Optimization of the Properties of Poplar and Willow Chemimechanical Pulps by a Mixture Design of Juvenile and Mature Wood

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Forest plantations of Salicaceae (poplars and willows) in Argentina are mainly used for the manufacture of pulp for newsprint. The rapid growth of these species results in a decrease in rotation age, which increases the proportion of juvenile wood. The aim of this work was to define the proportions of juvenile wood (JW) and mature wood (MW) of these species that can optimize the mechanical and optical properties of chemimechanical pulps for newsprint production. A two-component mixture type experimental design was used with proportions (JW:MW) of 0:100%, 25:75%, 50:50%, 75:25%, and 100:0%. When the mechanical properties were optimized, the highest desirability function was obtained with a JW:MW ratio of 100:0%, and the optimal ratio for optical properties was 0:100%. The pattern of variation of mechanical properties can be attributed to the higher density of MW, whilst that of the optical properties can be attributed to the higher content of extractives in the JW.

Keywords: Salicaceae; Chemimechanical pulp; Juvenile wood; Mature wood; Mixture design

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INTRODUCTION

Besides *Eucalyptus*, the most cultivated broadleaf trees in Argentina are members of Salicaceae (*Salix* and *Populus*). Genetic improvements have achieved, among other objectives, increased productivity, better quality of wood (for different uses) and pulp, crop adaptation to marginal areas, and cost reduction (Zobel 1961; Einspahr 1970; Goyal *et al.* 1999; Roth *et al.* 2007, Vermaak 2007). *Populus deltoides* and *P. nigra* should be mentioned because of their ease of hybridization, their adaptability to temperate and subtropical regions, and their ability to propagate vegetatively (Cortizo 2011). *P. alba* hybrids are also common in Argentina, whereas the most common clones and hybrids of willows are derived from *Salix babylonica* and *S. alba* (Norberto 2005). Most plantations are destined for the manufacture of chemimechanical pulp for newsprint (Monteoliva *et al.* 2007; Villegas *et al.* 2008, 2009, 2012; Villegas and Area 2009).

The cultivation of Salicaceae is so extensive because they are very versatile in terms of potential growth sites. There are species and clones adapted to both waterlogged soils and drought conditions. Their rapid growth produces trees reaching commercial size at an early age of 10 to 12 years. This decrease in rotation age increases the proportion of juvenile wood. The literature indicates that juvenile wood is associated with cambial cells of early physiological age and has lower density, shorter fibrous elements, and lower strength properties compared with mature wood (Hernández *et al.* 1998; Zobel and Sprague 1998; DeBell *et al.* 2002; Yu *et al.* 2008). The general trend indicates lower values of certain attributes (*i.e.* density, fiber length, microfibril angle) in the first ring, increasing relatively quickly for a few years, and then stabilizing or increasing gradually in the mature wood (Fang *et al.* 2006; Debell *et al.* 2002).

When kraft pulps made from three aspen (*Populus* spp.) clones grown under short rotation intensive culture were compared with pulps made from mature debarked native aspen stemwood at comparable delignification degrees, the clones gave slightly lower pulp yields and required less refining energy to develop acceptable handsheet strength than the mature aspen. Handsheets from clones grown for 5 or 7 years had similar (or even superior) strength properties to those from mature aspen (Zarges *et al.* 1980). In kraft pulping trials using 8-year-old *Populus* hybrids, the papermaking properties did not change significantly after the age of 7 years. This finding was corroborated by the fact that once the trees are 8 years old, the amount of wood having long fibers (0.9 mm and longer) is approximately four times the volume of the wood containing short fibers (0.7 mm) (Goyal *et al.* 1999). Studies on *Populus tremuloides* kraft pulping, neutral sulfite semichemical (NSSC) pulping, thermomechanical pulping (TMP), and chemithermo-mechanical pulping (CTMP) processes demonstrated that pulps produced from chips of juvenile wood pulps produced from chips of juvenile wood had lower mechanical properties than those from mature wood (Myers *et al.* 1996).

The strength, optical, and printing properties of mechanical pulps can be attributed to their complex composition (fibers, fines, and fiber bundles) and by the function of each fibrous element in the web formation. Pulp strength values are related to the ratio between the different fibrous elements, the fibrillation of fibre walls, the average fiber length, the shape of the particles, and the proportion and nature of fines (Forgacs 1963; Reme and Helle 1998; Broderick *et al.* 1999; Olander *et al.* 2005; Monteoliva *et al.* 2008). In addition to the cutting and fibrillation effects, refining also produces alterations in the fiber, such as curls and kinks, related to the bending of the fiber.

In previous work, the authors have built models of axial and radial variations of the anatomical characteristics, chemical properties, and wood density of *Populus deltoides* A129/60 (Australian poplar) (Cobas *et al.* 2008; Diaz *et al.* 2010; Cobas and Monteoliva 2011; Cobas *et al.* 2013) and *Salix babylonica* var. *sacramenta* (American willow) (Monteoliva *et al.* 2002; Monteoliva *et al.* 2005; Monteoliva *et al.* 2006).

The aim of the present work was to define the proportions of juvenile wood (JW) and mature wood (MW) of *Populus deltoides* and *Salix babylonica* var. *sacramenta* that optimize mechanical and optical properties of chemimechanical pulps for newsprint.

EXPERIMENTAL

Materials

Five 17-year-old *Populus deltoides* ("129-60 Australia" clone) trees and five 45-year-old *Salix babylonica* var. *sacramenta* (American willow) trees in good sanitary conditions were sampled from a commercial plantation located in the 4th Section of the Delta Islands, in the Province of Buenos Aires, Argentina (34 ° 30' Lat. South, 59 ° 00 'West). The plantation, for which the intended usage was for limination, was implanted with a spacing of 5 m x 4 m. A high mountain system was used, with two prunings up to

7 m high. The harvesting was set between 15 and 22 years. Samples were taken at five heights from the trunk: 0.3 m, 1.3 m, 4.2 m, 8.1 m, and 15.9 m.

Methods

The basic density of the wood was determined by the displacement of fluid technique (IRAM 9544). For the determination of fiber length (μm), 60 fibers were measured on growth rings of each cambial age per tree on digital images taken with an optical microscope (total measurements n = 300, considering age and height). Fibers were macerated by use of Franklin solution (1:1 glacial acetic acid and hydrogen peroxide 130 volumes, *i.e.* under standard conditions 130 volumes of oxygen would be released by one volume of solution). Fiber width, lumen, cell wall thickness, and vessels number were measured on cross-sectional slices by optical microscopy. Chemical components were quantified according to NREL-LAP (National Renewable Energy Laboratory-laboratory analytical procedure) standards, including: total solids and moisture (NREL/TP-510-42621), lignin soluble and insoluble in acid and structural carbohydrates: glucans, xylans, and arabinans, acetyl groups (NREL/TP-510-42618). HPLC determinations were performed with a Waters chromatograph, using an AMINEX-HPX87H (BIO-RAD) column, with the conditions: eluent: H_2SO_4 4 mM, flow: 0.6 mL/min, temperature: 35 °C, and detector: refraction index and diode array. Extractives content in water and alcohol were determined according to TAPPI standards.

The analysis of variations in the stem was performed radially at 1.3 m for all properties, and axially for fiber length and wood density. The age of transition from juvenile to mature wood in both species was determined through segmented regression analysis applied to the radial pattern of each property (density, anatomical, and chemical properties) at all sampling heights of the stem. This method assumes that in the radial pattern of the stem (associated with the age of the growth ring) there is a distinct change in the slope of the regression line of the property under consideration corresponding to the age of transition. To conduct the study, the regression model for overall segments and the models for portions of juvenile and mature wood, defined by Tasissa and Burkhart (1998) were applied. The Piecewise Linear Regression was used for the analysis (Breakpoint Regression, Statistica V6). Further details can be found in Cobas *et al.* (2013).

A representative sample of all trees at every studied height was chipped for each species, separating juvenile wood from mature wood. Chips approximately 2.5 cm long × 1.5 cm wide × 0.3 cm thick were handmade, avoiding parts with rotting or knots.

To determine which mixture optimized each studied property, a two-component mixture experimental design was applied. In this kind of design, the proportions of the ingredients, which are not independent since the sum has to be 1 or 100%, determine the properties of the mixture. The proportions of juvenile and mature wood (JW:MW) defined by the experimental design were 0:100%, 25:75%, 50:50%, 75:25%, and 100:0%.

The chips were exposed to steam at atmospheric pressure for 40 minutes before pulping. Chemical treatment was carried out in a 7 L laboratory digester (M/K System Inc.) under the following conditions: liquor-to-wood ratio: 5.5/1, Na_2SO_3 and NaOH: 2.6% (oven dry basis), cooking temperature: 80 °C, and time at temperature: 20 min.

The defibration and refining of chips (two refining stages) were performed in a Bauer atmospheric refiner of 5 HP with discs that were 8 inches in diameter, in a closed water circuit with fines recirculation. The pulps were screened in a Somerville-type of screen with slots of 0.15 mm (described in TAPPI Standard T 275 sp-02) for 20 minutes with fines recirculation. The shives were oven dried and weighed as rejects. Before mixing, pulps from juvenile and mature poplar wood had to be further refined with 600 and 1100 revolutions, respectively, in a PFI mill to reach 45 degrees SR. Regarding willow pulps, mature wood pulp had to be PFI refined with 120 revolutions, whereas juvenile wood pulp did not need to be further refined.

The test handsheets were manufactured according to TAPPI T205 sp-95, with water re-circulation to avoid loss of fines. Mechanical properties were determined according to TAPPI test methods and optical properties according to ISO 3688 standards (1977). The results were analyzed using Statgraphics software at 0.05% significance.

RESULTS AND DISCUSSION

In poplar, the estimated age of transition between juvenile and mature wood was not identical for all properties. It was 4, 5, 7, or 9 years, depending on the variable (vessels, fibers, or density). The maturation sequence of the fibrous characteristics in this species was vessel diameter, wall area, vessel frequency, fiber width, fiber length, density, and wall thickness of the fibers. In American willow, the fiber length and vessel diameter were the first characters to mature (5 to 10 years), whereas other properties had a transition age in the range of 10 to 15 years. The selected ages presented the greatest frequency in all analyzed wood characters and tree heights. The age of transition for poplar was 9 years, whereas it was 10 to 15 years for willow.

The anatomical and chemical characteristics of juvenile wood (JW) and mature wood (MW) of poplar and willow are shown in Table 1 (means and standard deviations, SD). Chemical composition is expressed on an oven-dry basis (% o.d.).

Table 1. Anatomical and Chemical Characteristics of Juvenile Wood (JW) and Mature Wood (MW) of Poplar and Willow

Species	Poplar				Willow			
Kind of wood	JW (mean)	JW (SD)	MW (mean)	MW (SD)	JW (mean)	JW (SD)	MW (mean)	MW (SD)
Density (g cm^{-3})	0.362 a	0.01	0.388 b	0.00	0.414 a	0.01	0.424 a	0.01
Fiber length (μm)	1262 a	68.61	1495 b	64.24	1623 a	32.51	1618 a	12.22
Fiber width (μm)	20.5 a	0.90	21.2 a	0.53	17.8 a	1.68	19.1 a	0.56
Lumen (μm)	13.7 a	0.85	13.7 a	0.47	12.8 a	1.08	13.8 a	1.12
Cell wall thickness (μm)	3.2 a	0.15	3.5 b	0.26	3.25 a	0.20	4.48 a	0.37
Vessel diameter (μm)	75.0 a	4.59	78.1 a	0.96	65.6 a	2.05	64.1 a	5.05
Vessel frequency (nº mm^{-1})	54.5 a	9.44	56.4 a	8.90	59.9 b	1.61	53.5 a	5.11
Lignin (% o.d.)	25.7 a	0.17	25.7 a	0.16	25.4 a	0.83	25.3 a	0.76
Hemicelluloses (% o.d.)	19.3 b	0.61	18.6 a	0.28	16.6 a	1.33	18.6 b	0.90
Cellulose (% o.d.)	47.8 a	0.93	48.6 a	0.15	44.8 a	2.60	43.1 a	2.43
Extractives (% o.d.)	4.94 a	0.33	4.53 a	0.34	5.48 a	0.27	5.79 a	0.44

Letters must be read horizontally. Different letters indicate significant differences ($p < 0.05$) between JW and MW from each species using Tukey's test.

Uronic acids were not included in HPLC determination of carbohydrates. This may be the reason why the values do not add up to 100%. Additionally, the content of hemicelluloses in the mature wood was higher than in the juvenile wood, especially in willow. Statistical analysis showed that in poplar, density, fiber length, and cell wall thickness in the juvenile wood were significantly lower, whereas the hemicelluloses content was significantly higher than the mature wood ($p < 0.05$). In the case of willow, vessel frequency was significantly higher and hemicelluloses content was lower in juvenile wood than in mature wood. The rest of the microscopic and chemical properties did not present significant differences in either species (Tukey's test in Table 1).

Since the studied willow and poplar were of different ages, only the properties of juvenile wood of both species were compared. One factor ANOVA statistic evaluation indicated that density ($p = 0.000$), cell wall thickness ($p = 0.030$), and extractives ($p = 0.021$) in the JW of poplar were significantly lower than those characters in the JW of willow. On the other hand, fiber length ($p = 0.000$), fiber width ($p = 0.000$), vessels diameter ($p = 0.003$), hemicelluloses ($p = 0.003$), and cellulose ($p = 0.043$) in the JW of poplar were significant higher than in the JW of willow. Fiber lumen and vessel frequency did not present significant differences between species. The results of some physical, mechanical, and optical properties of pulps from mixtures of juvenile wood (JW) and mature wood (MW) of poplar and willow are shown in Tables 2 and 3, respectively.

Table 2. Physical, Mechanical, and Optical Properties of Pulps from Mixtures of Juvenile and Mature Wood of Poplar

Mixture (%) JW:MW	0:100	25:75	50:50	75:25	100:0
Bulk (cm^3/g)	2.38	2.48	2.42	2.32	2.01
Air resistance (s)	9.2	10.1	14.1	24.3	60.3
Tear index (mN m^2/g)	3.23	3.28	4.16	3.96	4.40
Tensile index (Nm/g)	22.6	25.4	26.7	31.5	35.6
Elongation (%)	1.1	1.2	1.2	1.3	1.4
TEA index (J/g)	0.15	0.19	0.21	0.27	0.32
Z span index (Nm/g)	51.3	55.0	61.5	63.1	66.7
Brightness (% ISO)	50.8	49.0	46.5	44.9	41.0
Opacity (%)	93.3	94.5	94.8	94.4	93.5
Light absorption k (m^2/kg)	3.3	3.7	4.1	4.2	4.3
Light scattering s (m^2/kg)	42.3	43.3	42.4	41.0	35.0

Table 3. Physical, Mechanical, and Optical Properties of Pulps from Mixtures of Juvenile and Mature Wood of Willow

Mixture (%) JW:MW	0:100	25:75	50:50	75:25	100:0
Bulk (cm^3/g)	2.45	2.48	2.42	2.32	2.33
Air resistance (s)	7.2	6.6	7.2	8.8	8.8
Tear index (mN m^2/g)	3.47	3.36	3.97	4.53	4.60
Tensile index (Nm/g)	17.8	17.3	18.6	20.4	20.3
Elongation (%)	1.1	1.0	1.0	1.1	1.3
TEA index (J/g)	0.12	0.11	0.12	0.14	0.17
Z span index (Nm/g)	45.3	48.1	48.8	50.5	49.8
Brightness (% ISO)	31.5	28.7	26.6	25.2	23.5
Opacity (%)	99.3	99.4	99.6	99.7	99.5
Light absorption k (m^2/kg)	13.7	15.7	18.0	18.1	18.3
Light scattering s (m^2/kg)	36.7	34.0	33.5	31.3	26.9

The resulting physical, mechanical, and optical pulp properties of both species can be partially explained by some wood properties (Tables 1, 2, and 3). Tensile index correlated positively with fiber width and vessel diameter (both $p < 0.005$), probably because flexible cell elements collapsed, increasing bonding. Air resistance correlated positively with wood density and fiber length (both $p < 0.000$), since a high wood rigidity may produce more fines in a chemimechanical pulp, closing the sheet web. Opacity correlated positively with wood density, fiber length, and cell wall thickness, but negatively with fiber width and vessel diameter (all $p < 0.000$). This is typical behavior, due to these fiber characteristics' impact on fines production. Although the chemical composition seems to not have a direct influence on the physical properties of these chemimechanical pulps, the content of extractives correlated positively with opacity and with the light absorption coefficient and negatively with brightness, since extractives color the pulp.

Differences between pulps from mixtures of poplar and willow JW and MW were detected by a multivariate analysis of variance. The considered factors were the genus (*Populus* and *Salix*) and the JW:MW ratio. On average, the tensile index, elongation, TEA index, zero span, brightness, and the light scattering coefficient *s*, were significantly higher for poplar wood pulps, whereas opacity and the light absorption coefficient *k* were significantly higher for willow wood pulps. There were no significant differences between species in bulk, air resistance, and tear index. Considering pulps of both species, the JW:MW ratio did not affect air resistance, opacity, or *k*, but it produced significant differences in the mechanical properties, showing a tendency of progressive increase from 0% to 100% of juvenile wood. Bulk, brightness, and *s* properties showed the opposite behavior.

The equations of fitted mathematical models for each property are presented in Table 4 and 5, respectively. The linear model has linear terms for each component, whereas the quadratic model includes linear, first-order interactions, and quadratic terms. The cubic model adds third-order terms.

Table 4. Equations Obtained for the Physical, Mechanical, and Optical Properties of Pulps Made from Mixtures of Juvenile and Mature Poplar Wood

Property	Equation*	R^2
Bulk	2.02*JW + 2.38*MW + 0.98*JW*MW	99
Air resistance	57.797*JW + 11.277*MW - 87.337*JW*MW	97
Tear index	4.41*JW + 3.202*MW	96
Tensile index	34.78*JW + 21.94*MW	82
Elongation	1.392*JW + 1.096*MW	99
TEA index	0.3126*JW + 0.1446*MW	96
Z span index	67.27*JW + 51.726*MW	97
Brightness	41.7*JW + 51.18*MW	98
Opacity	93.46*JW + 93.34*MW + 5.6*JW*MW	99
Light absorption *k*	4.2914*JW + 3.2914*MW + 1.0286*JW*MW	99
Light scattering *s*	35.349*JW + 42.109*MW + 16.571*JW*MW	97

* JW and MW vary between 0 and 1

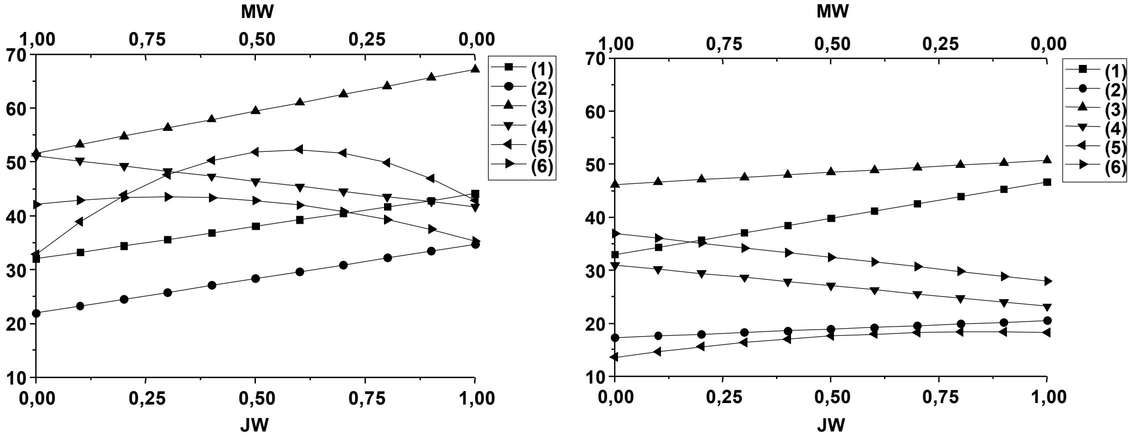
In the equations, the values of the components are specified as pseudo-components. The pseudo-component approach involves rescaling each component, making it equal to 0 at its minimum value, and equal to 1 at its maximum value. The sum of the pseudo-components must equal 1 in each experimental run. There are no equations for air resistance and opacity, as their values did not show significant differences. The equations did not fit as well in the case of willow as they did in the case of poplar (R^2 in Tables 4 and 5). The mathematical models can be used to determine which combination of factors improves the performance of a property, as shown in Table 6. Representations of the mechanical and optical properties of poplar and willow, according to the proportion of juvenile wood in the mixture, are shown in Figs. 1a and 1b.

Table 5. Equations Obtained for the Physical, Mechanical, and Optical Properties of Pulps Made from Mixtures of Juvenile and Mature Willow Wood

Property	Equation*	R^2
Bulk	2.32*JW + 2.48*MW	81
Air resistance	--	--
Tensile index	20.5*JW + 17.3*MW	81
Tear index	4.67*JW + 3.30*MW	88
Elongation	1.25*JW + 1.12*MW – 0.68*JW*MW	98
TEA index	0.17*JW + 0.12*MW – 0.10*JW*MW	99
Z span index	50.78*JW + 46.21*MW	80
Brightness	23.2*JW + 31.0*MW	98
Opacity	--	--
Light absorption *k*	18.25*JW + 13.61*MW + 6.63*JW*MW	98
Light scattering *s*	28.02*JW + 36.94*MW	93
* JW and MW vary between 0 and 1		

Table 6. Combination of JW:MW Ratio that Optimizes the Physical, Mechanical, and Optical Properties of Pulps from Poplar and Willow

Species		Poplar			Willow		
Property	Goal	Optimum value	JW (%)	MW (%)	Optimum value	JW (%)	MW (%)
Bulk	Maximize	2.48	32	68	2.48	0	100
Air resistance	Minimize	6.50	77	23	--	--	--
Tear index	Maximize	4.41	100	0	4.67	100	0
Tensile index	Maximize	34.8	100	0	20.0	100	0
Elongation	Maximize	1.39	100	0	1.2	100	0
TEA index	Maximize	0.31	100	0	0.16	100	0
Z span index	Maximize	64.3	100	0	50.8	100	0
Brightness	Maximize	51.2	0	100	31.0	0	100
Opacity	Maximize	94.8	51	49	--	--	--
Light absorption *k*	Minimize	3.29	0	100	13.6	0	100
Light scattering *s*	Maximize	43.5	30	70	36.9	0	100



a) b)

Fig. 1. Representation of the physical properties according to the proportion of juvenile wood in the mixture for a) poplar and b) willow. References: (1) tear index x 10, (2) tensile index, (3) Z span index, (4) brightness, (5) light absorption *k*, (6) light scattering *s*

The optimum values of tear index and tensile index were comparable to those obtained for chemithermomechanical pulps of 15-year-old poplar clones from Quebec (Law *et al.* 2000).

The desirability function is the most popular solution for multiresponse optimization problems. This approach to simultaneously optimize multiple equations, translates the functions to a common scale ([0, 1]) and combines them using the geometric mean, optimizing the overall metric. Taking into account the maximization of the mechanical properties (tensile index, elongation, tear index, Z span, and TEA index), the desirability function reached a value of 0.99 when the proportion of JW:MW was 100:0% (Table 6). However, the highest desirability function for optical properties was 0.82 (maximizing brightness, opacity, and light scattering coefficient *s*, and minimizing the light absorption coefficient *k*) and was obtained with a JW:MW ratio of 13:87%. The influence of the percentage of juvenile wood and mature wood in willow pulps was similar to poplar pulps for mechanical properties (100:0%), but different for optical properties (0:100%). Mature wood, mainly because of its higher density, required approximately twice the energy in comparison to juvenile wood to attain the same levels of refining degree. This difficulty in achieving sufficient fibrillation required to achieve good bonding implies that the mature wood should possibly be further refined to get similar strengths to those obtained with 100% of juvenile wood at 45 °SR.

In contrast to the results of this study, Myers *et al.* (1996) reported that even if aspen kraft pulps made with juvenile wood showed the best mechanical properties with respect to pulps made with mature wood, CTMP pulps showed the opposite behavior. Nevertheless, the authors did not evaluate mixtures of juvenile wood and mature wood, rather only the pure components. On the other hand, this study's results were consistent with those of Zarges *et al.* (1980) for *Populus* kraft pulps.

CONCLUSIONS

1. Short rotation (10 to 12 years) *Salix* and *Populus* hybrids were composed of 80 to 100% juvenile wood. This condition generally favored the mechanical properties of CMP pulps, in detriment to their optical properties.
2. When the mechanical properties were optimized, the highest desirability function was obtained with a JW:MW ratio of 100:0% and the optimal proportion for optical properties was achieved from the ratio of 0:100%.
3. The pattern of variation of mechanical properties can be attributed to the higher density of the MW, while that of the optical properties is due to the higher content of extractives in the JW.

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