Assessing ultrastructure and density properties to predict wood hardness of young fast grown plantation teak

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Abstract. The use of plantation timber has been increasing to supply raw material for timber manufacturing. Super teak (Tectona grandis Linn. f), a fast grown teak planted in Indonesia was successfully harvested in very young age (5 years old). This frequently rises queries whether the young super teak could meet the minimum quality criteria for wood working. Investigation on wood properties should be undertaken to measure the required wood properties. However, physical and mechanical testing requires a long time process and large size samples. Therefore, a non-destructive method to predict wood properties of standing trees need to be developed. This study aims to assess ultrastructure and density properties to predict wood hardness of young fast grown plantation teak. Samples were collected from super teak plantation in East and West Java. Crystallinity and its quantifiers were measured using X-Ray Diffraction. The hardness was determined using an Instron® universal strength testing machine. The result showed that wood density was significantly related to wood hardness for all structural directions; the power relationship explained 52-56% of the variation. Degree of crystallinity (DC) and crystallite width were found as parameters affecting wood hardness. DC was positively correlated to wood hardness for all structural directions (r=0.2; r²=0.04), whereas crystallite width was negatively correlated to radial and end-grain hardness (r=0.2; r²=0.04). The power of prediction increased only by 1-5% when crystallinity factors were included with density, thus the extra effort of using XRD is not warranted for non-destructive prediction of wood hardness.

1. Introduction
Young fast grown plantation teak has been widely developed to meet the high demand of timber for manufacturing in Indonesia. Super teak is one of fast grown teak (Tectona grandis Linn. f) derived from several clones. This has facilitated plantation rotations as short as 5 years. Harvesting of super teak started in 2012. Working with plantation timbers often raises questions if the trees have met minimum requirements for particular wood products. Such questions can be only answered if the information and data on wood properties are available. Generally, testing physical and mechanical properties necessitates time consuming destructive tests which are not suitable for predicting such properties from standing trees. Consequently, non-destructive techniques are desirable to enable low cost and rapid assessment of important wood properties.
The standard method for determining wood hardness requires destructive testing and the preparation of large standard size specimens 50 x 50 x 150 mm, using the standard Janka test method [1]. However, the standard method is time consuming and relatively costly and cannot be applied directly for predicting wood hardness of standing trees. Therefore, a non-destructive technique (NDT) to predict wood hardness in standing trees would provide significant benefits for evaluating optimal silvicultural treatments, and for selecting suitable trees for tree improvement programs.

Some non-destructive techniques have been described for measuring hardness, such as acoustic tomography [2] and nano indentation [3-5]. However, the results were inadequate because of many factors [6]: poor correlation between apparent acoustic velocity of the tomograms, the measured ultrasonic velocity, and end-hardness of small cubes was likely to have been associated with the wood anisotropy.

Another technique involving continuous nano indentation of single fibres for predicting wood hardness also experienced some problems because of the presence of extractives in the fibres [3]. With the hypothesis that any change in the smallest wood structure may reflect the wood properties at a higher level, Vincent et al. [5] applied nano indentation to the S2 layer and middle lamella at the secondary cell wall on early and late wood fibres to predict wood hardness obtained on the Brinell test [5]. The proposed model produced high adjusted R² (0.77) for predicting the average ring density. However, the authors concluded that the location of nano indentation measurement, moisture, and extractive content probably influenced the relationships. Finally they recommended that nano-measurement is too far removed from representing macro wood mechanical properties stemming from the complexity of wood structural hierarchy [5].

Other non-destructive testing approaches for wood hardness determination were further constrained by the complexity of the wood structure: the anisotropy behavior, the presence of moisture and the extractive content in the cell wall where nano indentation were noted [4-6]. It is well known that the physico mechanical properties of wood are controlled by three factors: the proportion of void volume (which can be determined from the wood density); the organization of the cell wall ultrastructure (in addition to the ultrastructure of the cell wall, variety and proportion of cell types), and the moisture content [7]. In addition, potentially wood extractives may influence wood density and need to be accounted for [7]. When density is used as a predictor of wood hardness, some of the aforementioned constraints may be overcome. Density, combined with the major cell wall structural component cellulose, was considered that it might provide additional information for predicting wood hardness.

As cellulose is the main component of wood and it acts as the skeletal polysaccharide of cell wall [8], it can be assumed from basic principles, that it should be related to wood hardness. The characteristics of cellulose can be quantified by crystallinity. Reportedly crystallinity affects significantly the physical, mechanical and chemical properties of cellulose fibres, including wood hardness [9,10]. This study considers the usefulness of crystallinity and its quantifiers (degree of crystallinity, dimension of crystallite and Micro Fibril Angle (MFA) as a non-destructive indicator of wood hardness.

Wood hardness is an important property for flooring, furniture products and structural utilization. It reflects a measure of resistance to indentation [11-13] and provides an indication of how well the wood performs in relation to wear and marking [14]. The standard test of hardness is empirical; it represents the force necessary to embed a hardened-steel hemisphere with diameter 0.444 inch [11,12]. Hardness is a practical mechanical property which is useful for identifying wood species that are suitable for flooring [15,16]. The selection of a hard timber can provide improved resistance to indentation and abrasion, but for domestic use, other aspect than hardness such as appearance sometimes determine the choice [17]. Therefore, this study aims to develop non-destructive test for predicting hardness based on ultrastructure and density.
2. Materials and Methods

2.1. Specimen preparation

Eighteen trees ranging from small, medium and large diameter stems from super teak plantation forests in Bogor, West Java, Indonesia, and Magetan, East Java, Indonesia (Error! Reference source not found.), were used for hardness measurements. For developing non-destructive testing techniques, fifteen cm thick discs were taken from five fixed sampling heights (60, 80, 100, 120, and 130 cm above ground), at middle (50% of the tree height), and at the top of the tree, that is with minimum stem diameter of 7 cm. This sampling approach was similar to that prescribed by Raymond and Muneri [18] for the determination of optimal sampling heights.

The discs (seven discs from each tree) were used for density, crystallinity and hardness measurements. Hardness samples, measuring 25 mm (radial) x 25 mm (tangential) x 100 mm (longitudinal) were prepared from each disc aiming to preserve true radial and tangential directionality [19]. Specimen’s radial position was also denoted as heartwood (HW), transition-wood (TZ) and sapwood (SW) accordingly. Six replications for each zone (depend on disc/stem diameter) were used.

Table 1. Tree selection used in the study

| No | Tree code | Diameter (cm) | Height* (m) | Clone** | Tree code | Diameter (cm) | Height* (m) | Clone** |
|----|-----------|--------------|-------------|---------|-----------|--------------|-------------|---------|
| Site 1 (West Java) | Site 2 (East Java) |
| 1 | SA22 | 30.25 | 16.0 | A | 198 | 29.3 | 10.5 | B |
| 2 | AC18 | 31.40 | 18.2 | B | 12 | 29.3 | 12.0 | C |
| 3 | AC23 | 32.20 | 13.6 | A | 101 | 28.7 | 9.2 | C |
| Class Diameter 1: large (> 26 cm) |
| 1 | S50 | 20.7 | 11.5 | - | 108 | 21.5 | 8.50 | - |
| 2 | S74 | 21.3 | 9.1 | - | 153 | 24.2 | 6.50 | - |
| 3 | S105 | 22.9 | 11.4 | - | 78 | 24.5 | 8.65 | - |
| Class Diameter 2: medium (17-25 cm) |
| 1 | S89 | 14.3 | 9.65 | - | 27 | 14.3 | 6.4 | - |
| 2 | S53 | 11.9 | 8.90 | - | 2 | 16.6 | 6.7 | - |
| 3 | S120 | 14.6 | 10.10 | - | 10 | 16.2 | 5.8 | - |

*Economic height: height of stem at the top with minimum diameter of 7 cm
**At the time of sample collection, only three clones were available in the plantations. In this study, DNA testing to check the tree clone was conducted only on wood from the large diameter classes.

Specimens for crystallinity measurement were end-matched with the density and shrinkage specimens as shown in Figure 1. The samples were 50 mm (tangential direction) x 15 mm (longitudinal direction) x 30-40 µm (radial direction).

2.2. Testing and calculation

Up to 42 samples (depending on the tree stem diameter), measuring 50 x 50 x 150 mm for Site 1 and 126 samples measuring 30 x 30 x 90 mm for Site 2, were prepared from each tree. There were difficulties in obtaining sufficiently large test specimens from the small diameter trees when preparing samples for hardness testing for Site 1 (wet site) where fewer specimens and sometimes no specimens especially at the top height were represented. Therefore, the size of hardness specimens at Site 2 (dry site) were decreased to 30 x 30 mm with length 90 mm (ratio 1:1:3). According to the Australian Standards for mechanically testing small clear specimens of timber [1], the standard dimension of
hardness specimens are 50 x 50 x 150 mm, and the test may also be made on a specimen of any other size, as long as the thickness is at least 25 mm.

The samples in this study were identified as heartwood (HW), transition-wood (TZ) and sapwood (SW) on the basis of wood colour, and the hardness was determined using an Instron® type 3369 universal strength testing machine according to the Australian Standards for mechanically testing small clear specimens of timber [1]. Wood specimens were tested in the radial (R hardness), tangential (T hardness) and longitudinal (end hardness) grain directions. The reason for testing all wood faces was to ensure a more representative value of each specimen [20] and to enable radial and tangential hardness to be tabulated as side hardness for ready comparison with data for other species. Before and after testing, the weight and dimensions of each specimen were measured for determination of wood density and moisture content.

Crystallinity and its quantifiers were measured using X-Ray Diffraction (XRD) (Shimadzu® QP-500). The X-ray beam was powered with a 40kV, 30mA source and scans made in the range from 0 - 40 degrees at a scan speed of 2 degrees per minute. The degree of crystallinity (DC) was calculated as the ratio between the crystalline and total non-crystalline and amorphous regions as shown in Figure 2. The dimension of crystallites (CW-width and CL-length) was determined using the Scherrer formula Equation 1 [21] for diffraction intensities (002) and (040), usually in the angular range from 20 to 24 degrees and from 32 to 37 degrees, respectively [22]. Cellulose lattice spacing (d) was calculated using Bragg Equation (Equation 2) [22]. The number of crystallite planes for each of the diffraction intensities was calculated by dividing the crystallite dimension with the crystallite lattice spacing (d).

Equation 1 Scherrer formula for crystallite size determinations

\[ B_{(2\theta)} = \frac{K \lambda}{L \cos \theta'} \]  

where, K = shape factor, 0.9 used to determine cellulose crystallite dimensions for the (200) reflection and 1.0 in the case of (004) reflection; \( \lambda \) = wave length of X-rays produced from a Cu target (0.154 nm); \( B (2\theta) \) = Peak width – Full Width at Half Maximum (FWHM), (radians); and \( \theta' = \frac{1}{2} \) the diffraction angle, (radians); \( L \) = Crystallite size.
Equation 2 Bragg Equation calculation of lattice spacing

\[ d = \frac{\lambda}{2 \sin \theta} \]  

where \( \lambda \) = Wavelength of the X-ray beam (0.154 nm, CuK\(\alpha \)); \( \theta \) = Bragg angle

Micro Fibril Angle (MFA) was determined from the (002) reflection plane, and calculated by using Cave’s method (Equation 3). Abe and Yamamoto’s equation (Equation 4) an alternative method was used for comparison [23]. The T parameter was calculated according to Stuart and Evans [23] by manually drawing tangents to the sides of plots of the diffraction arcs.

Equation 3 Cave’s method for calculating MFA

\[ \text{MFA} = 0.6 \ T \]  

Equation 4 Yamamoto’s equation for calculating MFA

\[ \text{MFA} = 1.575 \times 10^{-3} \ T^3 - 1.431 \times 10^{-1} \ T^2 + 4.693 \ T - 36.19 \]

2.3. Data analysis

Saphiro-Wilk’s W test (Statistica®) test was employed to determine if the density and wood hardness data were normally distributed. Variations between sites, tree growth rates and within trees were analyzed using T-test and ANOVA for normally distributed data, and the Mann-Whitney U test and Kruskal-Wallis ANOVA was applied for the above analyses when the data were not normally distributed.

MFA, Degree of Crystallinity (DC) and crystallite lattice spacing (d\(\text{002}\)) were distributed normally according to Saphiro-Wilk’s W test (Statistica®). However, crystallite width, crystallite plane (N\(\text{002}\)) at diffraction intensity (002), crystallite length, crystallite lattice spacing (d\(\text{040}\)) and crystallite planes (N\(\text{040}\)) at the 040 diffraction intensity were not normally distributed according to the above test for normality. Statistical analysis was performed with T-test and ANOVA for normally distributed data and Mann-Whitney U-Test and Kruskal-Wallis ANOVA for non-normal data.
3. Results and Discussion

3.1. Wood hardness and wood density

Wood hardness increases with density, but may not be linear [24]. Extensive tests of many wood species over a wide range of specific gravities showed that mathematical relationships were either parabolic or exponential of n degree [7,11,24]. However, mechanical properties are not all affected to the same degree by the changes in specific gravity [7], for example hardness of softwoods with \( n = 1.50 \) increases with specific gravity much more rapidly than MOE with \( n = 0.84 \). Equation 5 shows the general mathematical relationship between specific gravity and mechanical properties. Equation 5 shows the general mathematical relationship between specific gravity and various strength properties [11].

\[
S = aG^n
\]  

where, \( S \) is the strength property under consideration; \( G \) is specific gravity of the wood tested, and \( n \) is the slope of the curve.

Forest Product Laboratory in cooperation with the University of Wisconsin, Madison, developed formula for predicting hardness which has been presented in Equation 6 as follows:

\[
H_{\text{end grain}} = 21351 \, G^{2.25}; \quad H_{\text{radial}} = 16574 \, G^{2.25}; \quad H_{\text{tangential}} = 16992 \, G^{2.25}
\]  

When the relationship in Equation 6 was applied to the super teak and compared with the actual wood hardness from standard testing, the result showed that 54% (46-62%) of the actual hardness varied between the limits of 87-111% of the computed hardness. The range 87-111% (specifically 88-111% for radial hardness, 90-111% for tangential hardness, and 87-111% for end-grain hardness) was determined based on the limits obtained from the study by Forest Products Laboratory [25] from a comparison of actual hardness and computed values. In that comparable study by Forest Products Laboratory [25], the actual values of approximately half of the specimens examined were observed to vary between the 87-111% of the computed values; the remainder, half (one-fourth of the total number) exceeded 111%, and the rest was below 87%. In this present study, by applying the Error!
Reference source not found., the actual values were 20% (13-29%) below 87% and 23% (17-28%) exceeded 111% (Table 3) which is practically similar to the result obtained by Forest Products Laboratory [25].

The well accepted models (Equation 6) were developed based on empirical data fits for a large number of species. Because the numbers of species used to obtain the relationship by Forest Product Laboratory were large, they were collectively expected to exhibit a wider range in density than that for a single species; therefore the $K$ and $n$ coefficients are expected to be different for a species like super teak.

Table 2 presents Pearson correlations between wood hardness and specific gravity of super teak from both the wet and dry sites. The result showed that air dry specific gravity was significantly correlated with wood hardness positively, with $r$ values 0.7 (0.72-0.75). Specific gravity in this study was adjusted based on the density at the test moisture content (approximately at 15% MC), using Equation 7. Equation 7 Method of computing specific gravity at any moisture content [11].

$$S = \frac{W_M}{V(1 + \frac{M}{100})}$$

(7)

where $S$ is specific gravity, $W_M$ is the weight of sample at $M$ moisture content, $V$ is the volume-equivalent of the sample, and $M$ is the moisture content expressed as the percentage of the oven dry weight.

| Wood Hardness | R   |
|---------------|-----|
| Radial        | 0.73|
| Tangential    | 0.75|
| End           | 0.72|

Correlations marked in red are significant at $p < 0.05$

To obtain new $K$ and $n$ coefficients for super teak, specific gravity (G) and measured hardness values were regressed and a trend-line was fitted (Figure 3-5). Comparable results of new parabolic equations for predicting hardness with the actual hardness values are presented in Table 3.

![Figure 3. Relationship between radial hardness and specific gravity](image-url)
Table 3. Comparable results of parabolic equations for predicting hardness of by using existing equations from Forest Products Laboratory and proposed equations for super teak with the actual hardness values

| Equations      | Radial Hardness | Tangential Hardness | End-grain Hardness |
|----------------|-----------------|---------------------|--------------------|
|                | <88% 88-111%*   | >111%               | <90% 90-111%*      | >111%              | <87% 87-110%* | >110% |
| Existing equations |                 |                     |                    |
| $H_r = 16574 G^{2.25}$ | 29            | 17                  | 19                 | 33                  | 13            | 62     | 25 |
| $H_t = 16992 G^{2.25}$ | 46            | 19                  | 53                 | 28                  | 13            | 62     | 25 |
| $H_e = 21351 G^{2.25}$ | 53            | 22                  | 23                 | 13                  | 65            | 22     |    |
| New equations   |                 |                     |                    |
| $H_r = 14072 G^{2.10}$ | 22            | 20                  | 23                 | 13                  | 65            | 22     |    |
| $H_t = 14450 G^{2.01}$ | 51            | 56                  | 65                 | 13                  | 65            | 22     |    |
| $H_e = 11554 G^{1.45}$ | 27            | 20                  | 23                 | 13                  | 65            | 22     |    |

*The range of ratio between actual and computed hardness values were determined based on the study by Newlin and Wilson [25]; $H_r$ = Radial hardness; $H_t$ = Tangential hardness; $H_e$ = End-grain hardness

The results from Table 3 show that the hardness at unit density ($K$) of super teak was lower than that for the previously published models developed by Forest Product Laboratory. Furthermore, the increases with specific gravity were smaller for super teak, possibly because the range of specific gravity of the 5 years super teak material is smaller; only 0.3 to 0.6. It is interesting to observe that in this case the relationship between wood hardness and specific gravity is almost linear (the $n$ value is almost 1), for end-grain.
The new parabolic equations provided a better fit than the existing models (Equation 6), as shown by increases of the ratio between actual and computed hardness values for all hardness directions (Table 3). Consequently, the new K and n coefficients provided an improvement for predictions. The results obtained from this study are higher to those from Newlin and Wilson [25]. The result indicates that by using specific gravity as an approach, the maximum power of prediction for estimating wood hardness based on specific gravity is in the order of 65%.

The power of prediction of wood hardness was further tested by using the new coefficients by regression analysis from Statistica® (Table 4). The result confirmed that in general, the proposed models are significant but specific gravity on its own can only explain 52-56% of the variation in wood hardness. To improve predictability of wood hardness using additional properties, other wood properties are expected to also influence wood hardness, and consequently crystallinity was considered in that context as it has previously been reported that wood hardness increases with increasing crystallinity [9,10,23]. Table 4 Coefficients of determination (R^2) of proposed parabolic equations for predicting wood hardness based on specific gravity at both wet and dry sites.

**Table 4. Simple correlation between wood hardness and air-dry specific gravity of super teak at both wet and dry sites**

| Wood Hardness | r   | R^2  | P Value |
|---------------|-----|------|---------|
| Radial        | 0.72| 0.52 | 0.00    |
| Tangential    | 0.75| 0.56 | 0.00    |
| End           | 0.72| 0.52 | 0.00    |

Correlations marked in red are significant at p <0.05

### 3.2. Wood hardness and crystallinity

Considering that the specific gravity only explained approximately half of the variation in wood hardness, and that according to Panshin and Zeeuw [26] the mechanical properties of wood can be influenced by other properties, crystallinity was considered as a potential characteristic based on the association between the amount and arrangement of cellulose crystallites on the physical, mechanical and chemical properties of cellulose fibres, including wood hardness [10,27].

Table 5 presents the Pearson correlation between wood hardness and crystallinity properties determined at the opposite-end matched specimens by X-ray Diffraction. The result showed that degree of crystallinity (DC) was significantly positively correlated to hardness for all hardness directions (r = 0.2). Furthermore, crystallite width was negatively correlated to all hardness direction, and significant only on radial and end-grain hardness (r = -0.2). All the correlations were significant but very low. The results from DC and crystallite width were consistent with the relationships published by Damayanti et al. [28] and Damayanti et al. [29].

Wood crystallinity (degree of crystallinity) is defined as the weight fraction of crystalline material to non-crystalline material in wood [10]. This crystalline region is considered to be in the state of relatively good three-dimensional order [9]. An increase in the degree of crystallinity is also expected to give rise to increases in wood hardness [8,9,15].

**Table 5. Pearson correlation between wood hardness and crystallinity for super teak at both wet and dry sites**

| Wood Hardness (N) | MFA (°) | DC (%) | Crystallite Width (nm) | d002 | N002* | Crystallite Length (nm) | d040 | N040* |
|-------------------|---------|--------|------------------------|------|-------|-------------------------|------|-------|
| Radial            | -0.01   | 0.21   | -0.24                  | 0.12 | -0.24 | 0.11                    | -0.02| 0.11  |
| Tangential        | -0.10   | 0.16   | -0.07                  | -0.05| -0.06 | 0.05                    | -0.02| 0.05  |
| End               | 0.06    | 0.17   | -0.19                  | 0.03 | -0.18 | 0.00                    | 0.03 | -0.01 |

Correlations marked in red are significant at p <0.05; * the variable is not used in the regression
The negative correlation obtained between crystallite width and hardness in this study meant that smaller crystallite size resulted in harder wood possibly because of reasons of homogeneity via the expectation that smaller crystallites are associated with greater homogeneity and compact substance, thus potentially harder wood (Prof. Voichita Bucur, pers. comm.).

Considering the effect of MFA on wood hardness as another property of crystallinity it was found not to be usefully related to hardness. The result of this study is in good agreement with Gindl et al. [30] who reported that hardness determined by nano-indentation was independent of MFA, and suggested that indentation hardness was governed by yield processes in the matrix of the middle lamella. While MFA is known to be related to the wood elastic modulus [31], it is not likely to be useful for predicting wood hardness as confirmed by Konnerth et al. [32].

When general regressions were developed, only radial and end-grain hardness were found to be significantly related to the DC and crystallite width (Table 6), with coefficient of determinations (R²) 0.13 and 0.08, for radial and end-grain hardness respectively. This outcome implies that the crystallinity can only explain 8-13% of the variation in radial and end-grain hardness. With such small improvements it is hardly practically worthwhile using XRD. When crystallinity and specific gravity were combined, the power of prediction increased; from 52% to 57% for radial hardness, from 56% to 57% for tangential hardness, and from 52% to 54% for end-grain hardness.

| Wood Hardness (N) | R² | Adjusted R² | P Value |
|-------------------|----|-------------|---------|
| Radial            | 0.13 | 0.09        | 0.00 |
| Tangential        | 0.05 | 0.01        | 0.26 |
| End               | 0.08 | 0.05        | 0.03 |

Table 6. Coefficients of determination (R²) from regressions for predicting wood hardness of super teak based on ‘crystallinity’ at both wet and dry sites

The effort of using crystallinity to predict wood hardness only improved the prediction of hardness by 1 - 5%. Therefore, it is suggested that, wood density is singly the most useful property for non-destructive prediction even though only approximately 60% of variation in wood hardness can be explained. It is possible that there might be other wood properties not yet proposed and considered in this study which may improve the predictability. Potentially they may include extractive content [26], cellulose and lignin content. According to Ates et al. [33], tangential, radial and cross section Brinell hardness of Pinus brutia Ten. exhibited a “high” Pearson’s correlation with holocellulose and lignin content (r > 0.8), i.e. of a similar order to that obtained with density.

4. Conclusion
Wood density was significantly related to wood hardness for all structural directions; the power relationship explained 52-56% of the variation of the measured hardness. By using only crystallinity factors as independent variables, degree of crystallinity (DC) and crystallite width were found as
parameters affecting wood hardness. DC was positively correlated to wood hardness for all structural
directions ($r = 0.2; r^2 = 0.04$), whereas crystallite width was negatively correlated to radial and end-
grain hardness ($r = 0.2; r^2 = 0.04$). The power of prediction increased only by 1-5% when
‘crystallinity’ factors were included with density, thus the extra effort of using XRD is not warranted
for non-destructive prediction of wood hardness.

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