

Article

Intra-Ring Wood Density and Dynamic Modulus of Elasticity Profiles for Black Spruce and Jack Pine from X-Ray Densitometry and Ultrasonic Wave Velocity Measurement [†]

Wassim Kharrat ¹, Ahmed Koubaa ^{2,*} , Mohamed Khlif ³ and Chedly Bradai ³

¹ Centre de Recherche sur les Matériaux Renouvelables, Université Laval, Ville de Québec, QC G1V 0A6, Canada

² Institut de Recherche sur les Forêts, Université du Québec en Abitibi-Témiscamingue, Rouyn-Noranda, QC J9X 5E4, Canada

³ Laboratoire des Systèmes électromécaniques (LASEM), École Nationale d'ingénieurs de Sfax, Sfax 3038, Tunisia

* Correspondence: ahmed.koubaa@uqat.ca; Tel.: +01-819-761-0971 (ext. 2579)

[†] This manuscript is part of an M.S. thesis by the first author, available online at depositum.uqat.ca.

Received: 30 May 2019; Accepted: 4 July 2019; Published: 9 July 2019



Abstract: Currently, ultrasonic measurement is a widely used nondestructive approach to determine wood elastic properties, including the dynamic modulus of elasticity (DMOE). DMOE is determined based on wood density and ultrasonic wave velocity measurement. The use of wood average density to estimate DMOE introduces significant imprecision: Density varies due to intra-tree and intra-ring differences and differing silvicultural treatments. To ensure accurate DMOE assessment, we developed a prototype device to measure ultrasonic wave velocity with the same resolution as that provided by the X-ray densitometer for measuring wood density. A nondestructive method based on X-ray densitometry and the developed prototype was applied to determine radial and intra-ring wood DMOE profiles. This method provides accurate information on wood mechanical properties and their sources of variation. High-order polynomials were used to model intra-ring wood density and DMOE profiles in black spruce and jack pine wood. The transition from earlywood to latewood was defined as the inflection point. High and highly significant correlations were obtained between predicted and measured wood density and DMOE. An examination of the correlations between wood radial growth, density, and DMOE revealed close correlations between density and DMOE in rings, earlywood, and latewood

Keywords: ultrasonic wave velocity measurement; nondestructive assessment; wood mechanical properties; intra-ring variation; dynamic modulus of elasticity

1. Introduction

“Wood quality is the resultant of physical and chemical characteristics possessed by a tree or a part of a tree that enable it to meet the property requirements for different end products” [1]. Wood density is considered to be the most important wood quality attribute. It is one of the most widely used parameters to predict the mechanical and other physical properties of wood, such as dimensional stability [2]. However, wood density and all its related wood quality attributes are highly variable, with multiple sources of variation, including differences within and between trees, between sites, and between genetic origins. This high variability is due to genetic, environmental, and physiological factors [3,4]. In a same species, variations in wood density also result from variations

in anatomical characteristics such as earlywood and latewood width. Wood density is generally defined as the ratio of the wood mass to volume, and is expressed in kilograms per cubic meter (kg/m^3). However, this definition does not consider variations in wood density due to biological processes such as earlywood and latewood formation, juvenile wood formation, or environmental conditions. Modern nondestructive measurement methods such as X-ray densitometry are widely used to assess wood quality variations due to biological processes (intra-ring and inter-ring variation), genetic sources, and environmental conditions (e.g., tree-to-tree, site-to-site, and silvicultural treatments).

Intra-ring wood density profiles obtained with X-ray densitometry are generally used to calculate ring density (RD), earlywood density (EWD), latewood density (LWD), ring width (RW), earlywood width (EWW), and latewood width (LWW). These parameters have been determined for many wood species, such as European oak [5], black spruce [2], and *Thuja occidentalis* [6]. Intra-ring wood density profiles are used to determine the use-specific suitability of wood, especially for high value-added applications [5,7]. Intra-ring wood density variation can also indicate wood uniformity and provide information about the wood growth process and the wood fiber yield [7,8].

Earlywood and latewood properties depend on the earlywood–latewood transition point (E/L). Several methods have been reported in the literature to determine the E/L, notably Mork's index [9]. There are at least two interpretations of Mork's index [10]. According to the first, the E/L is obtained when the double wall thickness becomes greater than or equal to the width of the cell lumen. Under the second interpretation, the E/L is obtained when the double cell wall thickness multiplied by two becomes greater than or equal to the lumen width. While this index, using either interpretation, is arbitrary and time-consuming to measure, it allows consistent determination of earlywood and latewood features.

Because Mork's index is based on the double wall thickness and the lumen diameter, these anatomical wood features must be measured in individual growth rings on microscopic slides or using indirect microscopic procedures [11]. In addition, this method is difficult to integrate into X-ray computations. Good agreement was found between earlywood and latewood features determined by three methods: Mork's index, threshold density, and the maximum derivative [12]. However, use of Mork's index and the maximum derivative produced better estimates of physiological variations compared to threshold density. Intra-ring wood density profiles are generally modelled to define the earlywood–latewood transition. Pernestål et al. [8] and Ivkovic et al. [13] used modified spline functions to model intra-ring wood density profiles. The E/L transition was defined using a numerical derivative method. Koubaa et al. [2] demonstrated that high-order polynomial functions consider both profile and intra-ring density variation for E/L estimation. These functions gave consistent estimates of the E/L transition point, with correlation coefficients between measured and predicted density well above 0.90 for the six order polynomial. These results are significant, because modelled intra-ring wood density profiles can simplify the modelling of final wood product properties [13].

While wood density is considered to be the most important wood quality attribute, elastic properties are also important, especially for engineering design purposes [14]. The wood dynamic modulus of elasticity (DMOE), being an elastic constant that describes wood mechanical behavior, is computed from the wood density and the ultrasonic wave velocity [15]. Ultrasonic wave velocity measurement is one of the most widely used nondestructive methods to assess the strength properties of living trees, logs, sawn timbers, and wood-based materials, due to its rapidity, flexibility, portability, cost-effectiveness, and ease of use [16–19]. Wood DMOE has been determined using ultrasonic wave velocity parallel to the grain direction and wood density based on the mass-to-volume ratio of specimens [15,18–21]. However, no study to date has investigated intra-ring wood DMOE profiles to determine variations between earlywood and latewood DMOE. A nondestructive method based on X-ray densitometry and ultrasonic wave velocity measurement was proposed to determine intra-ring wood DMOE profiles.

The objectives of this study were therefore to (1) develop a nondestructive method to determine intra-ring wood DMOE profiles; (2) model intra-ring wood density and DMOE profiles in black spruce and jack pine wood using high-order polynomial functions [2]; (3) determine radial variations in ring

wood density and ring DMOE; and (4) analyze correlations between wood radial growth, density, and DMOE.

2. Materials and Methods

X-ray densitometry provides intra-ring wood density profiles and determines both annual ring width and wood density components. Intra-ring wood ultrasonic velocity profiles were determined using the developed prototype and a Sonatest Masterscan ultrasonic flaw detector. The superposition of these two profiles is a nondestructive method to obtain the intra-ring wood DMOE profile.

2.1. Study Materials

The experimental material used in this study consisted of subsamples from previous studies on the wood quality of jack pine [22] and black spruce [23] sampled from even-aged stands in the Abitibi region of Québec, Canada. Eight black spruce and eight jack pine trees were used. Discs taken at breast height were used in this study. Bark-to-bark samples passing through the pith were extracted from each disc. Thin strips (15 to 20 mm wide and 1.57 to 1.9 mm thick) were sawn from each sample. The sawn strips were extracted with cyclohexane/ethanol (2:1) solution for 24 h and then with distilled water for another 24 h to remove extraneous compounds [24]. After extraction, the strips were air-dried under restraint to prevent warping. Samples were then conditioned to 8% equilibrium moisture content before measurement. The same samples were used to determine wood density and ultrasonic wave propagation time. In this study, a nondestructive method based on X-ray densitometry and ultrasonic wave velocity measurement was used to determine intra-ring wood density and DMOE variation.

2.2. Wood Ring Density and Width Measurement

Ring density (RD) and ring width (RW) were measured for each ring using a QTRS-01X Tree-Ring Scanner (Quintek Measurement Systems, Knoxville, TN, USA). The QTRS passes thin strips from increment cores through an accurately collimated soft X-ray beam using a precisely controlled stepping system and linear bearing carriage. Video images of both the wood sample surface and the X-ray density graph are displayed at the same scale on the screen. A linear resolution step size of 40 μm was used for the X-ray densitometry. Rings from pith to bark were scanned in air-dry condition to estimate the basic wood density (ovendry weight/green volume) for each ring. Ring density (RD) and ring width (RW) for each ring were determined based on intra-ring microdensitometer profiles. Incomplete or false rings and rings with compression wood or branch tracers were eliminated from the analysis. Matlab software (R2016a, the MathWorks, Inc., Natick, MA, USA) was used to determine the intra-ring wood density profiles at 40 μm resolution.

2.3. Wood Ultrasonic Wave Velocity Measurement

An in-house prototype device was developed for measuring the ultrasonic wave propagation time with the same resolution as that used for the wood density measurement by X-ray densitometry (40 μm). The prototype (Figure 1) consists of a motorized linear translation stage that holds the sample and is controlled by a microcontroller. The ultrasonic wave propagation time in the wood sample is measured between the ultrasonic transmitter (Spot Weld Transducer) and the receiver transducers (Fingertip Contact Transducer CF) at 40 μm resolution. The ultrasonic transducers are mounted in parallel to two mini motorized actuators to ensure constant pressure during measurement.

Following the X-ray densitometry density measurement, nondestructive ultrasonic wave propagation measurement was applied to the same samples using a Masterscan 380 (Sonatest Inc., San Antonio, TX, USA) equipped with 10 MHz frequency transducers. Ultrasonic waves were applied to the samples through two transducers (transmitting and receiving). A coupling agent (Vaseline original petroleum jelly) was used to aid the transmission of the transducer pulses into the test specimens.

A correction factor (C_f ; s) was applied to calculate the ultrasonic wave velocity in the wood samples to take into consideration the transport time of the electric waves within the measuring circuit.

A Plexiglas sample having the same thickness as the wood samples (2 mm) was used as a reference to determine the correction factor (Equation (1)) [20,25]. The ultrasonic velocity (V ; m/s) was then calculated using Equation (2):

$$C_f = t_r - (d_r/v_r) \quad (1)$$

$$V = d/(T - C_f) \quad (2)$$

where d is the thickness of the wood sample (m), T is the ultrasonic wave propagation time (s), t_r is the wave propagation time through the reference Plexiglas core (s), d_r is the thickness of the reference Plexiglas core (m), and v_r is the wave velocity in the reference Plexiglas core (2670 m/s).

The dynamic modulus of elasticity (DMOE; MPa) based on the ultrasonic method was determined using the following one-dimensional wave equation:

$$\text{DMOE} = \rho \times V^2 \times 10^{-6}. \quad (3)$$

where ρ is the wood density measured by X-ray densitometry (kg/m^3) and V is the ultrasonic wave velocity calculated using Equation (1).

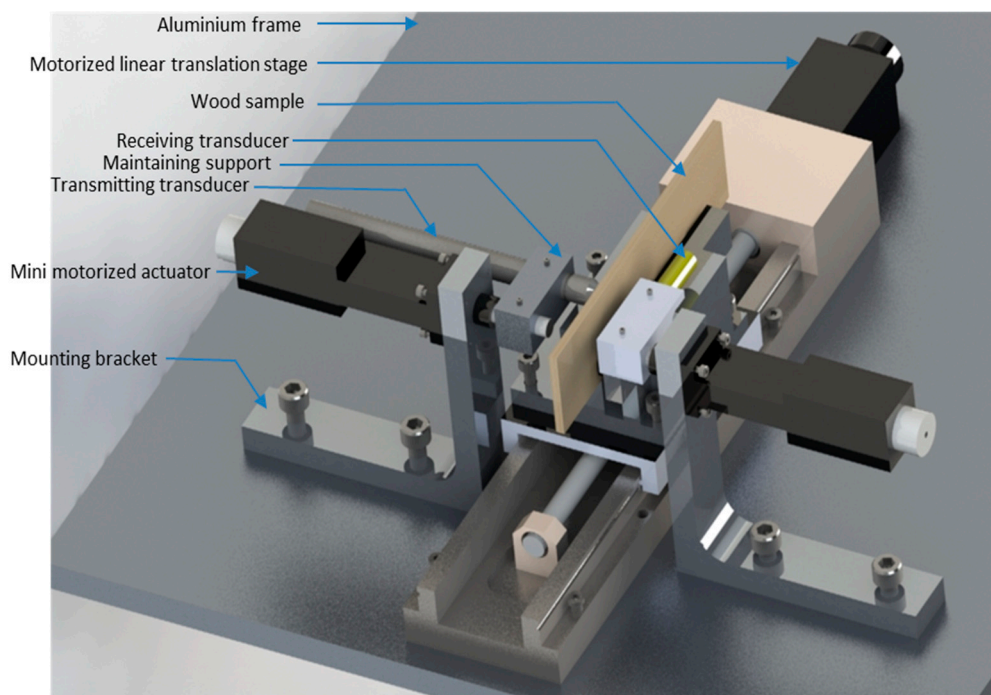


Figure 1. Prototype device for measuring the ultrasonic wave propagation time.

2.4. Modelling Intra-Ring Wood Density and Dynamic Modulus of Elasticity Profiles

In this study, we used 6th order polynomial functions to model intra-ring wood density and DMOE profiles for black spruce and jack pine wood (Equation (4)).

$$R = a_0 + a_1RW + a_2RW^2 + a_3RW^3 + a_4RW^4 + a_5RW^5 + a_6RW^6 \quad (4)$$

where R is the ring density or ring DMOE, RW is the ring width in proportion, and a_i are the parameters to be estimated.

The E/L transition was defined as the inflection point obtained from the within-ring density and DMOE profiles. The E/L transition is obtained by equalling the second derivative of the polynomial function to zero (Equation (5)). For a 6th order polynomial function, the second derivative gives 4 solutions, but only one solution is of interest. Certain restrictions were specified in the Matlab

program to obtain this unique solution. These restrictions specify that the solution should be included in a positive slope and in the range of 40 to 90% of the ring width proportion. If more than one solution is obtained, the highest value among the solutions is chosen.

$$d^2R/dRW^2 = 2a_2 + 6a_3RW + 12a_4RW^2 + 20a_5RW^3 + 30a_6RW^4 \quad (5)$$

2.5. Statistical Analysis

For both softwood species (black spruce and jack pine), the correlations between the wood radial growth, density, and DMOE components were determined using R software (Version 2.15.0 R, R Development Core Team, 2012, Vienna, Austria).

3. Results and Discussion

Typical X-ray density and DMOE profiles for jack pine wood are shown in Figure 2. Both the within-ring and radial pattern variation in these properties are shown.

3.1. Intra-Ring Wood Density and Dynamic Modulus of Elasticity Profiles

Figure 3 shows the within-ring variation in wood density and DMOE for black spruce and jack pine, revealing similar within-ring density patterns between the two species. Wood density and DMOE increase slowly in earlywood to reach a maximum in latewood. Both properties decrease thereafter at about mid-latewood width to reach a minimum at the boundary between two growth rings. The similarity between intra-ring density and DMOE profiles confirms the close relationship between wood density and wood stiffness, even at the intra-ring level. Some slight differences between the intra-ring density and DMOE profiles appear in earlywood. Thus, the intra-ring wood density profiles increase more slowly in earlywood compared to the DMOE profiles, which show a relatively sharp increase. Similar patterns of within-ring density variation were obtained by Koubaa et al. [2] for black spruce and by Ivkovic et al. [13] for Norway spruce and Douglas fir.

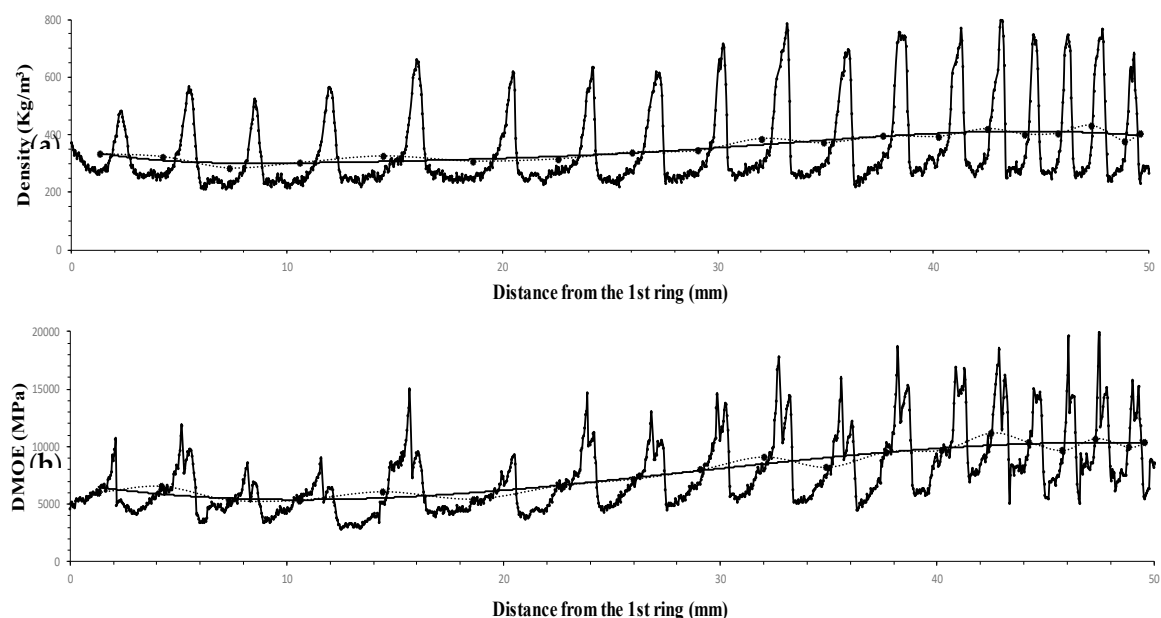


Figure 2. Examples of jack pine profiles showing radial variation in: (a) Wood density and (b) DMOE in (from ring 2 to 19).

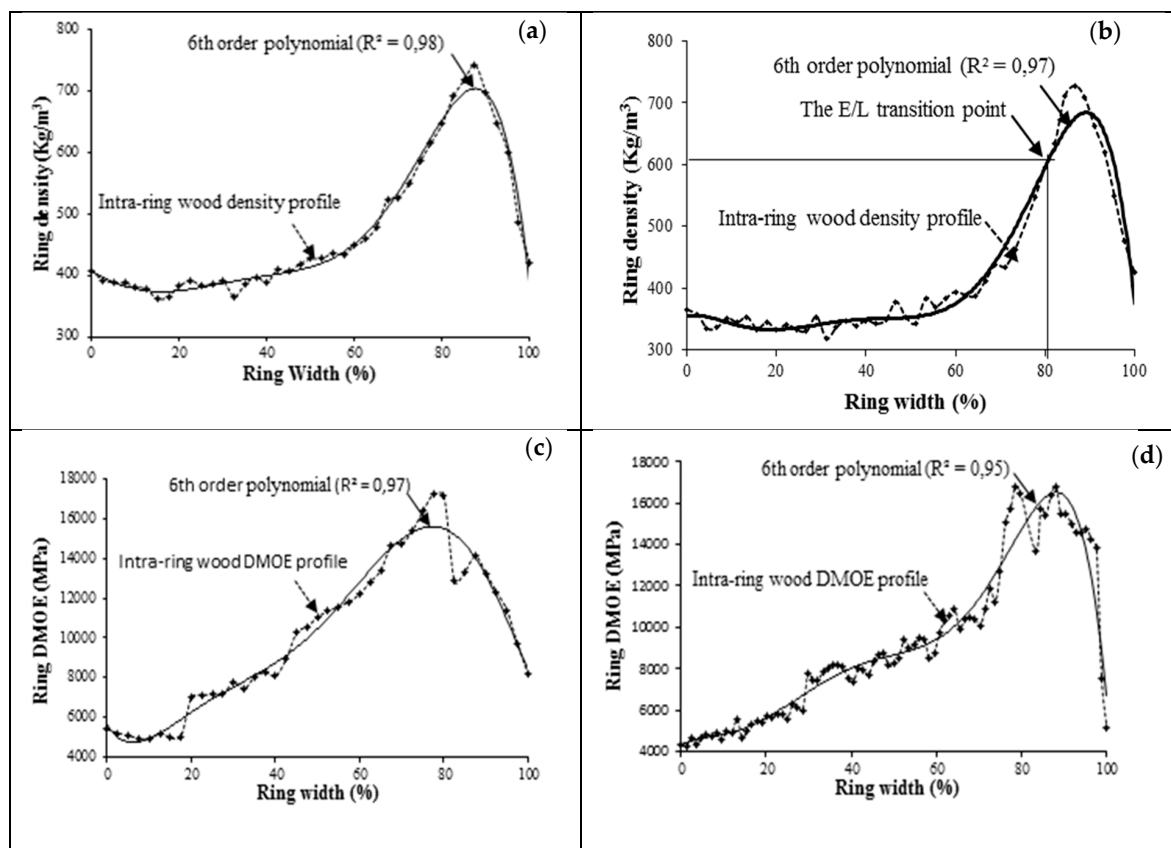


Figure 3. Examples of within-ring profiles and the fits obtained with the 6th order polynomials for: (a) Ring density in black spruce; (b) ring density in jack pine; (c) DMOE in Black spruce; and (d) DMOE in Jack pine.

The same 6th polynomial function modeling approach suggested by Koubaa et al. [2] was used to model within-ring density and DMOE profiles for the black spruce and jack pine samples in this study. Figure 3 illustrates the fitness of the 6th order polynomials for the intra-ring density and DMOE profiles for both softwoods species: (a) Black spruce and (b) jack pine. Table 1 confirms the fitness. The correlation coefficients obtained between the measured and predicted ring density data range from 0.88 to 1.00, with an average well above 0.95. These results indicate that these models can well describe intra-ring wood density profiles obtained from black spruce and jack pine, and probably other softwood species, as the coefficients are in good agreement with those obtained by Koubaa et al. [2] for black spruce.

Table 1 also indicates that high-order polynomials fit well the intra-ring DMOE profiles for black spruce and jack pine. The correlation coefficients obtained between measured and predicted DMOE data using the 6th order polynomial models range from 0.80 to 0.99, with an average well above 0.90 (Table 1). These results indicate that high-order polynomials can describe DMOE profiles well for these two softwoods.

The measured elastic properties of wood material yield essential information for the understanding of bonding at a very fine structural level [14]. As the elastic properties describe the mechanical behavior of wood, it is mandatory to determine intra-ring wood DMOE profiles. Moreover, modelled intra-ring wood DMOE profiles can serve as effective prediction tools for wood mechanical behavior.

3.2. The Earlywood–Latewood Transition

Table 2 shows that wood density at the E/L transition point (Figure 3b) (E/L transition density) as defined by the inflexion point method presents large variation for black spruce (Figure 4a) and jack

pine (Figure 4b). The radial variation pattern for the E/L transition density in black spruce (Figure 4a) is similar to that reported by Koubaa et al. [2], and is characterized by large variation with no specific trend. In contrast, the radial variation for the E/L transition in jack pine (Figure 4b) is characterized by a steady increase in juvenile wood and a tendency to level off in mature wood. Similar radial variation patterns for the E/L wood transition were observed by Park et al. [22].

Table 1. Average, standard variation (between parenthesis) and range of Pearson’s coefficient of determination between measured and predicted within-ring density and DMOE values from the 6th order polynomial models for different rings for black spruce and jack pine.

	Ring from Pith			
	5	10	15	20
Wood density profiles				
Black spruce				
Average profiles	0.96 (0.02)	0.97 (0.02)	0.96 (0.02)	0.97(0.02)
Range	0.88–0.99	0.91–0.99	0.91–0.99	0.92–1.00
Jack pine				
Average profiles	0.96 (0.02)	0.95 (0.02)	0.97 (0.02)	0.98 (0.01)
Range	0.92–0.98	0.90–1.00	0.89–0.99	0.96–0.99
Dynamic modulus of elasticity profiles				
Black spruce				
Average profiles	0.92 (0.03)	0.94 (0.04)	0.95 (0.03)	0.95 (0.02)
Range	0.82–0.99	0.88–0.99	0.86–0.99	0.91–0.99
Jack pine				
Average profiles	0.89 (0.04)	0.93 (0.02)	0.93 (0.03)	0.94 (0.02)
Range	0.80–0.97	0.88–0.98	0.82–0.99	0.91–0.99

Within a same ring, the E/L transition also shows substantial variation in the true measures, as indicated by the relatively large standard errors (Figure 4a,b, Table 2). For example, the E/L transition density for the 10th annual ring from the pith varies from 541 to 655 kg/m³ and from 548 to 672 kg/m³ in black spruce and jack pine, respectively (Table 2). These results concur with the findings by Koubaa et al. [26] for black spruce and by Park et al. [22] for jack pine. Earlywood and latewood density defined by this method also show large variation. Thus, for a same annual ring, earlywood density ranges from 383 to 432 kg/m³ for black spruce and from 318 to 367 kg/m³ for jack pine.

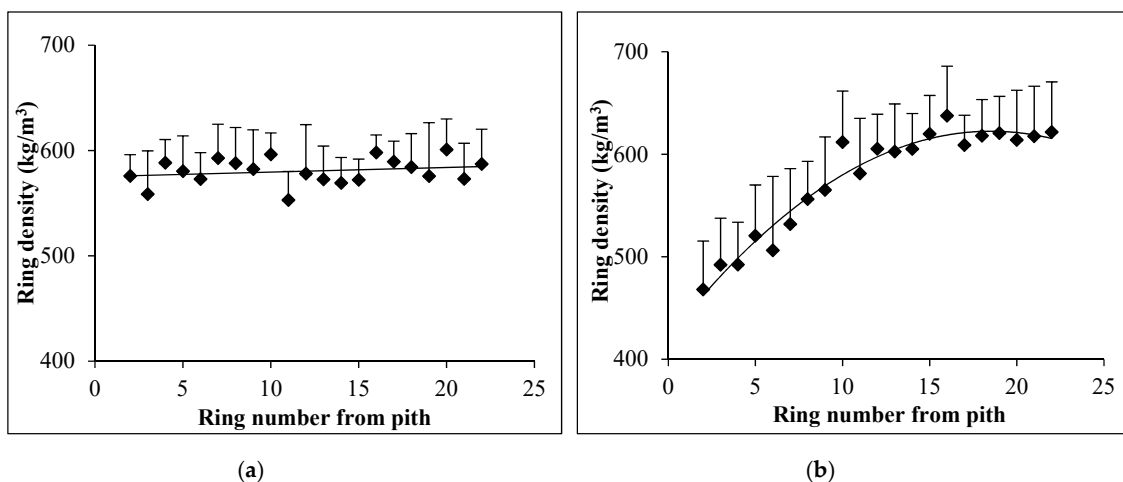


Figure 4. Radial variation in E/L transition density (bars indicate the standard error) for (a) black spruce and (b) jack pine.

As shown in Table 2, wood density is variable at the E/L transition point for black spruce and jack pine. For black spruce, the average wood density at the E/L transition point is variable and higher

than the 590 kg/m³ reported by Koubaa et al. [2], as well as the threshold wood density of 540 kg/m³ used for black spruce in X-ray densitometry programs. The results for jack pine are similar: The E/L transition density is variable and higher than the threshold density typically used for jack pine in X-ray densitometry programs. As the wood density at the E/L transition point, as defined by the inflexion point method, is variable and higher than the threshold wood density, the average earlywood and latewood width and density as defined by the inflexion point method will differ from those defined by the threshold method, for both black spruce and jack pine. Earlywood width defined by the inflexion point method will be greater, whereas latewood width will be smaller. Consequently, the latewood proportion defined by the inflexion point method will be lower. These results confirm the findings by Koubaa et al. [2] that the E/L transition point varied greatly among individual growth rings and that the use of a predetermined fixed threshold wood density does not reflect the variation in intra-ring wood density profiles across growth rings in a species.

Table 2. Average (Av), range (Ra), and standard variation for ring width, wood density, and wood DMOE at the earlywood–latewood transition and in earlywood and latewood, as defined by the inflexion method for different rings.

	Black Spruce			Jack Pine		
	Ring Number from the Pith					
	5	10	20	5	10	20
Earlywood width						
Av (mm)	1.32 (0.37)	1.26 (0.35)	1.02 (0.20)	2.65 (0.27)	2.05 (0.31)	1.23 (0.32)
Ra (mm)	0.69–1.95	0.81–1.88	0.72–1.37	2.05–3.17	1.40–2.74	0.87–2.09
Latewood width						
Av (mm)	0.39 (0.12)	0.30 (0.08)	0.23 (0.06)	0.58 (0.06)	0.50 (0.06)	0.38 (0.10)
Ra (mm)	0.22–0.61	0.15–0.48	0.16–0.31	0.47–0.67	0.44–0.58	0.25–0.63
Earlywood density						
Av (kg/m ³)	415 (27)	403 (14)	385 (17)	316 (16)	340 (13)	331 (22)
Ra (kg/m ³)	376–491	383–432	344–418	296–348	318–367	295–359
Latewood density						
Av (kg/m ³)	637 (47)	673 (20)	692 (36)	612 (58)	734 (89)	726 (62)
Ra (kg/m ³)	578–729	568–796	591–746	502–769	605–856	581–798
Density at the earlywood latewood transition						
Av (kg/m ³)	580 (33)	596 (20)	600 (29)	520 (49)	612 (49)	614 (48)
Ra (kg/m ³)	536–653	541–655	547–649	433–623	548–672	482–674
Earlywood dynamic modulus of elasticity						
Av (GPa)	11.2 (2.5)	11.1 (1.6)	11.8 (2.6)	6.4 (0.8)	9.9 (1.2)	10.7 (1.9)
Ra (GPa)	8.3–17.6	6.5–13.7	8.2–15.3	5.2–8.1	7.9–1.2	8.0–1.4
Latewood dynamic modulus of elasticity						
Av (GPa)	14.8 (2.4)	15.2 (2.5)	17.9 (2.4)	10.6 (1.6)	14.6 (1.0)	16.3 (3.6)
Ra (GPa)	11.9–21.7	10.3–18.8	14.0–22.1	8.1–12.1	12.9–16.9	11.9–21.0
Dynamic modulus of elasticity at the earlywood latewood transition						
Av (GPa)	13.4 (2.1)	13.6 (1.9)	15.4 (1.9)	9.8 (1.4)	12.9 (1.2)	14.9 (1.9)
Ra (GPa)	10.4–17.7	9.3–15.8	13.1–18.4	6.4–12.2	11.2–15.9	11.2–17.1

The same method was used to determine earlywood DMOE (EWDMOE), latewood DMOE (LWDMOE), and DMOE at the earlywood–latewood transition (Figure 5a,b). Thus, the wood DMOE at the E/L transition for the 10th annual ring varied from 9261 to 15,798 MPa for black spruce and from 11,162 to 15,950 MPa for jack pine. For the same annual ring, EWDMOE ranged from 6500 to 13,652 MPa for black spruce and from 7946 to 11,613 MPa for jack pine. LWDMOE also showed large variation: From 10,327 to 18,792 MPa for black spruce and from 12,873 to 16,916 MPa for jack pine (Table 2). The radial DMOE patterns at the E/L transition for black spruce (Figure 5a) and jack pine (Figure 5b) are shown. As shown in Figure 4a, the radial variation pattern for the E/L transition density in black spruce is characterized by large variation with no specific trend. Similar radial variation patterns for the E/L transition density were observed by Koubaa et al. [2]. In contrast, for jack pine,

the radial variation pattern for the E/L transition density is characterized by a steady increase in juvenile wood and a tendency to level off in mature wood (Figure 4b). The radial variation pattern for the E/L transition DMOE in black spruce (Figure 5a) is similar to jack pine (Figure 5b) characterized by a linear increase. These results confirm the importance of measuring ring density and RDMOE separately in order to obtain a more detailed characterization of wood mechanical behavior.

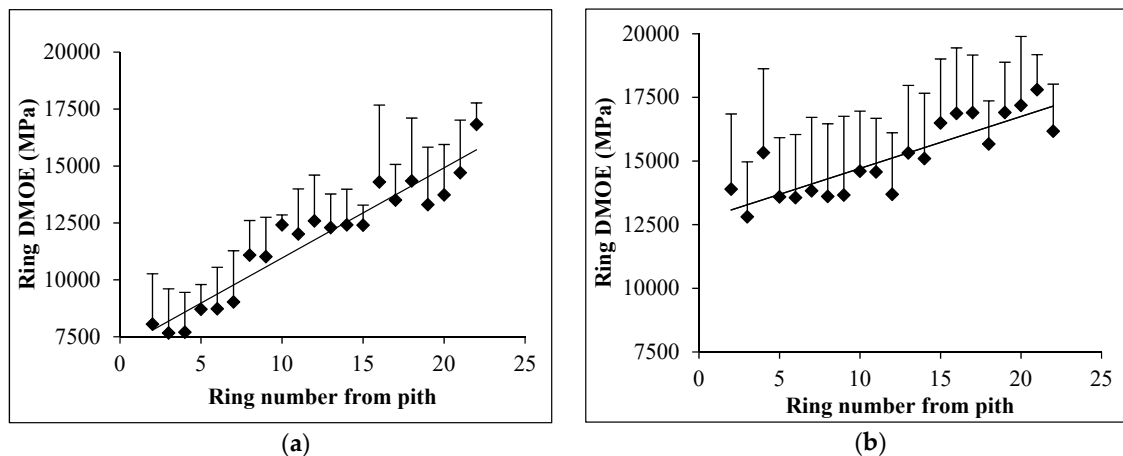


Figure 5. Radial variation in E/L transition DMOE (bars indicate the standard error) for (a) black spruce and (b) jack pine.

Figure 6 illustrates the close relationship between the transition DMOE measured for the earlywood–latewood transition density and the transition DMOE, as defined by the inflexion point method for all tested samples (black spruce and jack pine). A linear regression curve ($y = 1.06x$) was obtained. Student's t test was applied and results indicated no significant differences between the transition DMOE calculated for the earlywood–latewood transition density and the transition DMOE as defined by the inflexion point method calculated from DMOE data. These results reaffirm the close relationship between wood density and wood mechanical properties, and particularly wood DMOE.

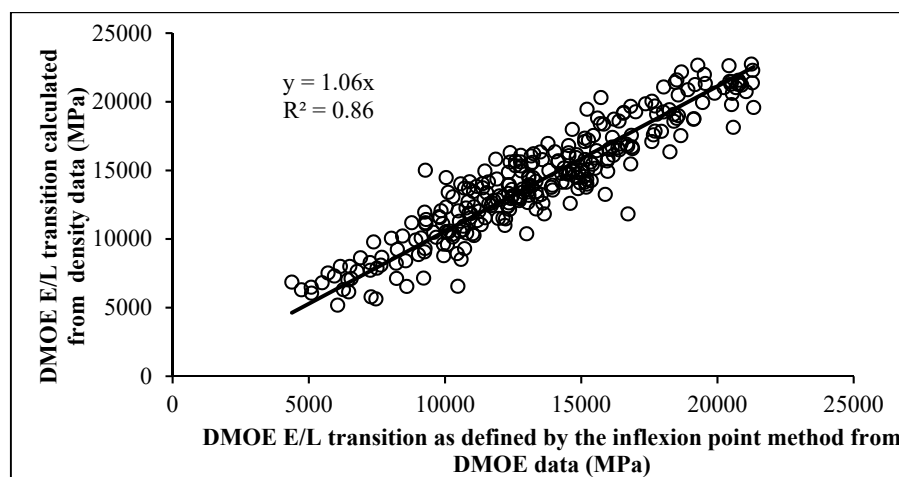


Figure 6. Relationship between transition DMOE calculated at the earlywood–latewood (E/L) transition density and transition DMOE, as defined by the inflexion point method for black spruce and jack pine.

3.3. Radial Variation in Ring Wood Density and Ring Dynamic Modulus of Elasticity

The mean values for intra-ring density over all samples for both black spruce (a) and jack pine (b) are shown in Figure 7a,b, respectively. The radial variation pattern for wood density is similar to that reported by Park et al. [22] for jack pine, Koubaa et al. [26] for black spruce, and Grabner et al. [24]

for European larch. The ring density is relatively high near the pith and decreases thereafter to reach a minimum in the transition zone leading into the mature wood, where a slow and steady increase is observed. Earlywood density (Figure 7a) decreases rapidly from a maximum near the pith to a low value in the transition zone. The density decreases slowly thereafter with age [26]. Latewood density (Figure 7a) increases almost linearly to a maximum at about ring 13, then levels off in the transition zone and the mature wood [22]. Similar typical variation patterns are seen for DMOE (Figure 8a,b). Ring DMOE increases with tree age, then levels off beyond the 13th ring. A similar radial variation pattern for DMOE was previously reported for hybrid poplar [25]. However, no study to date has investigated radial variation in earlywood and latewood DMOE (EWDMOE and LWDMOE). RDMOE and LWDMOE increase almost linearly in juvenile wood to a maximum at about ring 15, then decrease slowly thereafter through the outer rings in mature wood (Figure 8a,b).

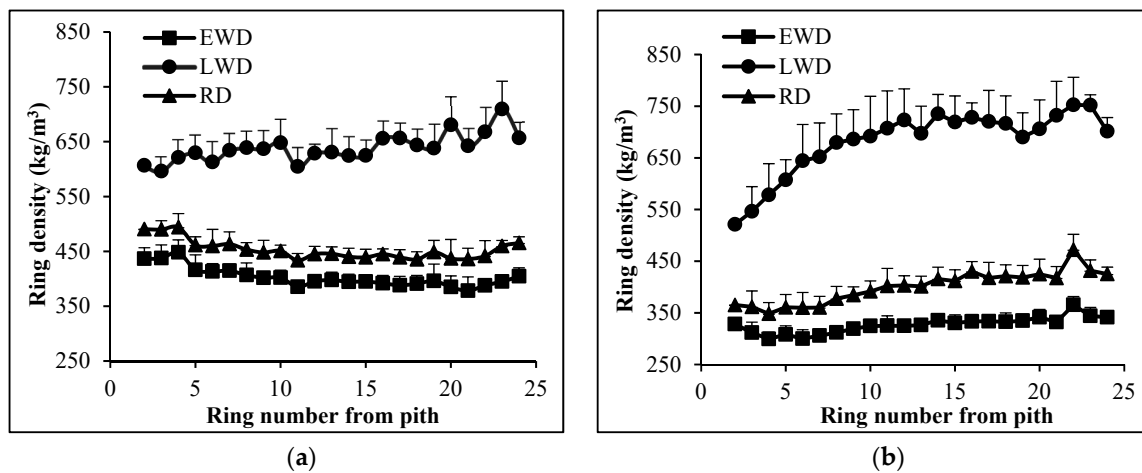


Figure 7. Radial variation in ring density (RD), earlywood density (EWD), and latewood density (LWD) for (a) black spruce and (b) jack pine.

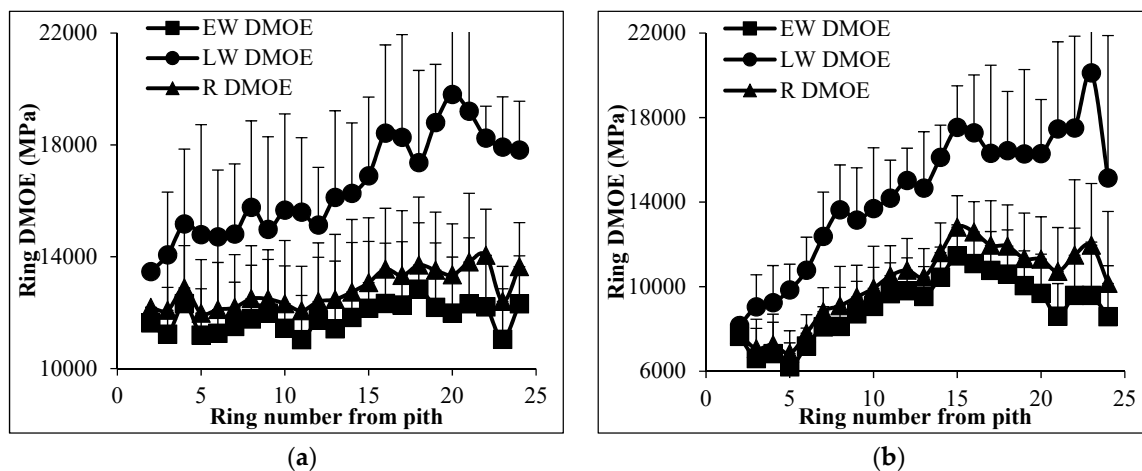


Figure 8. Radial variation in ring dynamic modulus of elasticity (RDMOE), earlywood dynamic modulus of elasticity (EWDMOE), and latewood dynamic modulus of elasticity (LWDMOE) for (a) black spruce and (b) jack pine.

3.4. Relationships between Growth, Density, and Elastic Properties

The developed prototype enabled determining relationships between ultrasonic velocity and wood density in rings, earlywood, and latewood (Figures 9 and 10). The coefficient of determination for the linear correlation between RD measured with X-ray densitometry and ring ultrasonic velocity obtained from the developed prototype was $R^2 = 0.66$ using black spruce and jack pine data (Figure 9).

Moderate linear correlations were also obtained between ring, earlywood, and latewood density and ultrasound speed of propagation for Jack pine (Figure 10) and black spruce (not shown).

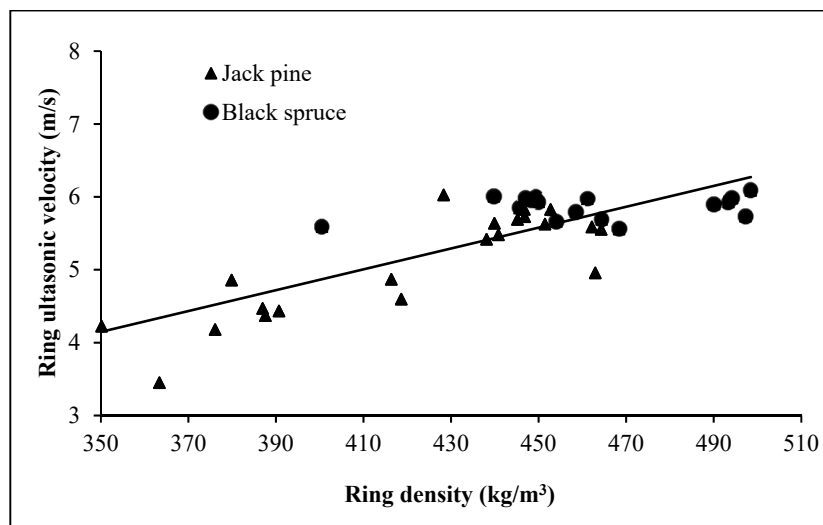


Figure 9. Relationship between ring density and ring ultrasonic velocity for black spruce and jack pine.

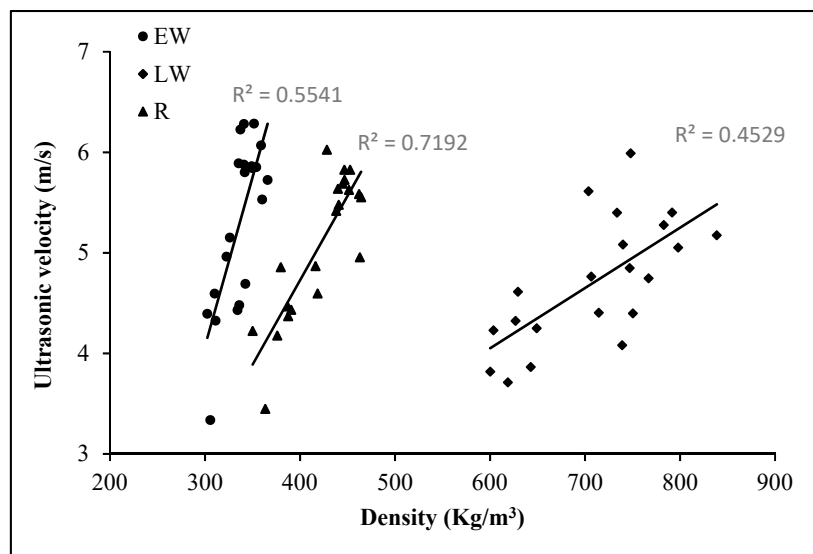


Figure 10. Relationships between wood density and ultrasonic velocity in rings (R), earlywood (EW), and latewood (LW) for jack pine.

Table 3 indicates that ring density is positively correlated with earlywood and latewood density. However, for both softwood species, the correlations between ring density and earlywood density are higher than between ring density and latewood density. These results concur with previous studies of black spruce [27]. Ring DMOE shows similar results. Thus, for both black spruce and jack pine, the correlations between ring DMOE and earlywood DMOE are higher than those between ring DMOE and latewood DMOE. The close correlations between DMOE and density shown in Table 3 are due to the fact that the DMOE is obtained from density (Equation (3)). Table 3 also shows a negative and statistically significant correlation between RDMOE and RW. Similar results were found for both earlywood and latewood. In contrast, high positive relationships were found between RDMOE and both earlywood and latewood DMOE, for both softwood species (Table 3). These results have practical implications for a considerably accurate, nondestructive determination of wood density, growth, and stiffness from small samples.

Table 3. Pearson's coefficient of correlations between the different traits for black spruce (upper row) and jack pine (lower row).

	RW	EWW	LWW	RD	EWD	LWD	RDMOE	EWDMOE	LWDMOE
RW		0.98 ***	0.81 ***	−0.21 *	0.00 ns	−0.36 *	−0.37 *	−0.29 *	−0.49 **
EWW	0.99 ***		0.73 ***	−0.25 *	−0.03 ns	−0.32 *	−0.38 *	−0.30 *	−0.49 **
LWW	0.82 ***	0.77 ***		−0.02 ns	0.08 ns	−0.49 **	−0.31 *	−0.25 *	−0.50 **
RD	−0.69 **	−0.72 ***	−0.42 **		0.91 ***	0.37 *	0.45 **	0.44 **	0.33 *
EWD	−0.58 **	−0.58 **	−0.39 *	0.85 ***		0.11 ns	0.40 **	0.46 **	0.18 ns
LWD	−0.50 **	−0.49 **	−0.52 **	0.75 ***	0.48 **		0.18 ns	0.09 ns	0.49 **
RDMOE	−0.65 **	−0.66 ***	−0.48 **	0.79 ***	0.65 **	0.66 ***		0.98 ***	0.85 ***
EWDMOE	−0.60 **	−0.61 **	−0.44 **	0.73 ***	0.65 **	0.57 **	0.98 ***		0.75 ***
LWDMOE	−0.61 **	−0.60 **	−0.55 **	0.74 **	0.54 **	0.80 ***	0.89 ***	0.78 ***	

* Significant at $\alpha = 0.05$; ** Significant at $\alpha = 0.01$; *** Significant at $\alpha = 0.001$; ns not significant. RW: ring width, EWW: earlywood width, LWW: latewood width, RD: ring density, EWD: earlywood density, LWD: latewood density, RDMOE: ring dynamic modulus of elasticity, EWDMOE: earlywood dynamic modulus of elasticity, LWDMOE: latewood dynamic modulus of elasticity.

4. Practical Implications

For this study, we developed a rapid nondestructive method to determine wood density and the dynamic modulus of elasticity (DMOE) based on X-ray densitometry and ultrasonic wave velocity measurement. This method was used to determine earlywood and latewood properties in order to obtain a more detailed characterization of wood mechanical behavior. Only a few studies have investigated earlywood and latewood elastic properties [28–32]. Roszyk et al. [29] reported that latewood modulus of elasticity (MOE) is higher than earlywood MOE for scots pine at low moisture content (8%). Similar results were obtained for Spruce wood [*Picea abies* (L.) Karst] [31] and loblolly pine [28–32] with an important increase in MOE values with the growth of annual rings. However, the preparation of initial and final wood samples was quite complicated and required perfectly parallel annual rings. Different methods have been used to retrieve two adjacent earlywood and latewood bands of 1 mm thick for loblolly pine [28–32]. Moliński et al. [31] reported that two adjacent wood samples were cut out from the region in which the borders of annual rings were straight lines parallel to the longer axis of the plank to obtain two earlywood and latewood samples of 200 μm in thickness for Spruce wood. Thus, it is important to use a rapid nondestructive method with easily prepared samples to determine wood intra-ring mechanical properties, which have direct impacts on wood processing performance. In fact, understanding mechanical properties variations at the earlywood-latewood scale will eventually allow a better knowledge of wood's areas of weakness in order to optimize the performance of wood products. At the wood processing industry scale, this information would be important for mechanical pulping processes where the pulping and refining energies and wood fractioning are closely related to the fiber characteristics including earlywood and latewood mechanical properties [33]. Similarly, oriented strand board (OSB) manufacturing and properties are directly related to earlywood and latewood mechanical properties [28,34]. The results of this study further confirm that ultrasonic measurement can be used to determine the elastic constants of wood with considerable accuracy (0.04 mm). Indeed, the relationships found between ultrasonic wave velocity, density, and wood stiffness demonstrate the experimental efficiency of ultrasonic measurement [35]. Whereas several studies have investigated relationships between static and dynamic MOE in wood [15,19,20,36] and have found significant linear correlations between them, only a few studies have investigated relationships between wood DMOE and wood density [37,38]. For example, linear correlations ($r = 0.70$) were found between DMOE and wood density for *Eucalyptus delegatensis* [39].

The relationships between tree growth and wood properties, especially in terms of mechanical properties, are critical for effective forest management strategies [19,39]. Russo et al. [39] reported that the effects of silvicultural practices (different intensities of thinning) on wood quality can be identified using acoustic measurement to assess the MOED of standing trees with non-destructive method in Calabrian pine. The authors demonstrated that using a low intensity of thinning induced better tree wood quality. In boreal species, enhanced growth through tree improvement programs or

intensive forest management strategies can significantly diminish the wood mechanical properties due to several biological factors, including increased earlywood proportion and the production of larger cells with thinner walls. Several anatomical and physical characterization studies have clearly demonstrated the impact of intensive forest management strategies on earlywood, latewood, and overall wood properties [40]. Nevertheless, the impact of intensive forest management on wood mechanical properties has received relatively little attention due to sample size constraints, the destructive nature of characterization tests, and the lack of effective tools for rapid, nondestructive characterization of these properties at the ring level. Nondestructive assessment is essential for understanding the impact of intensive forest management practices on wood mechanical properties as well as the physiological and biological processes involved in wood strength development [39]. The method developed here allows nondestructive measurement of intra-ring wood DMOE and provides deeper insights into wood strength development and its relationships to growth and wood density. As shown in Table 3, radial profiles enable investigating relationships between wood radial growth, density, and elastic properties in rings, earlywood, and latewood.

5. Conclusions

Based on the results of this study, the following conclusions can be drawn:

- (1) Intra-ring wood dynamic modulus of elasticity (DMOE) profiles can be determined using a nondestructive method based on X ray densitometry and ultrasonic wave velocity measurement.
- (2) Sixth order polynomials can well describe intra-ring wood density and dynamic modulus of elasticity profiles in black spruce and jack pine.
- (3) The inflexion point method can be used to determine with considerable accuracy the earlywood–latewood transition density and DMOE in black spruce and jack pine.
- (4) For black spruce and jack pine, the correlation coefficients between wood density and wood DMOE were positive and statistically significant in rings, earlywood, and latewood. Furthermore, high positive correlations were obtained between ring DMOE and both earlywood and latewood DMOE.

Author Contributions: Conceptualization, A.K., W.K., C.B. and M.K.; methodology, W.K. and A.K.; formal analysis, W.K. and A.K.; investigation, W.K. and A.K.; resources, A.K.; data curation, W.K. and A.K.; writing—original draft preparation, W.K.; writing—review and editing, A.K., M.K., C.B.; supervision, A.K., C.B. and M.K.; project administration, A.K.; funding acquisition, A.K.

Funding: This research was funded by the Canada Research Chair Program, Grant number: 557752; MITACS Grant number: IT04397 and Natural Resources Canada, CWFC 2017-2018.

Acknowledgments: The authors are grateful to Gilles Villeneuve and Williams Belhadeff for their invaluable technical assistance. The authors also thank Besma Bouslimi and Zahia Ait-Si-Said for assistance with X-ray density measurements. Thanks also to Margaret McKyes for linguistic editing.

Conflicts of Interest: The authors declare no conflict of interest. The funders had no role in the design of the study; analyses, interpretation of data; in the writing of the manuscript, or in the decision to publish the results.

References

1. Mitchell, H. *A Concept of Intrinsic Wood Quality and Nondestructive Methods for Determining Quality in Standing Timber*; Forest Service, U.S. Report No 2233; Forest Products Laboratory: Madison, WI, USA, 1961.
2. Koubaa, A.; Tony Zhang, S.Y.; Makni, S. Defining the transition from earlywood to latewood in black spruce based on intra-ring wood density profiles from X-ray densitometry. *Ann. For. Sci.* **2002**, *59*, 511–518. [[CrossRef](#)]
3. Panshin, A.J.; De Zeeuw, C. *Textbook of Wood Technology*; McGraw-Hill Book Co: New York, NY, USA, 1980; p. 772.
4. Rozenberg, P.; Franc, A.; Bastien, C.; Cahalan, C. Improving models of wood density by including genetic effects: A case study in Douglas-Fir. *Ann. For. Sci.* **2001**, *58*, 385–394. [[CrossRef](#)]
5. Zhang, S.Y.; Nepveu, G.; Owoundi, R.E. Intra-tree and inter-tree variation in selected wood quality characteristics of European oak (*Quercus petraea* and *Quercus robur*). *Can. J. For. Res.* **1994**, *24*, 1818–1823. [[CrossRef](#)]

6. Bouslimi, B.; Koubaa, A.; Bergeron, Y. Anatomical properties in *Thuja occidentalis*: Variation and relationship to biological processes. *IAWA J.* **2014**, *35*, 363–384. [[CrossRef](#)]
7. Zobel, B.J.; Van Buijtenen, J.P. *Wood Variation: Its Causes and Control*; Springer: Berlin, Germany, 1989; p. 363.
8. Pernal, K.; Jonsson, B.; Larsson, B. A simple model for density of annual rings. *Wood Sci. Technol.* **1995**, *29*, 441–449. [[CrossRef](#)]
9. Mork, E. Die qualität des fichtenholzes unterbesonderer rücksichtnahme auf schleif-und papierholz. *Papier-Fabrikant* **1928**, *26*, 741–747.
10. Denne, M.P. Definition of latewood according to Mork (1928). *IAWA Bull.* **1988**, *10*, 59–62. [[CrossRef](#)]
11. Evans, R.; Gartside, G.; Downes, G. Present and prospective use of Silviscan for wood microstructure analysis. In Proceedings of the 49th Appita Annual General Conference proceedings 1995, Hobart, Australia, 2–7 April 1995; pp. 91–96.
12. Barbour, R.J.; Bergqvist, G.; Amundson, C.; Larsson, B.; Johnson, J.A. New methods for evaluating intra-ring X-ray densitometry data: Maximum derivative methods as compared to Mork's index. In Proceedings of the CTIA/IUFRO International Wood Quality Workshop, Quebec, QC, Canada, 18–22 August 1997; p. 61.
13. Ivkovic, M.; Rosenberg, P. A method for describing and modelling of within-ring wood density distribution in clones of three coniferous species. *Ann. For. Sci.* **2004**, *61*, 759–769. [[CrossRef](#)]
14. Bucur, V.; Archer, R.R. Elastic constants for wood by an ultrasonic method. *Wood Sci. Technol.* **1984**, *18*, 255–265. [[CrossRef](#)]
15. Hassan, K.T.; Horacek, P.; Tippner, J. Evaluation of stiffness and strength of Scots Pine wood using resonance frequency and ultrasonic techniques. Dynamic test of wood. *BioResources* **2013**, *8*, 1634–1645. [[CrossRef](#)]
16. Najafi, S.K.; Bucur, V.; Ebrahimi, G. Elastic constants of particleboard with ultrasonic technique. *Mater. Lett.* **2005**, *59*, 2039–2042. [[CrossRef](#)]
17. Brashaw, B.K.; Bucur, V.; Divos, F.; Gonçalves, R.; Lu, J.; Meder, R.; Pellerin, R.F.; Potter, S.; Ross, R.J.; Wang, X.; et al. Nondestructive testing and evaluation of wood: A Worldwide Research Update. *For. Prod. J.* **2009**, *59*, 7–13.
18. Chiu, C.M.; Lin, C.H.; Yang, T.H. Application of nondestructive methods to evaluate mechanical Properties of 32-Year Old Taiwan Incense Cedar (*Calocedrus formosana*) wood. *BioResources* **2013**, *8*, 688–700. [[CrossRef](#)]
19. Proto, A.R.; Macri, G.; Bernardini, V.; Russo, D.; Zimbalatti, G. Acoustic evaluation of wood quality with a non destructive method in standing trees: A first survey in Italy. *IForest* **2017**, *10*, 700–706. [[CrossRef](#)]
20. Yang, J.L.; Fortin, Y. Evaluating strength properties of *Pinus radiata* from ultrasonic measurements on increment cores. *Holzforschung* **2001**, *55*, 606–610. [[CrossRef](#)]
21. Horáček, P.; Tippner, J. Nondestructive evaluation of static bending properties of Scots Pine wood using stress wave technique. *Wood Res.* **2012**, *57*, 359–366.
22. Park, Y.I.D.; Koubaa, A.; Brais, S.; Mazerolle, M.J. Effects of cambial age and stem height on wood density and growth of jack pine grown in boreal stands. *Wood Fib. Sci.* **2009**, *41*, 346–358.
23. Ourais, M. Variations intra-arbres de la largeur du cerne, de la masse volumique du bois et des propriétés morphologiques des trachéides de l'épinette noire (*Picea Mariana* (MILL.) B.S.P) avant et après traitements sylvicoles. Master's Thesis, Université du Québec en Abitibi-Témiscamingue, Rouyn-Noranda, QC, Canada, 2012.
24. Grabner, M.; Wimmer, R.; Gierlinger, N.; Evans, R.; Downes, G. Heartwood extractives in larch and effects on X-ray densitometry. *Can. J. Res.* **2005**, *35*, 2781–2786. [[CrossRef](#)]
25. Hernández, R.; Koubaa, A.; Beaudoin, M.; Fortin, Y. Selected mechanical properties of fast-growing poplar hybrid clones. *Wood Fib. Sci.* **1998**, *30*, 138–147.
26. Koubaa, A.; Isabel, N.; Zhang, S.Y.; Beaulieu, J.; Bousquet, J. Transition from juvenile to mature wood in black spruce (*Picea Mariana* (Mill.) B.S.P.). *Wood Fib. Sci.* **2005**, *37*, 445–455.
27. Koubaa, A.; Zhang, S.Y.; Isabel, N.; Beaulieu, J.; Bousquet, J. Phenotypic correlations between juvenile-mature wood density and growth in black spruce. *Wood Fib. Sci.* **2000**, *32*, 61–71.
28. Jeong, G.Y.; Zink-Sharp, A.; Hindman, D.P. Tensile properties of earlywood and latewood from loblolly pine (*Pinus taeda*) using digital image correlation. *Wood Fib. Sci.* **2009**, *41*, 51–63.
29. Roszyk, E.; Molinski, W.; Kaminski, M. Tensile properties along the grains of earlywood and latewood of scots pine (*Pinus Sylvestris* L.) in dry and wet state. *BioResources* **2016**, *11*, 3027–3037. [[CrossRef](#)]
30. Cramer, S.; Kretschmann, D.; Lakes, R.; Schmidt, T. Earlywood and latewood elastic properties in loblolly pine. *Holzforschung* **2005**, *59*, 531–538. [[CrossRef](#)]

31. Moliński, W.; Roszyk, E.; Puszyński, J. Variation in mechanical properties within individual annual ring of the resonance spruce wood [*Picea abies* (L.) Karst]. *Dev. Ind.* **2014**, *65*, 215–223.
32. Mott, L.; Groom, L.; Shaler, S. Mechanical properties of individual southern pine fibers. Part II. Comparison of earlywood and latewood fibers with respect to tree height and juvenility. *Wood Fib. Sci.* **2002**, *34*, 221–237.
33. Vehniäinen, A. Single Fiber Properties—A Key to the Characteristic Defibration Patterns from Wood to Paper Fibers. Ph.D. Thesis, Helsinki University of Technology, Helsinki, Finland, 2008.
34. Jeong, G.Y.; Hindman, D.P. Modeling differently oriented loblolly pine strands incorporating varying intraring properties using a stochastic finite element method. *Wood Fib. Sci.* **2010**, *42*, 51–61.
35. Ross, R.J. *Nondestructive Evaluation of Wood*, 2nd ed.; Forest Products Laboratory, Forest Service, WI: U.S. General Technical Report FPL-GTR-238; Department of Agriculture: Madison, WI, USA, 2015.
36. Sales, A.; Candian, M.; De Salles Cardin, V. Evaluation of the mechanical properties of Brazilian lumber (*Goupia glabra*) by nondestructive techniques. *Constr. Build. Mater.* **2011**, *25*, 1450–1454. [[CrossRef](#)]
37. Evans, R.; Ilic, J. Rapid prediction of wood stiffness from microfibril angle and density. *For. Prod. J.* **2001**, *51*, 53–57.
38. Lasserre, J.; Mason, E.G.; Watt, M.S.; Moore, J.R. Influence of initial planting spacing and genotype on microfibril angle, wood density, fiber properties and modulus of elasticity in *Pinus radiata* D. Don corewood. *For. Ecol. Manag.* **2009**, *258*, 1924–1931. [[CrossRef](#)]
39. Russo, D.; Marziliano, P.A.; Macri, G.; Proto, A.R.; Zimbalatti, G.; Lambardi, F. Does Thinning Intensity Affect Wood Quality? An Analysis of Calabrian Pine in Southern Italy Using a Non-Destructive Acoustic Method. *Forests* **2019**, *10*, 303. [[CrossRef](#)]
40. Wang, X.; Ross, R.J.; McClellan, M.; Barbour, R.J.; Erickson, J.R.; Forsman, J.W.; McGinnis, G.D. Nondestructive evaluation of standing trees with a stress wave method. *Wood Fib. Sci.* **2001**, *33*, 522–533.



© 2019 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<http://creativecommons.org/licenses/by/4.0/>).