

Article

A More Environmentally Friendly Method for Pulp Processing Using DES-like Mixtures: Comparison of Physical Properties with Oxygen Bleached Pulp

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Abstract: The traditional papermaking process uses petroleum-based additives, which raise environmental concerns. As a result, these concerns have attracted the scientific community to explore green additives by introducing environmentally friendly cellulose modifications as additives to the papermaking process. A promising way to process pulp is the application of deep eutectic solvent-like mixtures, which expand new possibilities for delignification processes. This article aims to characterize the physical properties of pulps modified with deep eutectic solvent-like mixtures and to compare these properties to untreated softwood kraft pulp and pulp obtained after oxygen delignification (commercially available pulp; obtained from Mondi Štětí a.s.). The physical properties (mechanical and optical) of the original pulp and delignified pulps were evaluated based on the degree of beating (Schopper–Riegler degree), zeta potential, water retention value, tensile strength, modulus of elasticity, and whiteness. Technology employing deep eutectic solvent-like mixtures shows great promise for sustainable pulp production; however, its full-scale adoption will require further research focused on process optimization, solvent recovery, and economic cost reduction.



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1. Introduction

The increasing demand for energy and declining fossil fuel reserves are driving the modern community to seek clean and sustainable energy sources to offset non-renewable resources. Such a source is lignocellulosic material, or lignocellulosic biomass, which consists of a heterogeneous complex of cellulose, hemicellulose, and lignin. Lignocellulosic biomass is an abundant, renewable, and affordable source of energy. However, the structural complexity of lignocellulose and its poor solubility impede easy degradation. Different methods are commonly employed to degrade lignocellulose, but these methods require additional energy input, raising operating costs [1]. Components such as cellulose, hemicellulose, and lignin are strongly bonded through either covalent or hydrogen bonds, resulting in a rigid complex structure that limits the use of lignocellulosic materials. Pretreatment of lignocellulosic biomass plays a crucial role in valorizing its resistant structure [1,2].

In the pulp and paper industry, current developments focus on greener and more sustainable delignification and fractionation processes for woody biomass due to environmental and ecological concerns, as well as stricter regulatory conditions [2]. High-quality

cellulosic fibers are presently produced through conventional pulping processes such as soda, organosolv, sulfate (kraft), and sulfite, but these traditional methods have drawbacks, including high energy consumption and inconsistent cleavage of chemical bonds within woody biomass components [3]. For the advancement of the pulp and paper industry, it is essential not only to manufacture high-quality pulp but also to recover lignin efficiently for conversion into value-added products. More recently, alternative processes have gained popularity, aiming not only to recover lignin as a by-product but also to extract cellulose fibers [3,4].

A promising approach that emphasizes greenness and sustainability is the application of green oil solvents instead of conventional chemicals for lignocellulosic biomass delignification processes and pulp production. This approach has the potential to overcome some shortcomings associated with traditional delignification processes, such as extreme conditions (high pressure, high temperature, specialized equipment) or environmental, ecological, and economic concerns [3–5].

Green solvents, specifically deep eutectic solvent-like mixtures (DES-like mixtures), have emerged as promising technology. DES-like mixtures have potential applications in the pulp and paper industry. Other applications include the removal of pollutants from extraction processes, as well as the isolation and fractionation of lignocellulosic biomass [5]. DES-like mixtures are characterized by a significant decrease in the transition temperature (liquid–solid) compared to the melting temperatures of the individual substances that make up the mixture. DES-like mixtures typically consist of two or three inexpensive and safe components that can bind through hydrogen-bonding interactions. DES-like mixtures comprise a hydrogen bond acceptor and a hydrogen bond donor and remain in the liquid state at ambient temperature. DES-like mixtures have distinct advantages, including easy synthesis and low cost due to the renewable and readily available raw materials. Furthermore, DES-like mixtures exhibit benign properties, such as low toxicity and flammability, and customizable physicochemical properties [5–7]. In addition, DES-like mixtures have several disadvantages that limit their use on an industrial scale. The main disadvantages include high viscosity, low volatility, limited solubility for certain substances, sensitivity to moisture, and thermal instability [4–6].

The process of wood delignification and fractionation using DES-like mixtures has emerged as a promising green alternative to chemical pulping methods for obtaining cellulose fibers, lignin, and hemicelluloses, as well as polymers derived from them [7]. This study aims to determine and characterize the physical properties of unbleached softwood pulp processed via the kraft method and subsequently modified with DES-like mixtures containing choline chloride (ChCl) and lactic acid (LacA). ChCl is an inexpensive, biodegradable, and non-toxic quaternary ammonium salt that can be extracted from biomass or easily synthesized from fossil fuels. In combination with safe hydrogen bond donors such as lactic acid, ChCl quickly forms DES-like mixtures [8,9].

Several authors have reported on the delignification and fractionation of various types of lignocellulosic materials using DES-like mixtures [10–18], including their applications for pulp delignification (Table 1). Pérez et al. [9] examined the delignification of low-energy mechanical pulp (*Asplund fibers*) using DES-like mixtures based on ChCl/LacA. The authors utilized DES-like mixtures with ChCl as a hydrogen bond acceptor and LacA as a hydrogen bond donor due to their demonstrated effectiveness in biomass fractionation. The study analyzed different operating conditions such as reaction time, temperature, and the ratio of DES-like mixtures to wood biomass fibers. The results showed that a delignification time of 30 and 45 min resulted in a pulp yield of approximately 50% with a lignin content in the fibers of 14% and a fiber length of 0.6 mm. The delignification temperature had the most significant impact on pulp quality [9].

Table 1. Delignification of different pulps using DES-like mixtures and mechanical properties of pulps.

DES-like Mixtures	Biomass	Kappa Number (-)	Mechanical Properties (Beating, Tensile Index, Burst Index, Tear Index, Stiffness, Brightness)	Ref.
-	Untreated hardwood kraft pulp	21.7	30 °SR; 72.02 Nm/g; 7.3 km; 4.2 kPa.m ⁻² /g 7.1 mN.m ⁻² /g; 126 mN; 27.02%	[10]
ChCl/LacA (1:9) Alanine/LacA (1:9)	Hardwood kraft pulp	13.5 12.3	30 °SR; 62.49 Nm/g; 6.4 km; 3.6 kPa.m ⁻² /g 6.4 mN.m ⁻² /g; 131 mN; 34.05% 30 °SR; 63.00 Nm/g; 6.4 km; 3.6 kPa.m ⁻² /g 6.6 mN.m ⁻² /g; 130 mN; 33.38%	[10]
	Untreated hardwood kraft pulp	14.3	30 °SR; 67.28 Nm/g; 6.9 km; 4.2 kPa.m ⁻² /g 6.3 mN.m ⁻² /g; 123 mN; 31.09%	[11]
ChCl/Malic acid (1:1) ChCl/Oxalic acid dihydrate (1:1)	Hardwood kraft pulp	11.1 12.3	60 °SR; 49.61 Nm/g; 5.1 km; 2.2 kPa.m ⁻² /g 3.2 mN.m ⁻² /g; 100 mN; 38.31% 34 °SR; 67.89 Nm/g; 6.9 km; 3.8 kPa.m ⁻² /g 5.4 mN.m ⁻² /g; 128 mN; 33.81%	[11]

In this paper, the physical properties, specifically mechanical and optical properties, of unbleached softwood kraft pulp were investigated. The input pulp samples included untreated pulp, i.e., the original pulp, pulps modified with DES-like mixtures and pulp processed through oxygen delignification. DES-like mixtures were synthesized at molar ratios of 1:4; 1:5, and 1:6 using choline chloride (ChCl) and lactic acid (LacA). Information on the chemical composition of untreated and treated pulps modified with DES-like mixtures containing ChCl/LacA, as well as the results of the characterization of both the original pulp and those obtained after DES-like mixture application (Fourier transform infrared spectroscopy, X-ray diffraction analysis, optical microscopy, and others) was described in our previous study by Jančíková et al. [17]. This document serves as a continuation of that publication focusing on the characterization of the physical properties of pulps modified with DES-like mixtures and their comparison with untreated softwood kraft pulp and pulp processed through oxygen delignification. Regarding the physical properties, we focused on analyzing several factors, including the determination of the degree of beating (Schopper–Riegler method), zeta potential, streaming potential, conductivity, shrinkage, water retention value, and mechanical properties (tensile strength; rupture index; tensile energy absorption, tensile index, modulus of elasticity, breaking length, tensile stress, tensile overload, and elongation). Additionally, part of the work examined optical properties such as the whiteness of the sheets made from untreated pulp, pulp modified with DES-like mixtures, and pulp processed through oxygen delignification.

2. Materials and Methods

2.1. Materials and Chemicals

For the experiments conducted in this study, unbleached softwood kraft pulp from Mondi Štětí a.s. (Štětí, Czech Republic) was utilized, and the pulps were treated with DES-like mixtures composed of choline chloride (ChCl) and lactic acid (LacA). The synthesis of DES-like mixtures, as well as the modification process of softwood kraft pulp, was detailed in a study by Jančíková et al. [17]. Three DES-like mixtures were prepared by mixing a hydrogen bond acceptor (ChCl) with a hydrogen bond donor (LacA) at molar ratios of 1:4; 1:5, and 1:6. These mixtures were synthesized using a heating method. All chemicals were weighed according to their respective molar ratios. The DES-like mixtures were stirred and heated in a round-bottomed glass flask at 80 °C using a water bath for approximately 1 h, until ChCl was completely dissolved and a clear liquid formed. Regarding pulp treatment with DES-like mixtures, pulp samples (softwood kraft pulp; 20 g of absolutely dry material) were mixed with 200 g of ChCl/LacA-based DES-like mixtures, resulting in a solid/liquid ratio was 1:10. However, in one case, a solid/liquid ratio of 1:5 (or 1:6 at 80 °C) was used. The DES-like mixtures were placed in an oxygen reactor. The processing time was set to 60 min at 80 °C for all processes. The obtained pulps were then dried, weighed, and stored in a desiccator until further characterization and analysis. To benchmark the modification results achieved with DES-like mixtures, we also examined pulp from Mondi Štětí a.s., which had undergone oxygen delignification (O₂ delignification).

2.2. Pulping and Beating of Samples

The first step was the pulp swelling process, which lasted 12 h on the day before pulp beating. The pulping was conducted using a wet method in accordance with ISO 5263-1 [19]. The prepared test portion was pulped in a standard laboratory pulper at a speed of 30,000 rpm for all analyzed pulps. A total of 30 g of air-dried pulp was pulped in 2 L of water.

The next step was pulp beating, which took place between the roller blades of the Valley Hollander (Valley laboratory pulp beater), following ISO 5264-1 [20]. The pulp beating was scheduled for 15, 25, and 30 min. For beating, 210 g of pulp and 20 L of water were used. After beating, a sample of the suspension was taken and used for the next tests.

2.3. Forming, Pressing, and Drying of Sheets

Laboratory sheets were produced in a manual sheet former, following ISO 5269-1 [21]. Sheets were produced using a grammage of 2 g air-dried sample, derived from untreated softwood kraft pulp, pulp subjected to O₂ delignification, and pulp modified through the application of DES-like mixtures (with a target basis weight of 80 g/m²). Sheets were prepared for each beating time (15 min, 25 min, and 30 min). The laboratory sheets produced were used to measure the mechanical and optical properties of the pulps.

The removal of sheets from the screen of the laboratory sheet former was carried out using felts. Laboratory sheets formed on felts were stacked in a pile along with the felts. These stacked sheets, individually placed between felts, were pressed using a hand press (pressing time: 5 min). Following pressing, the sheets underwent unrestrained drying. After pressing, we removed the sheets from the felts and arranged them between metal screens before placing them in a drying oven at 30 °C until completely dry.

2.4. Methods for Measuring the Properties of Suspensions of Prepared Pulps

2.4.1. Determination of the Degree of Beating

The dewatering capacity of a pulp suspension in water is expressed in terms of the degree of beating or the Schopper–Riegler degree (°SR). The Schopper–Riegler test

measures the rate at which a dilute suspension of pulp can be dewatered. The dewatering ability depends on the surface condition and swelling of the pulp fibers. This test was conducted according to ISO 5267-1 [22]. For this procedure, 200 mL of suspension was collected from the Valley laboratory pulp beater, with the measurement repeated 3 times.

2.4.2. Analysis of the Zeta Potential and the Water Retention Value

The total zeta potential of a pulp fiber suspension arises from the charge of colloiddally dissolved particles on one side and the charge of the fibers on the other. In this work, the zeta potential of the pulp fibers was determined based on the Helmholtz–Smoluchowski equation using the FPA touch instrument. A fiber layer was formed on a sieve electrode within the measuring cell of the FPA analyzer under vacuum conditions.

The zeta potential serves as a key indicator for studying the physical stability of nanoparticles in a colloidal suspension, and its value (positive/negative) reflects this stability due to electrostatic repulsion [7]. During measurement, the charge clouds absorbed onto the fibers move with the liquid flow and generate a “streaming current potential”, as well as conductivity, which is measured by a sieve and ring electrode (AFG Analytic GmbH). Approximately 500 mL of the suspension was utilized to measure the zeta potential of the pulp fibers. Measurements were conducted five times, and the average values from these trials are reported in the results. The suspension sample was mixed between each measurement to ensure consistency.

The water retention value (WRV) analyzes the ability of fibers to retain water. The WRV tends to increase during processing due to internal fibrillation, the expansion of microscopic internal pores, and delamination—also called swelling—which occurs simultaneously with the development of external fibrils that enhance water retention. This test was carried out according to the SCAN-C 62:00 standard [23], with each pulp sample undergoing four measurements. The WRV is expressed as the ratio of the weight of retained after centrifugation, conducted under specified conditions, by a wet pulp sample (mm) to the weight of the same absolutely dry sample (ms) according to Equation (1):

$$\text{Water retention value} = \frac{\text{mm} - \text{ms}}{\text{ms}} \times 100\% \quad (1)$$

2.5. Measurement of Physical Properties of Pulp Sheets

2.5.1. Determination of Shrinkage

To assess the contribution of polymers to drying shrinkage and evaluate the dimensional changes in paper subjected to in-plane compaction, the shrinkage was calculated according to Equation (2), comparing the perimeter of a square marked by four punctured holes on the sheet, before and after drying:

$$\text{Shrinkage} = \frac{P_w - P_d}{P_w} \times 100\% \quad (2)$$

where P_w and P_d are the perimeters of the square in the wet and dry sheet, respectively.

2.5.2. Determination of Mechanical Properties

The mechanical properties of the laboratory sheets were measured following ISO 21940-2 [24]. The measuring device was tensile testing equipment. This standard outlines the measurement of the tensile strength and elongation at break, and specifies methods for determining the tensile index, tensile energy absorption index, and modulus of elasticity.

From each set of prepared laboratory sheets, 3 to 5 sheets were selected; then, the next step involved measuring the weight of the sheets, which allowed for the calculation of the basis weight. Sheets with similar basis weights were selected, and the thickness was

measured. Thickness measurements were conducted at six points around the perimeter of each sheet, and the average thickness was calculated.

To measure the mechanical properties of laboratory sheets prepared from untreated unbleached kraft pulp, pulp subjected to O₂ delignification, and pulps modified using DES-like mixtures, it was necessary to prepare the sheets accordingly. The selected sheets were cut precisely using a cutter. The width of the test strip had to be 15 mm, while the length was standardized to 100 mm, which corresponded to the distance between the jaws. The sheet samples had to be sufficiently long to facilitate secure clamping. For each group, 10 to 15 strips were measured. The values reported in the results represent the average values obtained from at least 10 tests (10 strips). The mechanical property values recorded in Table 4 were determined using standard TAPPI methods [25–27].

2.5.3. Determination of Optical Properties and Optical Microscopy

The optical properties of the laboratory sheets were measured in accordance with TAPPI T452 [27]. The measuring device used was a spectrophotometer. This standard outlines a general procedure for measuring the diffuse reflectance of all types of pulp, paper, and paperboard. Brightness is a commonly analyzed factor of the sample when exposed to blue light with specific spectral and geometric characteristics. The brightness method was originally developed to monitor pulp bleaching.

To measure the optical properties of laboratory sheets prepared from untreated unbleached kraft pulp, pulp modified with DES-like mixtures, and pulp subjected to O₂ delignification, one sheet was selected, ensuring that test areas were not touched directly. The measurement was performed using an Elrepho 070 device (Lorentzen & Wettre; Kista, Sweden) which automatically conducted measurements based on user-specified conditions—in our case, daylight D65 with a UV component. Each sheet underwent five measurements at different locations, and the average of the recorded values was calculated.

We used a Leica DM6 M optical microscope (Leica Microsystems (SEA) Pte Ltd.; Singapore). Dry samples were examined by placing them on a glass slide. The fibers were separated using tweezers to observe their characteristics. A 10× magnification objective was used for analysis.

3. Results and Discussion

3.1. Changes in the Properties of Prepared Pulps Suspensions

Characterization of the analyzed pulps facilitates the comparison and evaluation of changes resulting from modification, either through the application of DES-like mixtures or O₂ delignification. Table 2 presents information regarding the dewatering ability of the suspension (degree of beating). Additionally, the suspensions underwent tests to measure the zeta potential, along with determinations of conductivity and streaming potential. As illustrated in Table 2, an increase in beating time enhances the ability of the paper stock to absorb water, which subsequently raises the Schopper–Riegler degree [28]. During the beating process, fibers undergo separation, shortening, and simultaneous expansion of their surface area. As surface area increases, water penetration also intensifies, contributing to enhanced fiber swelling [28–30].

The zeta potential values for the original disintegrated pulp (unbleached softwood kraft pulp) ranged from -28.40 ± 0.66 mV to approximately -19.40 ± 0.50 mV for pulp ground for 30 min. As demonstrated, the zeta potential values rise with increasing beating time. A similar trend was observed for pulps modified using DES-like mixtures, where the zeta potential exhibited negative values (with decreasing negativity) in disintegrated pulps—conversely, as the time and degree of beating increased, the zeta potential values approached zero for all modified pulps (negative values increased).

Table 2. Schopper–Riegler degrees, zeta potential, conductivity, and streaming potential of original pulp, modified pulps using DES-like mixtures, and pulp after O₂ delignification.

Sample	Beating (°SR)	Zeta Potential (mV)	Streaming Potential (mV)	Conductivity (mS/cm)
Original pulp (disintegrated)	13	-28.40 ± 0.66	-0.606 ± 0.027	0.56 ± 0.02
Original pulp (grounded 15 min)	19	-20.20 ± 0.43	-0.425 ± 0.032	0.58 ± 0.00
Original pulp (grounded 25 min)	29	-19.40 ± 0.22	-0.422 ± 0.007	0.57 ± 0.01
Original pulp (grounded 30 min)	39	-19.40 ± 0.50	-0.421 ± 0.022	0.56 ± 0.01
ChCl/LacA (1:4; 80 °C, disintegrated)	11	-30.06 ± 0.77	-0.626 ± 0.020	0.60 ± 0.01
ChCl/LacA (1:4; 80 °C, grounded 15 min)	19	-20.06 ± 0.23	-0.443 ± 0.034	0.57 ± 0.00
ChCl/LacA (1:4; 80 °C, grounded 25 min)	32	-19.70 ± 0.30	-0.412 ± 0.014	0.58 ± 0.00
ChCl/LacA (1:4; 80 °C, grounded 30 min)	43	-19.50 ± 0.22	-0.420 ± 0.014	0.59 ± 0.01
ChCl/LacA (1:5; 80 °C, disintegrated)	12	-31.40 ± 0.60	-0.692 ± 0.020	0.49 ± 0.01
ChCl/LacA (1:5; 80 °C, grounded 15 min)	20	-22.40 ± 0.15	-0.502 ± 0.026	0.49 ± 0.01
ChCl/LacA (1:5; 80 °C, grounded 25 min)	30	-21.80 ± 0.30	-0.517 ± 0.024	0.49 ± 0.01
ChCl/LacA (1:5; 80 °C, grounded 30 min)	40	-21.30 ± 0.19	-0.496 ± 0.009	0.49 ± 0.00
ChCl/LacA (1:6; 80 °C, disintegrated)	12	-38.12 ± 0.84	-0.867 ± 0.050	0.42 ± 0.01
ChCl/LacA (1:6; 80 °C, grounded 15 min)	20	-25.10 ± 0.43	-0.568 ± 0.027	0.44 ± 0.01
ChCl/LacA (1:6; 80 °C, grounded 25 min)	30	-24.20 ± 0.30	-0.555 ± 0.018	0.44 ± 0.00
ChCl/LacA (1:6; 80 °C, grounded 30 min)	45	-24.40 ± 0.22	-0.580 ± 0.024	0.44 ± 0.01
O ₂ -delignified pulp (disintegrated)	11	-35.70 ± 0.19	-0.818 ± 0.030	0.47 ± 0.01
O ₂ -delignified pulp (grounded 15 min)	18	-25.50 ± 0.28	-0.584 ± 0.047	0.47 ± 0.01
O ₂ -delignified pulp (grounded 25 min)	30	-24.40 ± 0.15	-0.556 ± 0.013	0.48 ± 0.01
O ₂ -delignified pulp (grounded 30 min)	44	-24.20 ± 0.36	-0.551 ± 0.036	0.48 ± 0.01

Regarding pulp subjected to O₂ delignification, the zeta potential values ranged from -35.70 ± 0.19 mV for disintegrated pulp to -24.20 ± 0.36 mV for milled pulp for 30 min, with a beating degree of 44.

As mentioned previously, zeta potential was assessed alongside streaming potential and conductivity in this analysis. As observed in Table 2, the results for streaming potential

exhibited a trend similar to that observed in zeta potential measurements. Regarding conductivity, values remained constant across all analyzed pulps. Leading to the conclusion that the beating time, beating degree, modification using DES-like mixtures, or O₂ delignification did not significantly impact the conductivity of the given pulp suspensions (Table 2) [31,32].

An increase in pulp beating time correlates with a rise in the Schopper–Riegler degree (°SR). No significant differences were observed between pulps. The pulp modified with ChCl/LacA achieved slightly higher °SR values compared to the pulp processed for a longer beating time. This could suggest a higher mechanical impact on fibers, resulting from either beating or modification using ChCl/LacA [28–30]. However, when comparing the ChCl/LacA-modified pulp with the pulp bleached via O₂ delignification, the results were nearly identical. Additionally, similarities were observed in beaten pulp samples between ChCl/LacA (1:6)-modified pulp and pulp bleached with O₂ delignification.

We observe a decline in zeta potential values as the beating degree increases. During pulping, we note a more pronounced decrease in zeta potential compared to the values recorded during beating. Zeta potential serves as an indicator of colloidal system stability [30]. Particles with lower zeta potential values (approximately 0) tend to aggregate more readily, resulting in reduced stability [28,29]. Based on this observation, we suggest that an increase in zeta potential during beating may signal enhanced fiber fibrillation [31,32]. Fibers exhibit a greater tendency to bond, either with each other or with added additives.

In the case of pulp delignified with ChCl/LacA, we observed higher zeta potential values compared to the original pulp. This result is linked to an increase in LacA concentration. Pulp delignified with ChCl/LacA at a molar ratio of 1:6 achieves similar zeta potential values to pulp delignified with O₂.

The water retention value (WRV) was employed as an additional parameter for assessing fiber mechanical treatment [28]. WRVs fluctuate throughout the pulping and beating process. An increase occurs during beating, resulting from fiber separation and both internal and external fibrillation. External fibrillation was facilitated by the beating machine type—Valley Hollander [28,29,33]. Delignified pulps treated with ChCl/LacA exhibit slightly lower WRVs compared to original pulp and O₂-delignified pulps (Figure 1). This difference may stem from the distinct fiber characteristics induced by the varied delignification method. Fibers delignified with ChCl/LacA are less fibrillated, featuring defects such as shorter and more twisted fibers (Figure 1). These factors may explain the diminished pulp water retention capacity, reflected in the lower WRVs. In further processing, the extent of external fibrillation could influence the mechanical properties of paper [29,32].

Table 3 summarizes the results for WRV determination, showing that for the original disintegrated pulp, the WRVs were 1.38 ± 0.04 g/g. The original pulp, ground for 15 min with a beating degree of 19, exhibited WRVs of 1.86 ± 0.08 g/g. As beating time and the degree of beating increased, the WRVs for the original pulp rose to 2.21 ± 0.09 g/g (ground for 25 min) and 2.63 ± 0.18 g/g (ground for 30 min). Pulps modified with DES-like mixtures containing ChCl/LacA recorded WRVs of 1.35 ± 0.17 g/g (molar ratio 1:4); 1.11 ± 0.05 g/g (molar ratio 1:5); and 1.20 ± 0.07 g/g (molar ratio 1:6). These values apply to defibered pulps. Regarding pulps milled for different durations, and consequently subjected to varying beating degrees, Table 3 reveals a trend of increasing WRVs with longer beating time. The highest WRV was observed for pulp modification with a DES-like mixture of ChCl/LacA at a molar ratio of 1:6 and beaten for 30 min (2.59 ± 0.22 g/g).

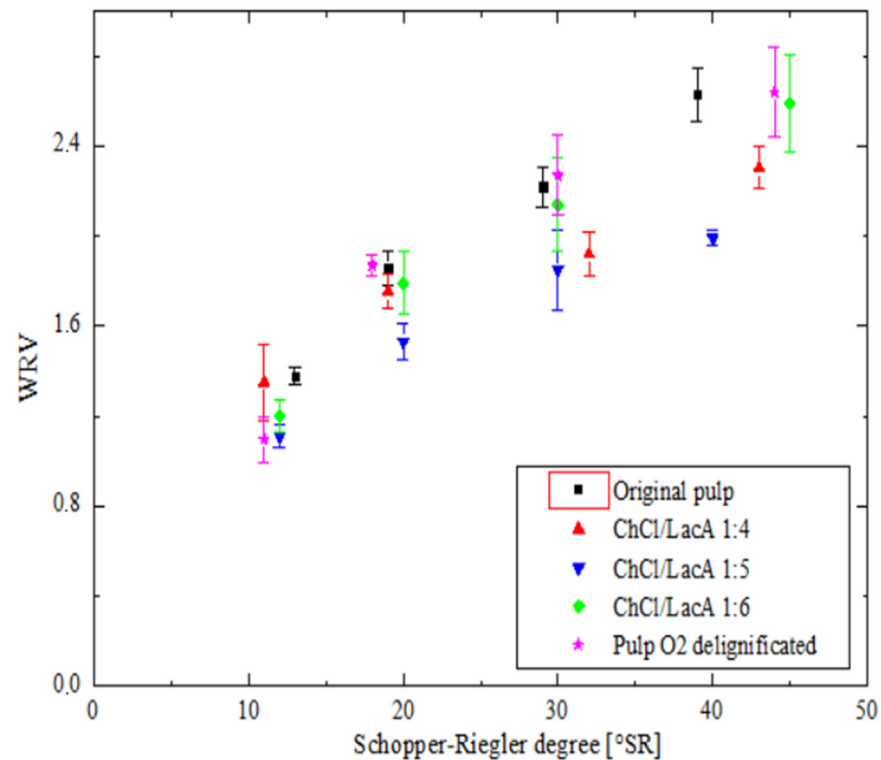


Figure 1. Schopper–Riegler degree vs. water retention value for individual suspensions of pulps analyzed.

Table 3. WRV of original pulp, modified pulps using DES-like mixtures, and pulp after O₂ delignification.

Sample	Beating (°SR)	WRV (g/g)
Original pulp (disintegrated)	13	1.38 ± 0.04
Original pulp (grounded 15 min)	19	1.86 ± 0.08
Original pulp (grounded 25 min)	29	2.21 ± 0.09
Original pulp (grounded 30 min)	39	2.63 ± 0.18
ChCl/LacA (1:4; 80 °C, disintegrated)	11	1.35 ± 0.17
ChCl/LacA (1:4; 80 °C, grounded 15 min)	19	1.76 ± 0.08
ChCl/LacA (1:4; 80 °C, grounded 25 min)	32	1.92 ± 0.10
ChCl/LacA (1:4; 80 °C, grounded 30 min)	43	2.30 ± 0.09
ChCl/LacA (1:5; 80 °C, disintegrated)	12	1.11 ± 0.05
ChCl/LacA (1:5; 80 °C, grounded 15 min)	20	1.53 ± 0.08
ChCl/LacA (1:5; 80 °C, grounded 25 min)	30	1.85 ± 0.18
ChCl/LacA (1:5; 80 °C, grounded 30 min)	40	1.99 ± 0.04
ChCl/LacA (1:6; 80 °C, disintegrated)	12	1.20 ± 0.07
ChCl/LacA (1:6; 80 °C, grounded 15 min)	20	1.79 ± 0.14
ChCl/LacA (1:6; 80 °C, grounded 25 min)	30	2.14 ± 0.21
ChCl/LacA (1:6; 80 °C, grounded 30 min)	45	2.59 ± 0.22
O ₂ -delignified pulp (disintegrated)	11	1.10 ± 0.10
O ₂ -delignified pulp (grounded 15 min)	18	1.87 ± 0.05
O ₂ -delignified pulp (grounded 25 min)	30	2.27 ± 0.18
O ₂ -delignified pulp (grounded 30 min)	44	2.64 ± 0.20

We also focused on determining the mechanical and optical properties of pulp delignified with O₂. Based on the results, we observed the same trend as in previously analyzed pulps: the lowest WRVs occurred in the defibered pulp, whereas the highest WRVs were found in the pulp with the longest beating time and highest beating degree (2.64 ± 0.20 g/g). This value is nearly identical to the WRVs achieved following the application of DES-like mixtures composed of ChCl/LacA at a molar ratio of 1:6. Shrinkage during drying has proven to be an effective method for enhancing the elastic properties of the paper [28,34]. Therefore, this technique is utilized to produce extensible paper. Moisture distribution is closely linked to paper shrinkage. As fibrillation intensifies, fibers absorb more water, facilitating increased shrinkage during drying [35]. This hypothesis was validated by the calculated shrinkage values. Pulp delignified with O₂ exhibited higher shrinkage values, confirming predictions based on the WRV results [35–37].

3.2. Mechanical and Optical Properties of Laboratory-Prepared Sheets from Different Pulp Suspensions

Most of the strength properties of paper are enhanced by pulp beating, as they depend on fiber bonding. Beating promotes fibrillation of the cellulose fibers, which expands the surface area of the fibers and strengthens the bonding between them in the final product. During beating, individual fibers are weakened and shortened due to cutting. When assessing the mechanical properties of laboratory sheets, it is possible to gather useful data for analyzing the quality of these sheets [28]. The data obtained for the original pulp (unbleached softwood kraft pulp), pulp modified with DES-like mixtures composed of ChCl/LacA at different molar ratios, and pulp subjected to O₂ delignification are presented in Table 4. For sheet samples, we observe that mechanical property values increase with extended beating times. Differences between analyzed pulps may result from variations in fiber length [28,38,39].

In the context of examining the mechanical properties of sheets produced from different pulp suspensions, we focused on determining tensile strength, the elastic modulus, and elongation. Regarding tensile strength, Table 4 displays the results, indicating that the tensile strength of the original defibered pulp measured 1.57 ± 0.09 kN/m. For the original pulp, ground for 15 min, 25 min, and finally 30 min, tensile strength values increased with beating time and degree, reaching 7.46 ± 0.55 kN/m (ground for 30 min). For defibered modified pulps, tensile strength values ranged from 0.89 ± 0.06 kN/m (molar ratio 1:4) to 1.10 ± 0.11 kN/m (molar ratio 1:6). As beating time and degree increased, the tensile strength of pulps modified with DES-like mixtures also rose, reaching approximately 4.52 ± 0.35 kN/m for a molar ratio of 1:4 (milled for 25 min), 4.65 ± 0.15 kN/m for a molar ratio of 1:5 (milled for 25 min), and 6.29 ± 0.20 kN/m for a molar ratio of 1:6 (ground 30 min). In terms of the tensile strength characterization for O₂-delignified pulps, we conclude from the results that these pulps exhibited the highest tensile strength of all analyzed samples, reaching 8.24 ± 0.43 kN/m (ground 30 min). By comparison, pulp modified with DES-like mixtures (ChCl/LacA; 1:6) achieved values comparable to O₂-delignified pulp (6.38 ± 0.34 kN/m; ground 15 min).

Another parameter examined in the analysis of mechanical properties in laboratory-prepared sheets was the elastic modulus. The results indicate that the elastic modulus for the original defibered pulp was 352.25 ± 45.95 MPa, and with an increase in beating degree and time, the elastic modulus rose to 786.59 ± 124.25 MPa (pulp ground for 25 min). However, as beating time increased to 30 min, the elastic modulus declined to 644.68 ± 113.54 MPa. A similar trend of increasing elastic modulus with beating time and degree was observed in the analysis of pulps modified with DES-like mixtures. For a molar ratio of 1:4 between ChCl/LacA, the elastic modulus increased from 269.75 ± 40.01 MPa (defibered pulp) to 711.85 ± 98.53 MPa (milled for 25 min). After further increasing the

beating time, the elastic modulus decreased to 632.03 ± 59.61 MPa, similar to that of the original pulp. Another system studied involved pulp modified with DES-like mixtures based on ChCl/LacA at different molar ratios, where the elastic modulus ranged from 288.92 ± 43.30 MPa (defibered pulp) to 644.19 ± 75.85 MPa (milled for 25 min). Subsequently, the elastic modulus dropped to 569.85 ± 81.15 MPa (milled for 30 min). For ChCl/LacA-modified pulp, elastic modulus values ranged from 235.69 ± 41.24 MPa (pulp- ing) to 710.50 ± 125.43 MPa (pulp ground for 30 min), where a decreasing trend at a higher beating times and degrees was not confirmed. Conversely, in the characterization of pulp delignified with O_2 , we observed a sharp increase in the elastic modulus, rising from 220.79 ± 26.30 MPa for pulped pulp to 751.39 ± 45.15 MPa for pulp ground for 15 min. Additionally, with an extended beating time and degree, the elastic modulus decreased to 481.64 ± 57.65 MPa for pulp ground for 30 min.

Table 4. Properties of original pulp, pulp delignified with O_2 , and modified pulp sheets with DES-like mixtures composed of ChCl/LacA at different beating levels.

Sample	Beating (°SR)	Tensile Strength (kN/m)	Modulus of Elasticity (MPa)	Elongation (%)
Original pulp (disintegrated)	13	1.57 ± 0.09	352.25 ± 45.95	3.00 ± 0.18
Original pulp (grounded 15 min)	19	5.98 ± 0.30	663.99 ± 56.44	6.16 ± 0.18
Original pulp (grounded 25 min)	29	7.72 ± 0.43	786.59 ± 124.25	7.39 ± 0.32
Original pulp (grounded 30 min)	39	7.46 ± 0.55	644.68 ± 113.54	9.08 ± 0.87
ChCl/LacA (1:4; 80 °C, disintegrated)	11	0.89 ± 0.06	269.75 ± 40.01	2.25 ± 0.15
ChCl/LacA (1:4; 80 °C, grounded 15 min)	19	4.21 ± 0.26	628.11 ± 65.10	5.83 ± 0.37
ChCl/LacA (1:4; 80 °C, grounded 25 min)	32	4.52 ± 0.35	711.85 ± 98.53	6.29 ± 0.43
ChCl/LacA (1:4; 80 °C, grounded 30 min)	43	4.36 ± 0.16	632.03 ± 59.61	7.36 ± 0.52
ChCl/LacA (1:5; 80 °C, disintegrated)	12	1.10 ± 0.09	288.92 ± 43.30	2.44 ± 0.24
ChCl/LacA (1:5; 80 °C, grounded 15 min)	20	4.32 ± 0.20	635.57 ± 105.68	5.61 ± 0.41
ChCl/LacA (1:5; 80 °C, grounded 25 min)	30	4.65 ± 0.15	644.19 ± 75.85	6.35 ± 0.37
ChCl/LacA (1:5; 80 °C, grounded 30 min)	40	4.34 ± 0.46	569.85 ± 81.15	7.08 ± 0.62
ChCl/LacA (1:6; 80 °C, disintegrated)	12	1.10 ± 0.11	235.69 ± 41.24	2.65 ± 0.34
ChCl/LacA (1:6; 80 °C, grounded 15 min)	20	5.52 ± 0.61	710.92 ± 111.42	6.49 ± 0.49
ChCl/LacA (1:6; 80 °C, grounded 25 min)	30	5.99 ± 0.36	695.69 ± 224.92	7.36 ± 0.33
ChCl/LacA (1:6; 80 °C, grounded 30 min)	45	6.29 ± 0.20	710.50 ± 125.43	8.68 ± 0.32
O_2 -delignified pulp (disintegrated)	11	1.13 ± 0.10	220.79 ± 26.30	3.81 ± 0.34
O_2 -delignified pulp (grounded 15 min)	18	6.38 ± 0.34	751.39 ± 45.15	7.29 ± 0.48
O_2 -delignified pulp (grounded 25 min)	30	8.06 ± 0.58	614.54 ± 52.61	8.82 ± 0.52
O_2 -delignified pulp (grounded 30 min)	44	8.24 ± 0.43	481.64 ± 57.65	10.72 ± 0.68

The mechanical properties of paper improve with an increasing degree of beating within the range of 0–45 °SR. This effect is attributed to the appearance of external fibrils, which function as bonding agents for inter-fiber adhesion [28,40–42]. As shown in Figure 2, the original pulp and O₂-delignified pulp exhibit higher tensile strength values than pulps delignified with ChCl/LacA. The closest tensile strength values to those of O₂-delignified pulp were achieved using ChCl/LacA at a molar ratio of 1:6. The tensile strength difference was approximately 2 kN/m. This may be due to fiber straightness, the increased presence of defects (small fragments, twisted fibers), and lower degree of fibrillation compared to O₂-delignified pulp. These characteristics were previously noted in the values reported in Section 3.1 [27–32].

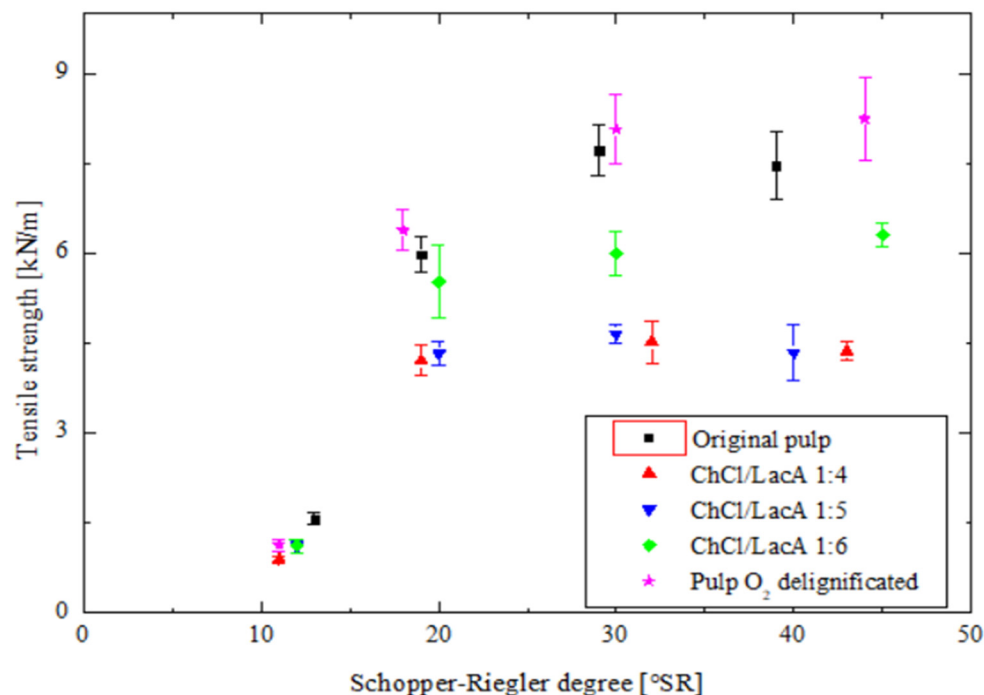


Figure 2. Effect of the degree of beating (Schopper–Riegler degree) on the tensile strength of laboratory-prepared sheets from different pulp suspensions.

We did not observe significant differences in paper elongation. Pulp delignified with ChCl/LacA at a molar ratio 1:6 exhibited elongation values similar to those of the original pulp. The highest elongation values were recorded for pulp delignified with O₂. These differences in elongation are not attributed to fiber length or external fibrillation but rather to shrinkage effects described in Section 3.1. This shrinkage influenced the observed paper elongation values (Figure 3) [43,44].

In this part of the publication, we also examined optical properties of laboratory-prepared sheets. Figure 4 presents the results of whiteness determination for sheets produced from various pulp suspensions, depending on the beating degree. Whiteness is an important paper characteristic, as it is often dictated by customer preferences. In this study, whiteness was additionally observed to assess the impact of different delignification methods. The original disintegrated pulp achieved a whiteness value of 72.88. An increase in whiteness of approximately one unit was noted when DES-like mixtures were applied. However, this whiteness increase was less pronounced compared to that observed with O₂ delignification. Following delignification with O₂, the pulp reached a whiteness value of up to 81.28. With increased beating time, whiteness decreased for all pulps by approximately seven units. This effect was attributed to beating, which causes fiber separation and releases excess lignin still present on the fibers [28,40,41].

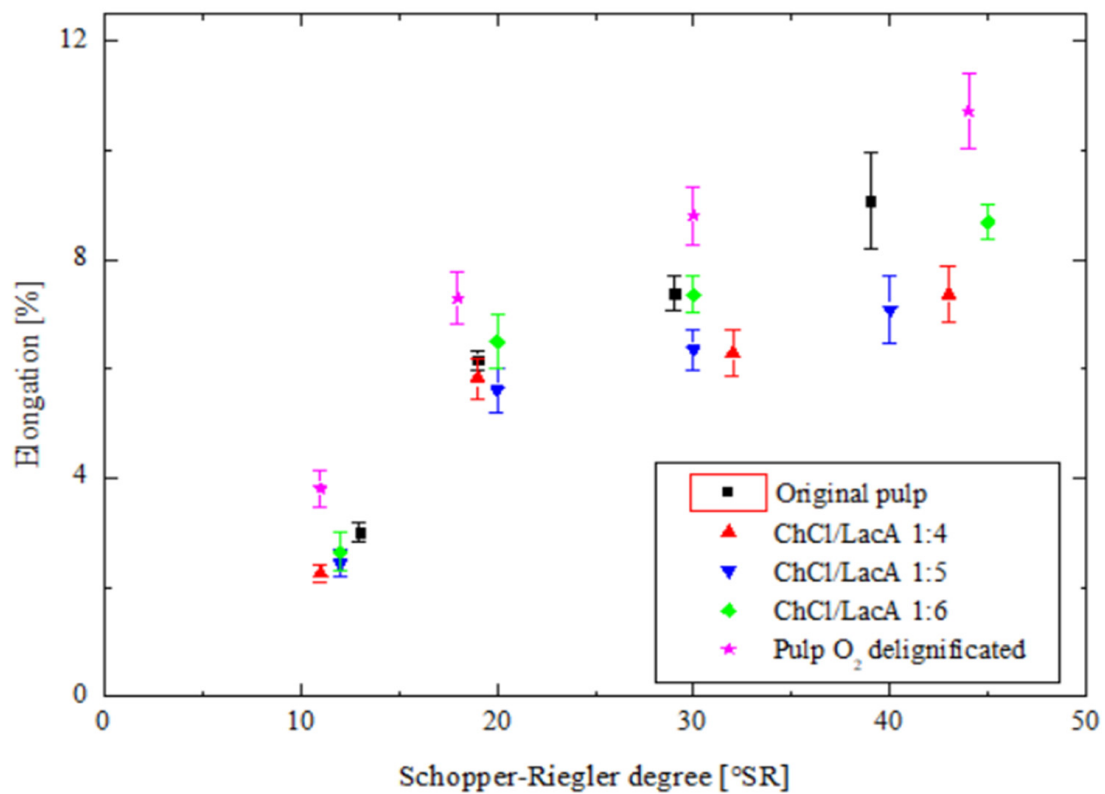


Figure 3. Schopper–Riegler degree vs. elongation for laboratory-prepared sheets from different pulp suspensions.

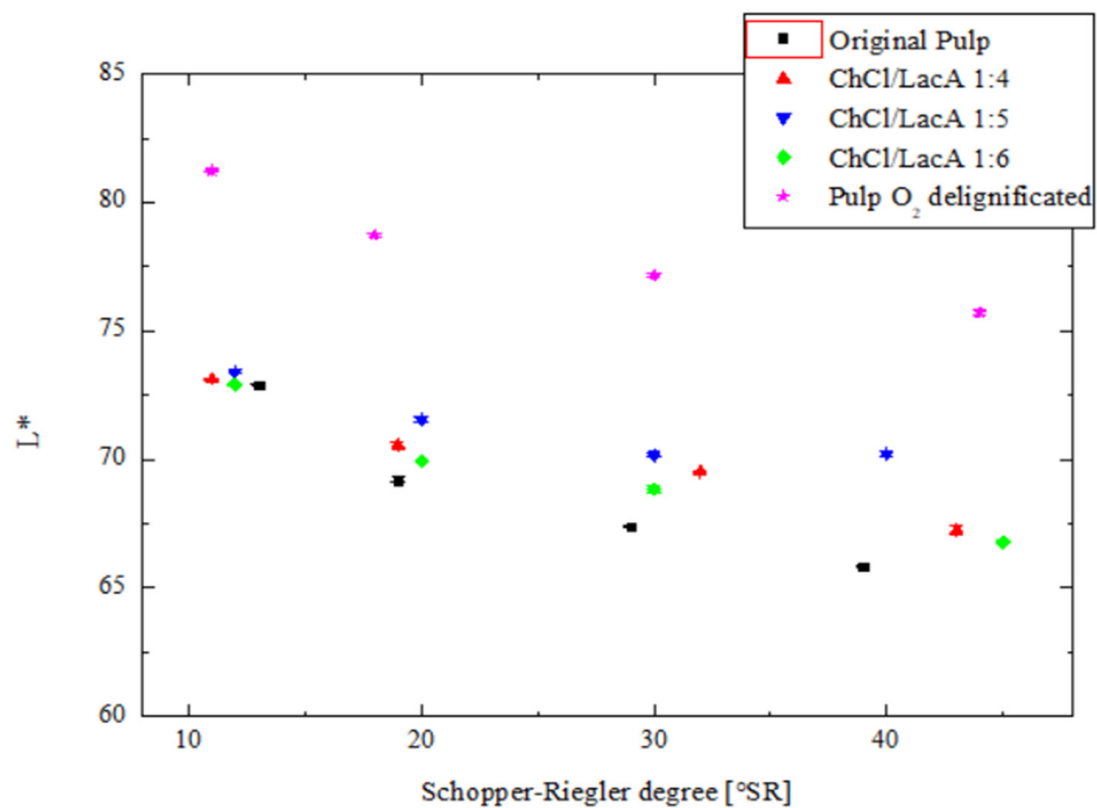


Figure 4. Whiteness values of laboratory-prepared sheets from different pulp suspensions vs. Schopper–Riegler degree.

After O₂ delignification, the lignin content, expressed by the Kappa number, was approximately 8.5. Following treatment with DES-like mixtures, Kappa number values for the pulps were 18.5 (the molar ratio between ChCl/LacA was 1:4; 80 °C); 19.3 (the molar ratio between ChCl/LacA was 1:5; 80 °C); and 20.88 (the molar ratio between ChCl/LacA was 1:6; 80 °C). These measurement results are discussed in more detail in the work by Jančíková et al. [17]. Given these findings, it was expected that the whiteness increase would not be as significant as in the pulp treated with O₂ delignification [16]. This decreasing whiteness was observed in both unbleached and modified pulps treated with DES-like mixtures.

3.3. Summary Overview

In the final part of this publication, we present a summary of the physical, mechanical or optical properties of the analyzed pulps, including unbleached softwood kraft pulp, pulp modified with DES-like mixtures containing ChCl/LacA at different molar ratios, and O₂-bleached pulp.

The summary in Table 5 compares the various pulps and their primary characteristics at a beating degree of 30 °SR [42–45]. Microscopy images reveal that pulp delignified with O₂ exhibits greater external fibrillation of fibers. Pulps delignified with ChCl/LacA also display fibrillation; however, an increase in the fine fraction is observed. Despite this, fibers delignified with ChCl/LacA retain a visual character similar to that of the original pulp.

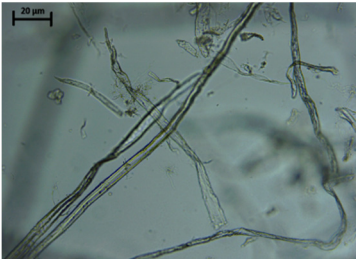
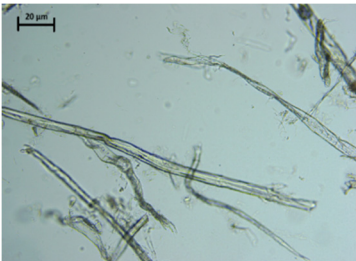
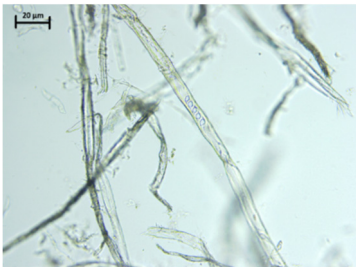
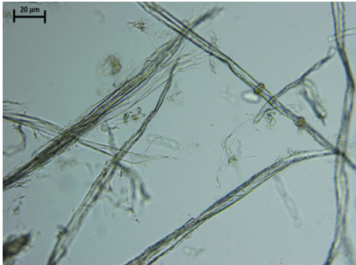
The original pulp exhibited a WRV of 2.25. Pulp delignified with ChCl/LacA at molar ratios of 1:4 and 1:5 showed lower WRVs compared to the original pulp. The ChCl/LacA (1:6) pulp reached a WRV of 2.14. Compared to the original pulp and the O₂-delignified pulp, which exhibited WRVs of 2.27, we observed a slight decrease. However, this reduction is not considered significant and may be related to lower fiber fibrillation, potentially impacting the mechanical properties of paper sheets. This is evident in tensile strength values. Pulp modified with ChCl/LacA exhibited lower tensile strength compared to both the original pulp and O₂-delignified pulp. The tensile strength difference between the O₂-delignified pulp and ChCl/LacA (1:6)-modified pulp was approximately 2 kN/m. Although this difference may not be substantial, its impact depends on the final applications.

The original pulp exhibited a zeta potential value of −19.6 mV. This value is comparable to that of pulp delignified with ChCl/LacA (1:4). For the remaining pulps, a noticeable change in zeta potential was observed, with value decreasing as LacA concentration increased in the DES-like mixtures. Pulp delignified with ChCl/LacA (1:6) reached a zeta potential value of −24.2 mV, while when delignified with O₂, we obtain a zeta potential value of −24.4 mV. This result is promising as it suggests that by replacing O₂ delignification with DES-like mixtures, a pulp with comparable characteristics can be obtained.

Whiteness values can also be observed in Table 5. All pulps in which delignification was performed achieve higher values of the L* parameter. The pulp after O₂ delignification achieves almost 10 units of L* increase over the original pulp. For pulps delignified with ChCl/LacA, we also observed an increase in the whiteness of the paper sheets. However, this increase is a maximum of 4 units L*.

The application of DES-like mixtures in pulp modification presents a promising alternative to O₂ delignification. Thus, DES-like mixtures appear to be a suitable replacement for O₂ delignification after kraft cooking in the future [11–14]. These mixtures offer straightforward integration into the pulp production process [43,44]. Due to their low environmental impact, DES-like mixtures provide an eco-friendly alternative to conventional pulping methods. Moreover, DES-like mixtures are cost-effective and biodegradable, making them an economically and ecologically viable option for pulp delignification [45,46].

Table 5. Physical characteristics of analyzed pulp suspensions and laboratory-prepared sheets with constant degree of beating.

Beating (30 °SR)	Optical Microscopy	WRV (g/g)	Zeta Potential (mV)	Tensile Strength (kN/m)	L* D65/10
Original Pulp		2.25	−19.6	7.69	66.72
ChCl/LacA (1:4; 80 °C)		1.90	−20.0	4.52	70.21
ChCl/LacA (1:5; 80 °C)		1.85	−21.8	4.65	70.82
ChCl/LacA (1:6; 80 °C)		2.14	−24.2	5.99	67.99
O ₂ -delignified pulp		2.27	−24.4	8.06	76.64

4. Conclusions

In this work, we examined the properties of pulp suspensions and laboratory sheets modified with DES-like mixtures containing ChCl/LacA at molar ratios of 1:4, 1:5, and 1:6, and compared them to the properties of pulp subjected to O₂ delignification. In

characterizing the pulp suspensions, we focused on parameters such as the Schopper–Riegler degree, water retention value, and zeta potential. Using the pulp suspensions, we then produced laboratory sheets, on which we measured physical properties, specifically mechanical and optical properties.

The effect of modifying pulp fibers after applying DES-like mixtures and O₂ delignification was compared at 30 °SR. This comparison was conducted because paper properties are most frequently assessed at this beating degree. The results indicated the greatest similarity between oxygen delignification and DES-like mixtures based on ChCl/LacA (1:6). Pulp delignified with ChCl/LacA (1:6) exhibited a WRV of 2.14. This value was slightly lower than that observed after O₂ delignification, which reached 2.27. This minor difference in WRVs is attributed to an increase in the fine fraction within the pulp suspension and reduced fibrillation after delignification with ChCl/LacA, as confirmed by microscopic images. The zeta potential measurements were promising, as the pulp delignified with ChCl/LacA (1:6; −24.2 mV) yielded results nearly identical to those of O₂-delignified pulp (−24.4 mV). For mechanical properties of the laboratory sheets, we assessed tensile strength. Laboratory sheets delignified with ChCl/LacA exhibited a tensile strength of 5.99 kN/m, while sheets treated with O₂ delignification reached 8.06 kN/m. This difference was more pronounced and can be attributed to the increased fine fraction in the pulp.

Among the applied DES-like mixtures based on ChCl/LacA, the best results in terms of physical properties were obtained using a molar ratio of 1:6 (ChCl/LacA) when compared to O₂-delignified pulp. While other DES-like mixtures contributed to the enhancement of mechanical or optical properties in laboratory-prepared sheets, their effects were not as substantial as those observed with the ChCl/LacA (1:6) mixture. Further studies should explore additional molar ratios between ChCl/LacA, as well as investigate increasing processing temperature or reducing modification time at higher temperature.

DES-like mixtures have been intensively investigated in recent years as an alternative to traditional pulp production methods, such as the kraft process (alkaline delignification), the sulfite process, and oxygen delignification as part of the bleaching sequence. These mixtures hold great potential for sustainable pulp production, particularly within low-emission and bio-oriented processes. However, on an industrial scale, they have yet to surpass oxygen delignification in terms of efficiency, cost-effectiveness, and proven reliability. While DES-like mixtures offer advantages from a green chemistry and specialized application standpoint, technological and economic obstacles must be addressed before they can replace conventional methods in large-scale pulp production. Although oxygen delignification is inexpensive, fast and well-established, DES-like mixtures have yet to gain widespread adoption in industrial settings. Nonetheless, improvements in viscosity reduction, recovery efficiency, and optimized reaction conditions may enable DES-like mixtures to provide more selective and sustainable solutions for lignocellulosic biomass processing in the future.

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