wood. *Coatings* 12:1867.
515 <https://doi.org/10.3390/coatings12121867>

516 Schroeder HA (2007) Shrinking and Swelling Differences Between Hardwoods and
517 Softwoods. *Wood and Fiber Science* 4:20–25

518 Thybring EE (2013) The decay resistance of modified wood influenced by moisture
519 exclusion and swelling reduction. *International Biodeterioration & Biodegradation*
520 82:87–95. <https://doi.org/10.1016/j.ibiod.2013.02.004>

521 Thybring EE, Glass SV, Zelinka SL (2019) Kinetics of Water Vapor Sorption in Wood
522 Cell Walls: State of the Art and Research Needs. *Forests* 10:704.
523 <https://doi.org/10.3390/f10080704>

524 Xie Y, Hill CAS, Xiao Z, et al (2010) Water vapor sorption kinetics of wood modified
525 with glutaraldehyde. *J of Applied Polymer Sci* 117:1674–1682.
526 <https://doi.org/10.1002/app.32054>

527 Yang Q (2012) Dimensional stability of chemical modified aspen by using oxidized
528 glucose. Master's thesis, Lulea University of Technology

529 Yang T, Ma E, Zhang J (2016) Dynamic moisture sorption and hygroexpansion of
530 *Populus euramericana* Cv. under two cyclic hygrothermal conditions. *Holzforschung*
531 70:1191–1199. <https://doi.org/10.1515/hf-2016-0015>

532 Zelinka SL, Altgen M, Emmerich L, et al (2022) Review of Wood Modification and

533 Wood Functionalization Technologies. Forests 13:1–46. [https://](https://doi.org/10.3390/f13071004)
534 doi.org/10.3390/f13071004
535