Cleaner Engineering and Technology 31 (2026) 101172

Contents lists available at ScienceDirect

Cleaner Engineering and Technology

journal homepage: www.sciencedirect.com/journal/cleaner-engineering-and-technology

Thermo-pressed specimens coated with lignin-containing thermo-mechanical pulp fines and modified enzymatically to increase hydrophobicity

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ARTICLE INFO

Keywords:
Thermo-mechanical pulp
Laccase
Lauryl gallate
Coating
Hydrophobic
Wet-molding
Dry-forming

ABSTRACT

This study investigates the valorization of fines derived from thermomechanical pulp and made hydrophobic by enzymatic treatment to fabricate thermo-pressed specimens with reduced surface roughness and enhanced hydrophobicity. Thermomechanical pulp was fractionated into three main fractions: (i) a coarse fraction, average fiber length 2.6 mm and a fines content of 13.8%; (ii) a middle fraction, average fiber length 1 mm and fines content of 24.5 %; and (iii) a fines fraction, average fines length 0.3 mm and fines content of 66.5 %. Scanning electron microscopy of the three fractions revealed various structural components including intact fibers, split fibers, and fibrillated objects. Chemical composition analysis confirmed differences in lignin content among the fractions, with the fines fraction exhibiting the highest lignin content (30%). Wet and dry-formed thermopressed specimens were produced from the coarse and middle fractions, which exhibited notable differences in mechanical properties, i.e., tensile strengths between 4 and 20 MPa. Application of the fines fraction as a surface layer effectively reduced both the porosity and surface roughness of the wet- and dry-formed specimens. Hydrophobized fines increased the water contact angle of the wet-formed and thermo-pressed specimens to 86.8 ± 5.3° and 99.5 ± 1.4°. The deposition of hydrophobized fines (20 wt%) onto dry-formed samples created a water barrier that lasted for more than 20 min. However, assessment over a period of 4 months showed that the water barrier was reduced to some 6 min.

1. Introduction

Plastic waste remains one of the most pressing environmental challenges all over the world. The situation is particularly challenging in the food packaging industry, a sector which consumes over 40% of the global industrial packaging with 40% of the packages made from plastics (Semple et al., 2022). Only in Europe, plastic packaging waste generation was approximately 16 million tons in 2022 (European-commission, 2024). Of this waste stream, about 32% was recycled, 40% incinerated with energy recovery, and 28% disposed of in landfills. In other regions, such as Southeast Asia, Africa, or Latin America, the situation is even more alarming, with thousands of tons of plastic waste being inadequately processed each year, further exacerbating the pollution of both marine and terrestrial ecosystems. The persistence and partial degradation of plastic materials in the environment leads to the gradual formation of both microplastics and nanoplastics (MNPs) which could be involved in serious ecological and human health risks, including endocrine disruption, oxidative stress, and inflammatory responses (Galloway and Lewis, 2016; Wright and Kelly, 2017). Moreover, recent studies have confirmed the presence of MNPs in human kidney, liver and brain tissues (Nihart et al., 2025).

Now, more than ever, a coordinated action involving citizens, public authorities and the private sector is essential to address the challenges derived from decades of massive plastic use, especially for food packaging. In response to these challenges, the European Commission has implemented a range of regulatory measures aimed at curbing plastic pollution and promoting sustainable packaging alternatives. The Single-Use Plastics Directive (SUPD), introduced in 2019, for example, mandates significant reductions in the consumption of disposable plastics, thereby stimulating innovation in eco-friendly materials

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https://doi.org/10.1016/j.clet.2026.101172
Received 22 September 2025; Received in revised form 5 January 2026; Accepted 11 February 2026
Available online 12 February 2026

(European-commission, 2019). Additionally, the Packaging and Packaging Waste Regulation (Regulation (EU) 2025/40) enforces a reduction in packaging waste generation and in the use of primary natural resources. These legislative actions, along with the broader objectives of the European Green Deal and the Circular Economy Action Plan, seeks to not only reduce plastic waste generation but also enhance recycling and recovery practices while encouraging the use of biodegradable and bio-based materials (European-commission, 2023). Parallel to these governmental initiatives, the private sector has been proactively developing sustainable packaging solutions. Major companies in industries such as food and beverage have committed to reducing their use of conventional plastics by developing alternative materials and technologies.

Among the most promising developments is the emergence of high-performing cellulose-based thermoformed products based on two technologies, wet-molding and dry-forming, which are achieving properties similar to traditional plastics while largely mitigating their environmental impacts (Semple et al., 2022). Derived from renewable resources, these materials are biodegradable and recyclable, contributing to both the reduction of plastic waste generation and the decarbonization of the packaging sector.

Despite these advantages, cellulose-based thermoformed products still present challenges that must be addressed to fully replace plastic packaging. One of them is their very high sensitivity to moisture and water, which compromise both their mechanical and barrier properties. Currently, cellulose is typically combined with other materials, such as plastic films, aluminum layers, or polymer coatings to reduce its water sensitivity and enhance its barrier properties, resulting in a multi-material product. Such combination allows to extend the shelf life of food and other perishable products but at the same time poses challenges for recycling (Şahin and Karaboyacı, 2021). Firstly, the separation of the different layers, especially when plastic films or aluminum are involved, is technically complex and inefficient separation leads unavoidably to downcycling, disposal in landfills or incineration (Tamizhdurai et al., 2024). Secondly, probably the main issue in this type of cellulose-based multi-material products is the breakdown of plastic components or polymeric coatings into MNPs during the mechanical recycling process (Dube and Okuthe, 2023).

The development of environmentally sustainable solutions, particularly a fully biodegradable one, to improve the barrier properties and water resistance remains a critical challenge to the deployment of a fully circular cellulose-based packaging product. With that aim, chemical modification reactions such as silylation or esterification have been proposed (Rodríguez-Fabià et al., 2022). However, such chemical modifications typically rely on, e.g., organic solvents or require harsh conditions—high temperatures, extended reaction times, toxic reagents, thereby increasing production costs and environmental impact. Alternatively, laccase enzymes can be used to hydrophobize lignocellulosic fibers by grafting hydrophobic compounds to lignin (Filgueira et al., 2017). This eco-friendly process significantly reduces the need for organic solvents and operates under mild conditions, lowering energy consumption compared to chemical modifications. Additionally, the oxidative potential of laccase can be enhanced by incorporating a mediator, which generates reactive radicals capable of reaching sites that the enzyme alone cannot access (Filgueira et al., 2017). This approach offers a sustainable and energy-efficient alternative for improving the water resistance of lignocellulosic materials while maintaining their structural integrity.

The present study explores a novel approach to improve the water sensitivity of cellulose based thermoformed products through the valorization of enzymatically hydrophobized fines applied as a top layer. Generally, the length of those fines lays below 200 μm, with a major fraction in the 20–100 μm range, and their width below 25 μm. Such particular morphology could be intrinsically beneficial to reduce the surface porosity of cellulose packaging. Besides, the disruption of the middle lamella during refining is expected to confer fines a higher lignin content compared to fibers (Kangas and Kleen, 2004), which may also contribute to reduce their hydrophilicity. Finally, enzymatic modification of thermomechanical pulp (TMP) fines would provide an additional mechanism to enhance the hydrophobic properties of thermoformed specimens. All these aspects are investigated in this study.

2. Materials and methods

2.1. Materials

The TMP fibres (from *Picea abies*) were obtained from the reject press of Norske Skog Saugbrugs. Laccase (NS51003) was provided by Novozymes (Bagsværd, Denmark). The laccase activity was assessed by the 2,2′ azinobis(3-ethylbenzothiazoline-6-sulfonic acid) (ABTS) oxidation assay. One unit of laccase activity was defined as the amount of enzyme that oxidized 1 μmol of ABTS/min at 25 °C and pH 5 (0.1 M citric acid buffer). All of the other chemical reagents were purchased from Sigma-Aldrich (St. Louis, MO, USA) at reagent grade and used without further purification.

2.2. TMP fractionation

TMP fibers were fractionated using a Bauer McNett fibre classifier, with 5 tanks with wires: 16-, 30, 50-, 100- and 200-mesh McNett. The 16-mesh McNett fraction corresponds to the coarse fibers, while the 200-mesh McNett corresponds to the fine materials. The fractions 30-, 50- and 100-, were considered as one fraction (P16R100). Three fractions were collected, according to Table 1.

2.3. Chemical and morphological analyses

The carbohydrate composition (cellulose and hemicellulose) was quantified by NREL/TP-510-42618 “Determination of Structural Carbohydrates and Lignin in Biomass”, using high performance liquid chromatography (HPLC, 1260 Infinity II, Agilent) after hydrolysis of the pulp. The acid soluble lignin was measured according to TAPPI UM 250 and the acid insoluble lignin was measured following NREL/TP-510-42618-method.

The Canadian Standard Freeness (CSF) of the pulp fractions was measured in duplicate according to ISO 5267-2:2001 (Standardization, 2001).

The samples were analyzed with a Lorentzen & Wettre Fiber Tester Plus in triplicate, considering the following parameters: length (mm), width (μm), fines (length averaged, %) and fibril area (%).

The pulp was imaged with scanning electron microscopy (SEM, Hitachi SU3500), in secondary electron imaging mode. The samples were coated with gold before imaging at various magnifications.

2.4. Hydrophobized fines

The fines fraction (P100R200) was enzymatically hydrophobized

Table 1
Fractionation of TMP fibers.

Pulp code	Wire	Pulp fraction	Description
TMP	-	-	Starting raw pulp
R16	R16	16-mesh McNett	Fibers rejected in 16-mesh
P16R100	P16R30	30-mesh McNett	Fibers from the 3 fractions; P16R30, P30R50 and P50R100
	P30R50	50-mesh McNett	
	P50R100	100-mesh McNett	
P100R200	P100R200	200-mesh McNett	Fines, collected in 200-mesh wire

and used as coating to improve the water-resistance of the thermo-pressed specimens (Fig. 1). For the enzymatic reaction, 1 g of fines was suspended in 100 mL of citric acid aqueous solution buffer (0.1 M, pH 5) at 55 °C. After 30 min under constant agitation, 0.5 mL of laccase enzyme (325 units/mL) and 20 μL of guaiacol (G) were added to the solution. Guaiacol was selected as mediator based on our previous study, for details see (Filgueira et al., 2017). Thirty min later, 5 mL of an acetone solution containing 0.2 g of lauryl gallate (LG) was added. This ratio (lauryl gallate-acetone) was selected based on previous studies (Filgueira et al., 2017, 2021). The reaction was conducted for 2 h at 55 °C under permanent agitation. After the enzymatic treatment, the fines were filtered and washed twice with 100 mL of distilled water/-acetone solution (50:50 v/v) for 30 min at room temperature. Finally, the fines were extensively washed with distilled water.

2.5. Forming of wet-molded specimens

Four samples (500 g/m^2, 150 mm × 80 mm) were produced for each treatment in a mini sheet-former equipment. The samples coated with a fines layer were produced by pre-forming a base sheet (either 490 or 480 g/m^2), and then carefully pouring the fines suspension (correspondingly 10 or 20 g/m^2) on top (Table 2). The samples were dewatered and pressed in a sheet press (PTI Austria GmbH, Laakirchen, Austria), for 2 min at 4.1 bar. After forming and dewatering the samples were dried in a climate room at 23 °C and 50% relative humidity.

2.6. Dry-forming

The fraction P16R100 was selected for further work with air-laid forming technology, because this pulp provided the strongest samples in wet-forming, but also the largest decrease of density and mechanical properties when coated with hydrophobic fines. The dried pulp was fluffed with a hammermill (at 4500 rpm and 5 mm opening, based on preliminary internal trials). The fluffed fibers (1g, target grammage 500 g/m^2) were inserted into a funnel that was placed over a wire screen and connected by an outlet to the vacuum. The fluffed fibers formed an air-laid mat of diameter = 50 mm. The fines were knife-milled using a 10-mesh screen. 0.1 or 0.2g of fines were deposited on the surface of the preformed sample, using the same air-laid procedure described above (Table 3). After forming, the samples were transferred to an aluminum cylinder (inner diameter = 50 mm) pressed cold to compact the samples to approximately 1-2 mm.

Table 2
Composition and structure of wet-molded specimens.

Wet-molded-specimens	Pulp	Fines layer (P100R200, 10 g/m^2)	Structure
S-TMP	TMP	-	Uncoated
S-R16	R16	-	Uncoated
S-P16R100	P16R100	-	Uncoated
S-R16, coated w/fines	R16	P100R200	Coated
S-P16R100, coated w/ fines	P16R100	P100R200	Coated

Table 3
Samples made by dry-forming using an air-laid set-up.

Dry-formed samples	Pulp (g)	Hydrophobized fine material (g)
DF-P16R100	1.0	-
DF-P16R100-10F	0.9	0.1
DF-P16R100-20F	0.8	0.2

2.7. Hot-pressing

The wet- and dry-formed samples were hot-pressed between two metal plates at 200 °C and 10 MPa pressure, during 30 s. Previously it was reported that these settings (temperature and pressure) provided adequate tensile strength in wet-molding trials (Pasquier et al., 2023). The equipment used for hot-pressing was a LabPro 2000 press (Fontijne, the Netherlands).

2.8. Characterization of thermo-pressed specimens

The surface roughness of wet-molded samples was assessed with a Lorentzen & Wettre Parker Print Surf (PPS) tester, following the ISO 8791-4 (Standardization, 2021). The thickness and density were measured following the standard ISO 534 (Standardization, 2011). The equipment for thickness measurements was a Lorentzen & Wettre micrometer 51.

The mechanical properties (tensile strength, modulus and elongation) were assessed with a Zmart.Pro universal material tester (Zwick, Germany) following the ISO 1924-3 (Standardization, 2005). The assessed samples were 70 mm × 15 mm. The distance between the clamps was 40 mm. The samples were conditioned at 23 °C and 50% relative humidity for at least 24 h before measurements.

The water contact angle (WCA) of wet-molded samples was

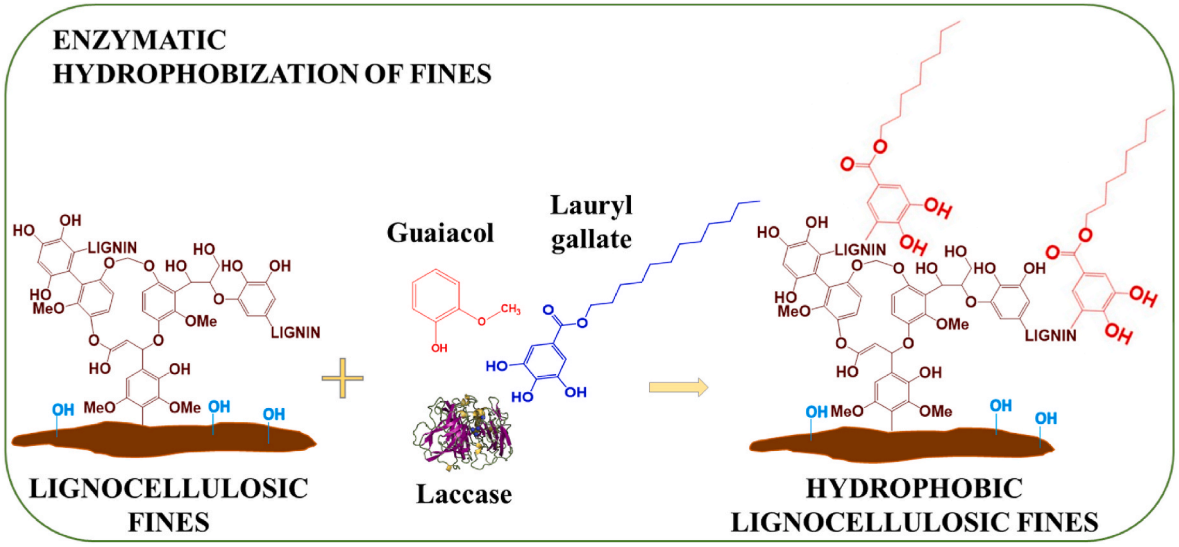

Fig. 1. Schematic representation of the enzymatic hydrophobization of fines, modified from (Filgueira et al., 2017).

measured with a Dynamic Absorption Tester (DAT 1100).

Digital images of the dry-formed samples were acquired with an Epson Perfection Scanner in reflection mode at 1200 dpi (Supplementary information, Fig. S1). Due to the limited size of the dry-formed samples, it was not possible to quantify the WCA using the DAT 1100. Therefore, the water barrier of dry-formed samples was assessed with a tailor-made device that was 3D printed, using an Original Prusa 3D Printer and white polylactic acid filament (diameter = 1.75 mm). A syringe (1 ml) was placed on a 3D printed support device to deposit water droplets (5 droplets, 0.02 ml each) on the dry-formed samples. Images were acquired for 30 min to follow the water penetration. The WCA was measured manually after 1 min using the angle tool in the ImageJ program (version 1.53t). The water penetration was assessed in duplicates: i) 23 days (at room temperature and humidity conditions) and 136 days (in climate room, 23° and 50% relative humidity), after manufacturing the samples. The samples were kept in transparent plastic bags and partly exposed to sunlight from manufacturing to assessment. One new sample per series was assessed after 139-144 days (at varying room temperature (23-24 °C) and humidity conditions (36-40%)). Five droplets of water were deposited, the water barrier was assessed, and the sample was allowed to dry for 24 h at room temperature 23-24 °C and 36-40% humidity conditions before performing the same water drop test on the same sample. The barrier against water was assessed and is considered in this study as the number of minutes until 3 of 5 deposited droplets were completely absorbed into the sample.

Dry-formed samples (20 × 10 mm) were embedded in epoxy resin, ground and polished as described by (Chinga et al., 2007). Five images were acquired in SEM backscatter electron imaging mode from each dry-formed sample DF-P16R100, DF-P16R100-10F and DF-P16R100-20F. The images were automatically thresholded and in cases manually edited to remove remaining noise particles. The root-mean-square roughness (SEM roughness) and the surface layer porosity (SEM porosity) were measured (Chinga et al., 2007). The surface layer was considered the upper-most 10% of local thickness and defined by a rolling ball with diameter of 15 μm as described by (Chinga et al., 2007).

3. Results and discussion

3.1. Characteristics of pulps samples

SEM revealed the structural components such as intact fibers, split fibers and fibrillated materials. At low magnification (100 ×), the long fibers can be observed as well as the network formed by the fines in between the fibers (Fig. 2, Upper left). The coarse fibre fraction (R16) is composed mostly of long and intact fibers, while the middle fibre fractions show an increasing development of well-refined fibrous elements from P16R30 to P50R100. The P100R200 fraction forms a relatively smooth surface (Fig. 2, Lower right).

The qualitative characteristics (SEM analysis) were confirmed by a fibre dimension assessment using a Fiber Tester device, i.e. the coarsest fraction (R16) contained longer and wider fibers, and a relatively minor fraction of fibrils and fines, compared to the TMP and middle fraction (P16R100) (Table 4). The fines fraction (P100R200) contained the smallest fibrous objects (length = 0.3 mm and width = 24 μm), and the highest fibrils and fines content (23% and 67%, respectively).

As expected, the coarsest fibre fraction (R16) provides the highest freeness (721 mL), i.e., water is able to drain relatively easy due to the formation of a fibrous network with low tortuosity and high porosity. The middle fibre fraction (P16R100) has lower freeness compared to the coarse fibre fraction (R16) due to the larger fraction of fines forming a denser network that limits the water flow. It can be noted that the TMP has relatively high freeness (433 mL) compared to highly refined TMP (<100 mL), as refining disrupts the fibre wall, increasing the fraction of fines and decreasing the freeness (Somboon, 2008). Chemi-thermomechanical pulp (CTMP) is commonly used in wet-molding products and the freeness of CTMP from spruce has been reported to be between 420 and 720 ml (Norgren et al., 2018). Therefore, the TMP used as raw material in this study was collected from the reject press of an industrial production, i.e., the TMP was not refined as much as the TMP used for the final application (super-calendered paper) to limit the fraction of fines and secure an adequate freeness for the application. It was not possible to measure the freeness of the fines fraction (P100R200) because the fines material passed freely the wire of the CSF equipment.

Table 4
Characteristics of TMP fibers and fines.

Pulp code	Mean length (mm)	Mean width (μm)	Mean fibril area (%)	Fines (%)	Freeness (mL)
TMP	1.4 ± 0.0	32.8 ± 0.1	11.4 ± 0.4	46.8 ± 0.2	433
R16	2.6 ± 0.0	40.6 ± 0.1	8.2 ± 0.6	13.8 ± 0.6	721
P16R100	1.1 ± 0.0	31.9 ± 0.1	10.6 ± 0.5	24.5 ± 0.1	524
P100R200	0.3 ± 0.0	24.3 ± 0.1	23.2 ± 0.1	66.5 ± 0.2	-

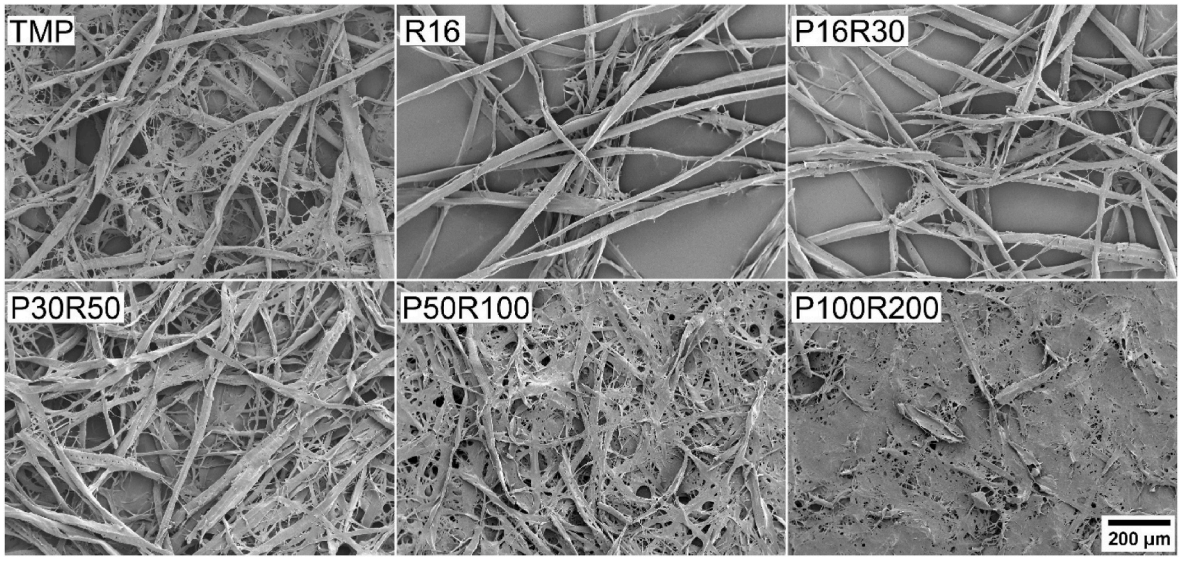

Fig. 2. SEM image of TMP and the corresponding TMP fractions. All images acquired at 100 × magnification.

During the TMP manufacturing process, wood chips are heated with steam or hot water to soften the lignin and then forced to enter the disk refiner where the fibers are separated from the middle lamella and individualized. The length of TMP fibers and the fines content can be adjusted by varying the process conditions. Higher temperature and pressure generally result in longer fibers and lower fines content, whereas lower temperatures and pressures lead to shorter fibers and increased fines content (Petit-Conil et al., 2016). In the pulp & paper sector, fines play a crucial role in paper properties, improving formation, strength, opacity, and barrier performance through the filling of voids and the enhancement of fiber-fiber bonding (Alince et al., 2001). This crucial role of fines justifies the relatively high fines content (46%) in the TMP fibers used in this study.

Based on the production process described above, it was expected that fines would contain a larger fraction of lignin compared to the coarser fibers. As can be seen in Table 5, the fines fraction (P100R200) showed the highest lignin content, 22% higher than the coarsest fraction (R16) and 15% higher than the middle fraction (P16R100). The higher lignin content in the fines fraction (P100R200, lignin content roughly 30%) can be attributed to its origin from various cellular components, including the primary cell wall layer, ray cells, or middle lamella, with the specific source depending on the type of fines in question (fibrillar or flake-like) (Kangas and Kleen, 2004). Inversely, cellulose content was lower in the fines fraction compared with the coarsest and middle fractions. These results are consistent with previous studies focusing on the characterization of fines from mechanical pulps (Sundberg, 2004).

3.2. Characteristics of thermo-pressed specimens

Thermo-pressed specimens prepared from fibers from the coarsest fraction (S-R16) and the middle fraction (S-P16R100) were compared with the TMP fibres. In addition, thermo-pressed specimens coated with a layer of fines (denoted as “Coated w/fines”) and fines made hydrophobic (denoted as “Coated w/hydrophobic fines”) were prepared to assess the effect of the fines coating and their hydrophobic characteristics. S-R16 samples showed the highest surface roughness, whereas S-P16R100 samples showed a surface roughness similar to TMP (Fig. 3). These results highlight the role of fines on reducing the surface roughness of the thermo-pressed specimens. The coating with fines reduced significantly the surface roughness of S-R16 samples (from 7.2 to 5.3 μm) yielding thermo-pressed specimens with a surface roughness similar to both S-P16R100 and TMP. These results were further validated by SEM images, which confirmed that the fines coating effectively covered the surface of the fibre web, resulting in improved surface smoothness (Fig. 4). Coating with hydrophobic fines also reduced the surface roughness of S-R16 and S-P16R100 samples but to a lesser extent than untreated fines (Figs. 3 and 4).

In an aqueous suspension, the TMP fines made hydrophobic through the 12-carbon aliphatic moieties from lauryl gallate, tend to agglomerate driven by hydrophobic interactions (Yan et al., 2016). Water molecules preferentially form hydrogen bonding with other water molecules, leading to an entropically driven exclusion of the nonpolar species from the aqueous phase (Widom et al., 2003). As a result, the hydrophobic species tend to associate, thereby reducing the overall free energy of the system (Sun, 2022). This process is further stabilized by van der Waals forces among the long aliphatic chains, promoting close packing and minimizing contact with water (Chandler, 2005; van Oss et al., 1980). Following this mechanism, hydrophobic fines suffered agglomeration before coating the surface of the samples, reducing their homogeneous distribution over the surface. This explains the increased roughness of samples coated with hydrophobic fines compared to untreated fines (Fig. 3), and was confirmed by SEM analysis of the surface of thermo-pressed specimens (Fig. 4), where it was clear that the hydrophobized fines did not cover the surface as uniformly as the non-hydrophobized ones.

The thickness of thermo-pressed specimens was higher in S-R16 samples compared to the reference (S-TMP) and S-P16R100 samples (Fig. 3, right). This difference appears to be due to the higher fines content in the TMP, which likely compacted the fiber web during formation. Moreover, coating with fines reduced the thickness for both S-R16 and S-P16R100 samples, further confirming the capability of fines to compact the fiber web. However, the coating with hydrophobic fines increased the thickness, suggesting that hydrophobized fines could behave differently from the original fines fraction.

Fines coating also increased the density of the thermo-pressed specimens (Fig. 5). During the preparation of the coated samples, part of the fibers (10 g/m^2) was substituted by fines to produce samples with the same target grammage. Likely, fibers substitution by fines reduced the final volume of the samples and thereby increased the corresponding densities. This effect was similar for all the thermo-pressed specimens, independently of the type of fibers used (i.e., R16 and P16R100). S-R16 but also coated S-R16 showed a lower density than S-TMP and S-R16P100 specimens due to the lower fines and higher fraction of coarse fibers which leads to less densified structures. The coating with fines did not change significantly the mechanical properties for S-R16 and S-P16R100. S-R16 samples exhibited the lowest mechanical properties due to their lower fines content compared with TMP and P16R100 pulps.

Among other factors, the fines play a crucial role in improving the surface contact between the fibers, thus improving the consolidation of the fibre network, which strengthens the corresponding mechanical properties (Sirviö and Nurminen, 2004). This effect is especially relevant when fines are well dispersed in the furnish, where they can accumulate at fiber crossings and enhance bonding within the fiber network (Motamedian et al., 2019). It is well established that, in dry paper, mechanical properties are primarily governed by fiber strength and the bond strength at fiber crossings (Tejado and van de Ven, 2010). Coating with non-hydrophobized fines did not affect significantly the mechanical properties of the thermo-pressed specimens. On the other hand, coating with hydrophobic fines lowered considerably the mechanical properties of both S-R16 and S-P16R100 samples.

Previously, we have reported that LG-hydrophobized TMP fibers increased the contact angle to ~100° (Filgueira et al., 2017). The fines made hydrophobic were also confirmed in this study (Supporting information, Fig. S2). Fines made hydrophobic have lowered their surface energy (Primc et al., 2024), thereby restricting their ability to form hydrogen bonds with the hydroxyl groups of the fibers within the fiber web. Furthermore, in the preparation of samples coated with fines (both hydrophobized and non-hydrophobized), a specific amount of fibers was replaced with an equivalent amount of fines to maintain a consistent grammage across samples. The partial substitution of fibers, with a high ability to chemically interact by hydrogen bonding with adjacent fibers, by hydrophobic fines with a notably lower ability to form hydrogen

Table 5
Chemical composition of TMP fibers and fines.

Pulp code	Sugars					Lignin	
	Glucose (%)	Xylose (%)	Galactose (%)	Arabinose (%)	Mannose (%)	Acid Insoluble (%)	Acid soluble (%)
TMP	47.50 ± 0.02	5.03 ± 0.28	1.30 ± 0.08	0.61 ± 0.03	12.87 ± 0.92	26.13 ± 0.13	0.52 ± 0.52
R16	49.63 ± 1.00	5.00 ± 0.65	1.25 ± 0.13	0.77 ± 0.01	12.84 ± 0.37	23.97 ± 0.32	0.48 ± 0.03
P16R100	49.21 ± 0.32	5.16 ± 0.29	1.40 ± 0.00	0.60 ± 0.03	11.61 ± 0.43	25.37 ± 0.04	0.50 ± 0.01
P100R200	44.64 ± 1.07	5.02 ± 0.16	1.92 ± 0.00	0.83 ± 0.07	10.13 ± 0.14	29.20 ± 0.19	0.57 ± 0.05

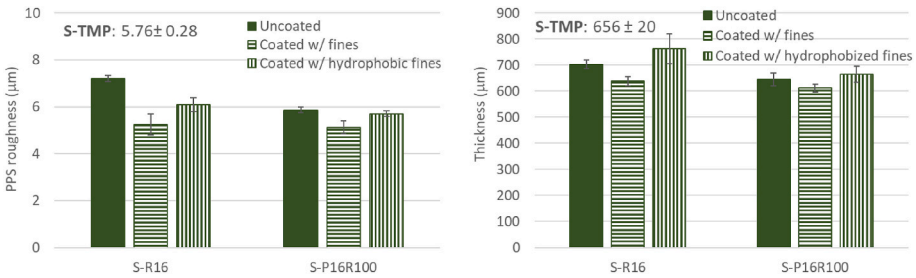

Fig. 3. Surface roughness and thickness of uncoated and coated samples.

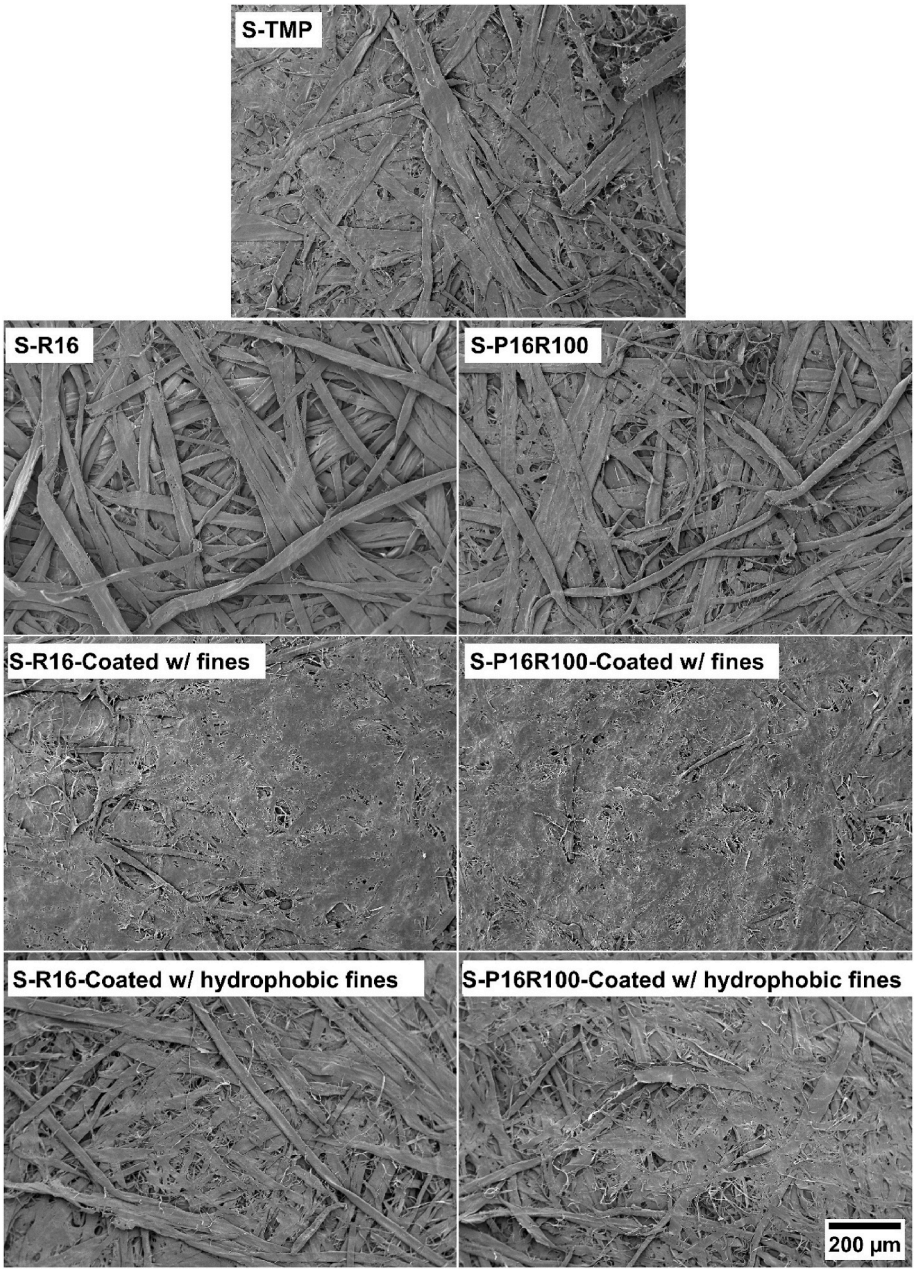

Fig. 4. SEM surface analysis of wet-molded samples. First row) S-TMP sample. Second row) Samples S-R16 and S-P16R100. Third row) The corresponding samples coated with a layer of fines (10 g/m^2). Fourth row) Samples coated with hydrophobic fines. Images acquired with 100 × magnification.

bonds with adjacent fibers could also explain the lower mechanical properties observed in the thermo-pressed specimens coated with hydrophobic fines.

As explained above, hydrophobic species reduce the overall free energy of the system (Sun, 2022). Furthermore, the decrease of wettability of pulp fibers reduces the tensile strength of paper, and this was

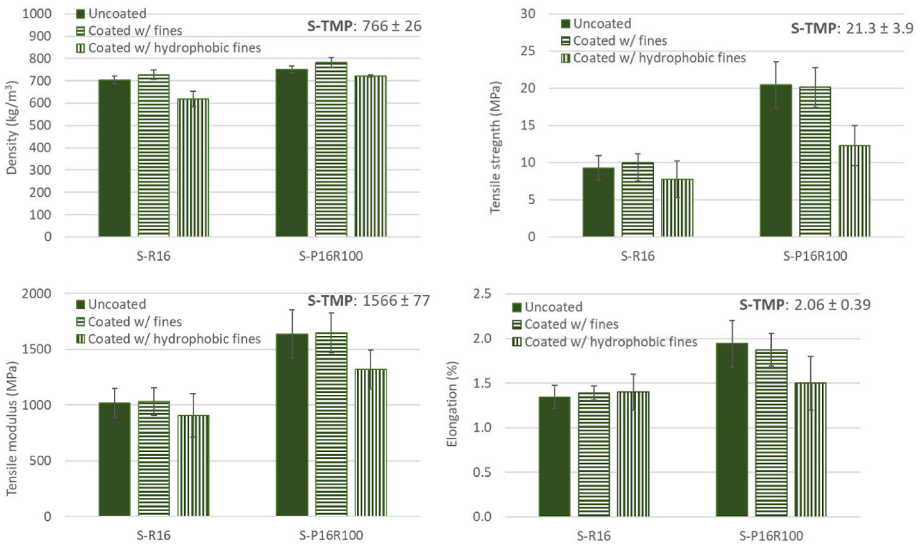

Fig. 5. Density and mechanical properties (tensile strength, tensile modulus and elongation) of uncoated and coated samples.

attributed to a decrease of the fiber-fiber bond strength (Jin et al., 2022). Consequently, hydrophobized fines reduced the density and mechanical properties of both S-R16 and S-P16R100 samples (Fig. 5). The results suggest that hydrophobized fines may have partially penetrated the fibre network, especially in S-R16 specimens with a more open structure (Figs. 3 and 4). In contrast, non-hydrophobized fines slightly increased density and maintained the mechanical performance. These results highlight the importance of fines–fibre compatibility and the need to improve: i) the untreated fines dispersion in the bulk to enhance the mechanical characteristics of thermo-pressed materials and ii) the deposition of hydrophobized fines on the surface to render hydrophobicity.

3.3. Coating with hydrophobized fines fraction

Water contact angle (WCA) measurements confirmed that the non-hydrophobized fines coating slightly reduced the hydrophilicity of the thermo-pressed samples (Fig. 6). As previously mentioned, the fines fraction had a higher lignin content than the R16 and P16R100 fractions. Since lignin, due to its chemical nature, has less affinity to water than cellulose, coating with fines reduced the exposure of hydroxyl groups of cellulose fibers to water molecules, thereby improving the WCA. A surface is classified as hydrophobic if the water contact angle (WCA) exceeds 90°; the WCA measured on the coated specimens was approximately 60°, showing that the samples did not reach that level. WCA of 67° and 73° have been previously reported for lignin and TMP fibers, respectively (Liukkonen, 1997).

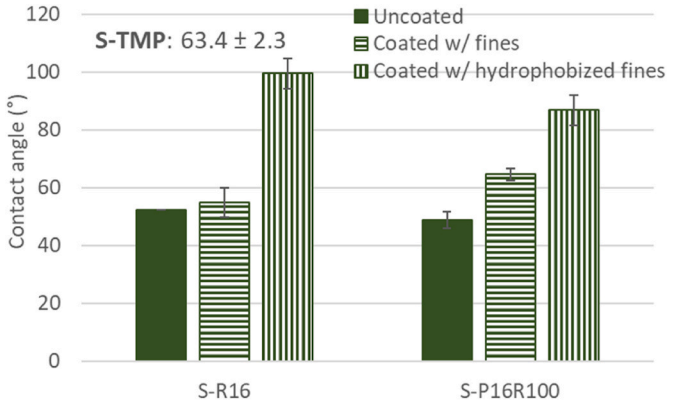

Fig. 6. Water contact angle measured on S-TMP, the coarsest fraction (S-R16) and the middle fraction (S-P16R100), uncoated and coated samples (coating = 10 g/m^2) are included.

The ability of laccase to oxidize phenolic moieties generates phenoxy radicals that can react among them, facilitating the grafting of phenolic substrates (Chandra and Ragauskas, 2002). Accordingly, the higher lignin content of the fines fraction is expected to improve the efficiency of the enzymatic modification (Kalia et al., 2014). Compared to previous studies (Widsten and Kandelbauer, 2008), in this study, a highly sustainable process was designed, carrying out the reaction in aqueous media containing only a minimum amount of an organic solvent (95/5 water/acetone) and under mild reaction conditions (55 °C and pH 5).

WCA measurement confirmed the efficiency of the hydrophobized fines to improve the hydrophobic properties of the thermo-pressed specimens (Fig. 6). WCA increased from 55° to 99° for S-R16 and from 65° to 88° from S-P16R100 samples with the coating of 10 g/m^2 of hydrophobized fines (Fig. 6). However, SEM inspection revealed the presence of small pores on the surfaces of the coated thermo-pressed specimens (Fig. 4, lower panel). These small pores could enhance capillary action due to stronger capillary forces, thereby improving the wettability of the samples. The influence of this physical effect could explain the moderate WCA achieved in this study (86.8 ± 5.3° and 99.5 ± 1.4°).

Theoretically, laccase-assisted reaction functionalizes only the phenolic moieties in TMP which are primarily found in lignin, leaving cellulose and hemicelluloses unmodified. As a result, the hydroxyl groups in polysaccharides remain free to absorb water, potentially reducing the WCA of the pulp. The incorporation of a mediator (i.e., guaiacol) increases the oxidation potential of laccase allowing it to oxidize other chemical structures (Gutiérrez et al., 2007). Guaiacol-derived radicals can also contribute to the modification of aliphatic extractives commonly present in TMP fibers. These extractives, which include fatty acids, waxes, and other hydrophobic aliphatic compounds, often hinder the accessibility of reactive sites necessary for efficient covalent grafting. The reactive guaiacol radicals generated during laccase oxidation could be capable of initiating oxidative transformations that either degrade or functionalize these extractives. This process reduces their hydrophobic barrier effect and exposes lignin and other aromatic structures of the fiber surface, creating more favorable conditions for the covalent attachment of lauryl gallate (Euring et al., 2011). Thus, guaiacol not only serves as a redox mediator for phenolic activation but may also play a complementary role in surface cleaning and activation, enhancing the overall efficiency of the enzymatic

grafting process.

The enhanced oxidation potential of the laccase-mediator system does not appear to be sufficient to oxidize the hydroxyl groups in cellulose and hemicelluloses, leaving their hydrophilic nature unchanged. Additionally, TMP used in paper manufacturing contains a significant fraction of split fibers (Kure et al., 1999). While these fibers enhance paper properties by increasing surface area and flexibility in the fibre web matrix, they may adversely affect the paper hydrophobicity. This is because larger fragments of the S2 layer, mainly composed of cellulose and hemicelluloses, are exposed, making the fibre more prone to water absorption. As an attempt to increase the water barrier properties of the thermo-pressed specimens, the coating layer was increased from 10 to 20 g/m^2. The achieved WCA was 95.7 ± 10.1° and 99.5 ± 1.4° for the specimens S-R16 and S-P16R100 respectively. However, the droplet of water was absorbed rapidly and within 10 s. SEM analysis (Supplementary, Fig. S3) revealed that the surface of the 20 g/m^2-coated samples was porous and confirmed the observations made on the 10 g/m^2 samples, i.e., the hydrophobic fines seemed to agglomerate in water, yielding an inhomogeneous surface coating.

3.4. Fines deposition in a dry-forming process

Previous observations in wet-molding trials indicated two main limitations: i) hydrophobic fines seemed to lower the ability to form hydrogen bonds with adjacent fibers thus reducing the mechanical properties, and ii) the fines agglomerate on the specimen surface thus reducing the ability to form a homogeneous hydrophobic coating. Therefore, the performance of hydrophobic fines was assessed in a dry forming process. The assessment was based on the P16R100 pulp fraction since this provided the best mechanical properties and with acceptable hydrophobic characteristics, i.e., WCA 86.8 ± 5.3° and 99.5 ± 1.4° for the samples coated with 10 and 20 g/m^2, respectively.

Dry-forming usually yields lower mechanical properties compared to wet-molding (Pasquier et al., 2024) and this was confirmed in this study. Considering similar densities between roughly 750 and 800 kg/m^3 (Figs. 5 and 7), the dry formed samples have considerably lower strength (4-6 MPa, Fig. 7) compared to the wet-molded samples (12-20 MPa, Fig. 5). The modulus and elongation were also reduced. This is due to the relatively poor inter-fiber bonding in the absence of water (Hirn and Schennach, 2015), during dry-forming. On the other hand, and considering only the dry-forming samples, deposition of hydrophobic fines on the surface did not affect the mechanical properties negatively. This is an indication that the hydrophobic fines were deposited primarily on the surface of the specimen, i.e., the hydrophobic fines did not penetrate into the network of pulp fibers as was probably the case during wet-molding.

SEM revealed that the hydrophobic fines formed a homogeneous layer on the surface of the specimens (Fig. 8). Note that the surfaces of the dry-formed samples coated with hydrophobic fines (Fig. 8) appeared smoother than the surfaces of the coated wet-molded samples (Fig. 4). The densities of the dry-formed samples increase as the coating increases (Fig. 7), while the corresponding thicknesses decrease (Supplementary, Table S1). However, a macro-scale assessment (based on optical images) demonstrated that the amount of fines also affected the homogeneity. Compared to the DF-P16R100-20F samples (20% of fines), the DF-P16R100-10F samples (10% of fines) had some surface spots that were apparently not covered completely (Supplementary information, Fig. 1). This difference on the surface coating also determined the water absorption behavior of the samples.

A water drop test was conducted and the WCA was measured (Supplementary information, Fig. S4). The water penetrated almost immediately into the DF-P16R100. The WCA after 1 min of samples DF-P16R100-10F and DF-P16R100-20F were 81.3 ± 1.8° and 84.1 ± 0.7°, respectively. The highest contact angle obtained for the samples coated with hydrophobic fines is close to 100° (Fig. 6), which indicates moderate hydrophobicity. When compared to commercial packaging materials, these values are in the same range as fiber-based food service products treated with sizing agents such as AKD (alkyl ketene dimer) and ASA (alkenyl succinic anhydride), which typically exhibit contact angles between 85° and 105° (Ono and Miyanishi, 2000). This suggests that the developed coating provides a level of water resistance comparable to widely used fiber-based solutions in the market.

For reference, untreated molded fiber generally shows much lower contact angles (30°–60°), while PFAS-treated fiber or advanced bio-based coatings can exceed 110° (Chemistry, 2020). Common plastics used in food packaging, such as PET (~72–76°), polystyrene (~87°), and polypropylene (~102°), span a similar or slightly higher range (Smith, 2023). Therefore, achieving values close to 100° positions the proposed material as an interesting and competitive alternative to conventional fiber-based packaging solutions without relying on fluorochemicals.

Although, the WCA after 1 min was less than 90° (hydrophobic surface), the modification formed a water barrier that lasted for relatively long periods. The dry-formed samples resisted the water penetration for more than 5 min (Fig. 9 and Supplementary information, Fig. S4) compared to the wet-molded samples (<10 s). This was

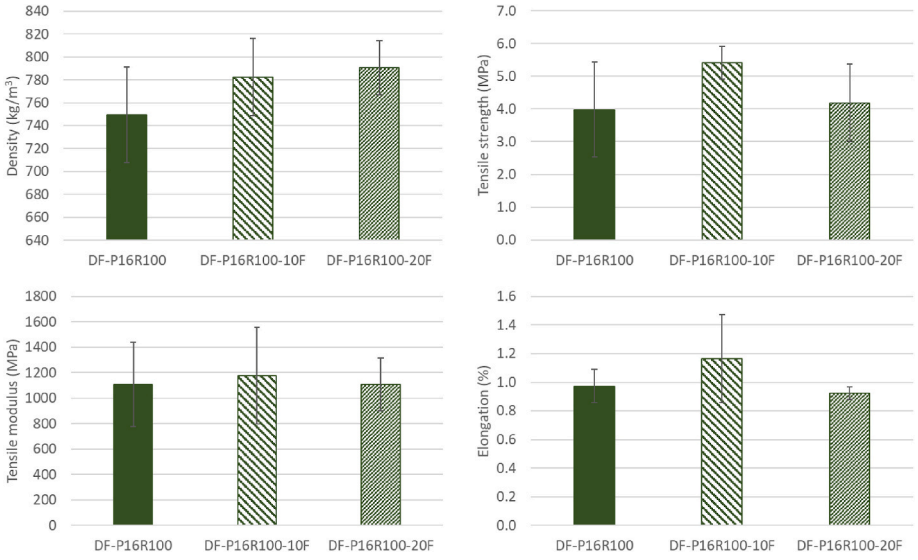

Fig. 7. Density and mechanical properties of dry-formed samples.

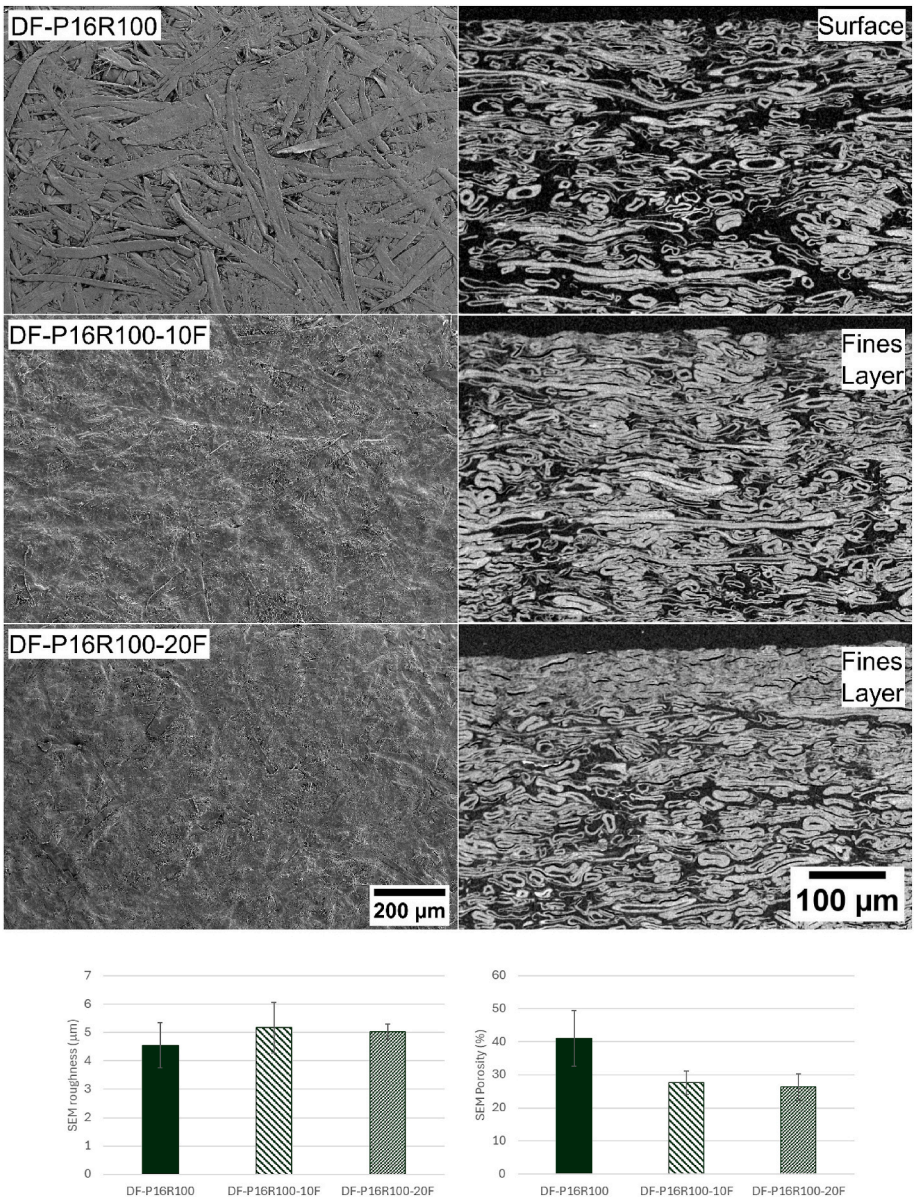

Fig. 8. Left panel) SEM surface images of dry-formed samples DF-P16R100, DF-P16R100-10F and DF-P16R100-20F. Right panel) The corresponding SEM cross-sectional images of the coated surface. Lower panel) Roughness and surface layer porosity measured on SEM cross-sectional images.

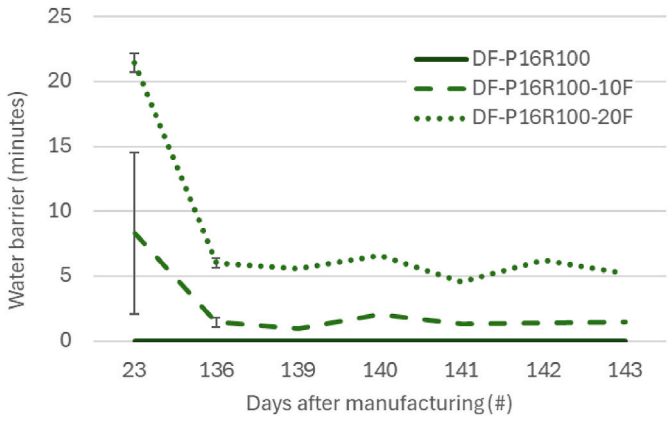

Fig. 9. Barrier against water as a function of time.

probably caused by the larger fraction of fines on the surface of the dry-formed samples (50-100 g/m^2) compared to the wet-molded samples (10-20 g/m^2). Additionally, a higher fines content on the surface resulted in a greater water barrier effect, i.e., the sample with 100 g/m^2 hydrophobized fines resisted the water penetration for some 21 min compared to the sample with 50 g/m^2 fines where the water penetrated before 8 min (Fig. 9 and Supplementary information, Fig. S4).

The water drop test was repeated 136 days after the samples were manufactured and it was noted a reduction of the water barrier, i.e. the DF-P16R100-10F and DF-P16R100-20F resisted the water penetration approximately 1.4 and 6 min, respectively (Fig. 9). The same samples were allowed to dry for 24 h before assessing the water barrier again. The humidity and temperature during the assessment are provided in Supplementary Information Table S2. Although, the water barrier was reduced from 8.3 and 21.4 min to 1.4 and 6 min for samples DF-P16R100-10F and DF-P16R100-20F, respectively, the water barrier was relatively constant during 5 days of assessment. It is speculated that the reduction of water barrier may be due to a long-term degradation of the grafted lauryl gallate, due to e.g. oxidation and UV light. It is

important to emphasize that in most of the cases when barrier against water is assessed, this is based mostly on initial water contact angles (~1 s) and assessment over time is omitted (Rodríguez-Fabià et al., 2022). In the present study, we have assessed lauryl gallate-modified surfaces, the conferred barrier against water and the long-term (4 months) stability of the surface modification.

In this study, coating with a layer of hydrophobized fines was selected as the application method to reduce the water wettability of the thermo-pressed samples. However, as stated above, the fines agglomerated during the formation of the wet-formed samples. Dry-forming seems to be a better alternative in this respect, and an homogeneous layer was formed. Increasing the fines layer to 50 and 100 g/m^2 was beneficial for securing water barrier properties, without significantly compromising the mechanical properties in dry formed samples.

Finally, it is worth mentioning that the main bottlenecks of the process are the high costs of the enzyme and lauryl gallate, and the need for washing steps with acetone. Laccase is already produced at industrial scale for several applications, including the textile sector, which suggests that enzyme costs could be reduced through scalable process optimization. Ongoing research is also aimed at lowering the dosage of lauryl gallate without compromising performance and at replacing acetone washing with more sustainable alternatives based on e.g. surfactants. These strategies aim to make the enzymatic hydrophobization process more economically viable and environmentally friendly.

4. Conclusion

Cellulose-based materials are among the most promising alternatives to plastic-derived packaging products. However, their low barrier properties against oxygen and moisture remain a significant challenge. Modifying the surface chemical and physical properties of these materials is essential to enhance their performance and broaden their applications in packaging, especially for food products. Such modifications must also be sustainable, minimizing the consumption of oil-derived products, energy, and water. The present study focuses on valorizing fines from thermomechanical pulp (TMP) to improve the surface properties of thermo-pressed specimens. A layer of unmodified fines resulted in lower surface roughness and porosity, which can enhance barrier properties, printing quality, and lamination performance.

The relatively high lignin content in fines (lignin content ~30 %) offers an opportunity to develop laccase-assisted modification, which activates lignin phenolic groups to graft hydrophobic phenolic compounds. Water contact angle (WCA) measurements confirmed the grafting of lauryl gallate onto the fines, achieving hydrophobic properties (WCA >90°) in the coated samples. However, the hydrophobic fines also reduced the mechanical properties of the wet-molded and thermo-pressed specimens. On the other hand, deposition of hydrophobized fines on dry-formed specimens was successful, and a homogeneous layer that covered the surface pores was achieved. Deposition of fines in a dry-forming process did not affect the mechanical properties negatively. Additionally, the water barrier was demonstrated and this was maintained for some 20-30 min. However, long-term assessment (4 months) revealed that the water barrier was reduced from 21.4 to 6 min. A balance between hydrophobicity, mechanical performance and recyclability must be established in future work to further improve the functional properties of thermo-pressed specimens.

CRediT authorship contribution statement

Gary Chinga-Carrasco: Writing – review & editing, Writing – original draft, Visualization, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Jaime Garcia-Garrido:** Writing – review & editing, Methodology, Investigation. **Alvaro Tejado-Etayo:** Writing – review & editing, Supervision, Resources, Project administration, Investigation, Funding acquisition. **Daniel Martinez-Filgueira:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Resources, Methodology, Investigation, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

The authors acknowledge Berit Leinsvang, Ingebjørg Leirset, Kenneth Aasarød, Johnny K. Melbø and Steinar Seehuus from RISE PFI for skillful laboratory work. This work was supported by funding from the Circular Bio-based Europe (CBE) Joint Undertaking and its members under Grant agreement No 101112521 (REDYSIGN Project).

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.clet.2026.101172.

Data availability

Data will be made available on request.

References

Alince, B., Porubská, J., van de Ven, T.G.M., 2001. Effect of model and fractionated TMP fines on sheet properties. In: Baker, C.F., e (Eds.), The Science of Papermaking, Trans. of the Xiith Fund. Res. Symp. Oxford. FRC, Manchester, pp. 1343–1355.
Chandler, D., 2005. Interfaces and the driving force of hydrophobic assembly. Nature 437 (7059), 640–647.
Chandra, R.P., Ragauskas, A.J., 2002. Evaluating laccase-facilitated coupling of phenolic acids to high-yield kraft pulps. Enzym. Microb. Technol. 30 (7), 855–861.
Chemistry, B.C.f.G., 2020. Greener solutions case study: PFAS and molded fiber packaging. https://bcgc.berkeley.edu/sites/default/files/food_packaging_case_study_-_final_-_web.pdf. (Accessed 24 November 2025), 2025.
Chinga, G., Solheim, O., Mörseburg, K., 2007. Cross-sectional dimensions of fiber and pore networks based on Euclidean distance maps. Nord. Pulp Pap Res. J. 22 (4), 500–507.
Dube, E., Okuthe, G.E., 2023. Plastics and Micro/nano-plastics (MNPs) in the environment: occurrence, impact, and toxicity. Int. J. Environ. Res. Publ. Health 20 (17).
Euring, M., Rühl, M., Ritter, N., Kües, U., Kharazipour, A., 2011. Laccase mediator systems for eco-friendly production of medium-density fiberboard (MDF) on a pilot scale: physicochemical analysis of the reaction mechanism. Biotechnol. J. 6 (10), 1253–1261.
European-commission, 2019. Directive - 2019/904 - EN - SUP directive - EUR-Lex. https://eur-lex.europa.eu/eli/dir/2019/904/oj/eng. (Accessed 12 February 2025).
European-commission, 2023. Circular economy action plan - european commission. https://environment.ec.europa.eu/strategy/circular-economy-action-plan_en. (Accessed 12 February 2025).
European-commission, 2024. Packaging waste statistics - statistics explained. https://ec.europa.eu/eurostat/statistics-explained/index.php?title=Packaging_waste_statistics. (Accessed 12 February 2025).
Filgueira, D., Bolaño, C., Gouveia, S., Moldes, D., 2021. Enzymatic functionalization of wood as an antifouling strategy against the marine bacterium Cobetia marina. Polymers 13 (21), 3795.
Filgueira, D., Holmen, S., Melbø, J.K., Moldes, D., Echtermeyer, A.T., Chinga-Carrasco, G., 2017. Enzymatic-assisted modification of thermomechanical pulp fibers to improve the interfacial adhesion with poly(lactic acid) for 3D printing. Acs Sustain. Chem. Eng. 5 (10), 9338–9346.
Galloway, T.S., Lewis, C.N., 2016. Marine microplastics spell big problems for future generations. Proc. Natl. Acad. Sci. U. S. A. 113 (9), 2331–2333.
Gutiérrez, A., Rencoret, J., Ibarra, D., Molina, S., Camarero, S., Romero, J., Del Río, J.C., Martínez, Á.T., 2007. Removal of lipophilic extractives from paper pulp by laccase and lignin-derived phenols as natural mediators. Environ. Sci. Technol. 41 (11), 4124–4129.
Hirn, U., Schennach, R., 2015. Comprehensive analysis of individual pulp fiber bonds quantifies the mechanisms of fiber bonding in paper. Sci. Rep. 5 (1), 10503.
Jin, H., Kose, R., Akada, N., Okayama, T., 2022. Relationship between wettability of pulp fibers and tensile strength of paper during recycling. Sci. Rep. 12 (1), 1560.
Kalia, S., Thakur, K., Kumar, A., Celli, A., 2014. Laccase-assisted surface functionalization of lignocellulosics. J. Mol. Catal. B Enzym. 102, 48–58.

Kangas, H., Kleen, M., 2004. Surface chemical and morphological properties of mechanical pulp fines. Nord. Pulp Pap Res. J. 19 (2), 191–199.
Kure, K.-A., Dahlqvist, G., Helle, T., 1999. Morphological characteristics of TMP fibres as affected by the rotational speed of the refiner. Nordic pulp & paper research. Journal 14 (2), 105–110.
Liukkonen, A., 1997. Contact angle of water on paper components: sessile drops versus environmental scanning electron microscope measurements. Scanning 19 (6), 411–415.
Motamedian, H.R., Halilovic, A.E., Kulachenko, A., 2019. Mechanisms of strength and stiffness improvement of paper after PFI refining with a focus on the effect of fines. Cellulose 26 (6), 4099–4124.
Nihart, A.J., Garcia, M.A., El Hayek, E., Liu, R., Olewine, M., Kingston, J.D., Castillo, E.F., Gullapalli, R.R., Howard, T., Bleske, B., Scott, J., Gonzalez-Estrella, J., Gross, J.M., Spilde, M., Adolphi, N.L., Gallego, D.F., Jarrell, H.S., Dvorscak, G., Zuluaga-Ruiz, M. E., West, A.B., Campen, M.J., 2025. Bioaccumulation of microplastics in decedent human brains. Nat. Med. 31 (4), 1114–1119.
Norgren, S., Pettersson, G., Höglund, H., 2018. Strong paper from spruce CTMP – part II: effect of pressing at nip press temperatures above the lignin softening temperature. Nord. Pulp Pap Res. J. 33 (1), 142–149.
Ono, H., Miyanishi, T., 2000. Comparative study on the sizing efficiency and kinetics between ASA and AKD sizing. Jpn. Tappi J. 54 (6), 812–819, 021.
Pasquier, E., Kathrin, M., Kristin, S., Ruwoldt, J., 2024. Effect of raw material and process conditions during the dry forming of CTMP fibers for molded pulp products. J. Nat. Fibers 21 (1), 2409890.
Pasquier, E., Skunde, R., Ruwoldt, J., 2023. Influence of temperature and pressure during thermoforming of softwood pulp. J. Bioresour. Bioprod. 8 (4), 408–420.
Petit-Conil, M., Lecourt, M., Meyer, V., 2016. High-yield pulps: an interesting concept for producing lignocellulosic fibers. Lignocellulosic Fibers Wood Handb. 157–205.
Primc, G., Vesel, A., Zaplotnik, R., Gorjanc, M., Gselman, P., Lehocký, M., Mozetič, M., 2024. Recent progress in cellulose hydrophobization by gaseous plasma treatments. Polymers 16 (6), 789.
Rodríguez-Fabià, S., Torstensen, J., Johansson, L., Syverud, K., 2022. Hydrophobization of lignocellulosic materials part II: chemical modification. Cellulose 29 (17), 8957–8995.
Şahin, G.G., Karaboyacı, M., 2021. Process and machinery design for the recycling of tetra pak components. J. Clean. Prod. 323, 129186.
Semple, K.E., Zhou, C., Rojas, O.J., Nkeuwa, W.N., Dai, C., 2022. Moulded pulp fibers for disposable food packaging: a state-of-the-art review. Food Packag. Shelf Life 33, 100908.
Sirviö, J., Nurminen, I., 2004. Systematic changes in paper properties caused by fines. Pulp Paper Canada 105 (8), 39–42.
Smith, R., 2023. Critical surface tension and contact angle with water for various polymers. https://www.accudynetest.com/polytable_03.html?sortby=contact_angle. (Accessed 24 November 2025), 2025.
Somboon, P., Paulapuro, H., 2008. Surface mechanical treatment of TMP pulp fibers using grit material. TAPPI J. 7 (12), 4–9.
Standardization, I.O.f., 2001. ISO 5267-2:2001 Pulps — Determination of Drainability Part 2: "Canadian Standard" Freeness Method, p. 14.
Standardization, I.O.f., 2005. ISO 1924-3 Paper and Board — Determination of Tensile Properties — Part 3 Constant Rate of Elongation Method (100 mm/min), p. 10.
Standardization, I.O.f., 2011. ISO 534:2011 Paper and Board — Determination of Thickness, Density and Specific Volume, p. 13.
Standardization, I.O.f., 2021. ISO 8791-4 Paper and Board – Determination of roughness/smoothness (Air Leak Methods) - Part 4: Print-Surf Method, vols. 2021–05, p. 17.
Sun, Q., 2022. The hydrophobic effects: our current understanding. Molecules 27 (20), 7009.
Sundberg, A., 2004. Fines in spruce TMP, BTMP and CTMP - Chemical composition and sorption of mannans. Nord. Pulp Pap Res. J. 19 (2), 176–182.
Tamizhdurai, P., Mangesh, V.L., Santhosh, S., Vedavalli, R., Kavitha, C., Bhutto, J.K., Alreshidi, M.A., Yadav, K.K., Kumaran, R., 2024. A state-of-the-art review of multilayer packaging recycling: challenges, alternatives, and outlook. J. Clean. Prod. 447, 141403.
Tejado, A., van de Ven, T.G.M., 2010. Why does paper get stronger as it dries? Mater. Today 13 (9), 42–49.
van Oss, C.J., Absolom, D.R., Neumann, A.W., 1980. The "hydrophobic effect": essentially a van der Waals interaction. Colloid Polym. Sci. 258 (4), 424–427.
Widom, B., Bhimalapuram, P., Koga, K., 2003. The hydrophobic effect. Phys. Chem. Chem. Phys. 5 (15), 3085–3093.
Widsten, P., Kandelbauer, A., 2008. Laccase applications in the forest products industry: a review. Enzym. Microb. Technol. 42 (4), 293–307.
Wright, S.L., Kelly, F.J., 2017. Plastic and human health: a micro issue? Environ. Sci. Technol. 51 (12), 6634–6647.
Yan, Y., Amer, H., Rosenau, T., Zollfrank, C., Dörrstein, J., Jobst, C., Zimmermann, T., Keckes, J., Veigel, S., Gindl-Altmutter, W., Li, J., 2016. Dry, hydrophobic microfibrillated cellulose powder obtained in a simple procedure using alkyl ketene dimer. Cellulose 23 (2), 1189–1197.