

THE INFLUENCE OF LONGITUDINAL LENGTH AND SATURATION TIME ON THE PERCENTAGE OF VOID VOLUME FILLED AND WOOD DENSITY DETERMINED BY THE MAXIMUM MOISTURE CONTENT METHOD**İlker USTA**

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ABSTRACT

An intensive investigation on the effects of longitudinal length (5 and 10 mm) and saturation time (5 and 10 days) on density determination of Sitka spruce (*Picea sitchensis* (Bong.) Carr.) by the maximum moisture content method showed that the longitudinal uptake of deionised water decreased as the length of specimen increased. According to the results, the shorter longitudinal lengths were effectively saturated in comparison to the longer lengths, and also their percentage of void volume filled were observed at the highest level. The reason for the decreased absorption with increased specimen length was due to the saturation time factor by vacuum process. If vacuum applied was prolonged for further period, further movement and saturation could have occurred in longer blocks. The shorter blocks of short longitudinal flow pathways tended to be saturated more rapidly in short period of vacuum application than longer flow pathways. In this case, the maximum moisture content method gives lower variation only when the experimental blocks are properly saturated, hence the short block length (5 mm in longitudinal direction) and the longer saturation time (10 days) can be suggested to use as to be the most reliable conditions to determine wood density of Sitka spruce.

Key Words: Sitka Spruce, Density Determinations, Maximum Moisture Content Method, Longitudinal Length, Saturation Time, Percentage of Void Volume Filled.

LİF YÖNÜ UZUNLUĞUNUN VE ISLATMA SÜRESİNİN EN YÜKSEK RUTUBET ORANI YÖNTEMİNE GÖRE BELİRLENEN BOŞLUK HACİM DOLULUK YÜZDESİNE VE AĞAÇ MALZEME ÖZGÜL AĞIRLIĞINA ETKİSİ**ÖZET**

En yüksek rutubet oranı yöntemi ile Sitka ladini (*Picea sitchensis* (Bong.) Carr.)'nin özgül ağırlık belirlemede lif yönü uzunluğu (5, 10 mm) ve ıslatma süresi (5, 10 gün) etkilerinin araştırılması için yapılan bu çalışmada, sıvı alımının lif yönü uzunluğuna bağlı olarak değiştiği ve uzunluğun artmasıyla damıtılmış su nüfuz derinliğinin azaldığı ortaya çıkarılmıştır. Lif yönü kısa hazırlanmış deney parçalarının daha fazla ıslandıklarından, boşluk hacimlerindeki su içerme miktarının lif yönü uzun hazırlanmış deney parçalarına göre daha yüksek düzeyde gerçekleştiği belirlenmiştir. Uzunluğun artmasına bağlı olarak sıvı absorplama oranının azalması, vakumlama işlemi ile gerçekleştirilen ıslatma süresinin bir faktörü olarak ortaya çıkmaktadır. Eğer vakum uygulaması, deneyde belirlenen en uzun test süresinden biraz daha uzatılırsa, sıvı içerme miktarının lif yönü uzun hazırlanmış deney parçalarında da artabileceği beklenebilir, ancak bu ıslanmanın hızı ve miktarı lif yönü kısa olanları kadar kapsamlı olamayacaktır. Çünkü, kısa parçalar

uzunlara göre kısa süreli vakum uygulamasında hızlı ve etkin bir şekilde ıslanmaktadırlar. Bu noktada, en yüksek rutubet oranı yöntemi ile eğer deney parçaları damıtılmış su ile tamamen ıslanırlarsa, grubu oluşturan her bir parçanın grup ortalamasına olan uzaklığı en aza inmektedir. Bu nedenle, Sitka ladini'nin özgül ağırlık değerinin belirlenmesinde, lif yönünün 5 mm uzunlukta hazırlanması ve 10 gün su içerisinde ıslatılması yönteminin en güvenilir ve geçerli bir koşul olarak kullanılması önerilebilir.

Anahtar Kelimeler: Sitka Ladini, Özgül Ağırlık Belirlemeleri, En Yüksek Rutubet Oranı Yöntemi, Boy Uzunluğu, İslatma Süresi, Boşluk Hacim Doluluk Yüzdesi

1. INTRODUCTION

Density, used here synonymously with specific gravity, is a measure of a quantity of cell wall material contained in a certain volume of the piece of wood and is an index of void volume [1], and is calculated as the ratio of dry weight of wood to its volume and is measured in units such as kilograms per cubic meter. On the other hand, void volume is the amount of empty/air spaces composed by cell cavities and intercellular spaces in a given volume of wood, and thus Siau [2] defined porosity as fractional void volume of wood.

Wood is a cellular/porous material [3; 4] composed of cell wall substances and cavities containing air and extractives [5]. Without cavities and intercellular spaces, the relative density of the cell wall materials is practically constant for all timbers having a specific gravity of 1.53 gm^{-3} on oven-dry mass and volume basis, thus, the cell wall materials are one and half times heavier than water [6]. That is, an ideal physical value for a lignified cellulosic cell wall which is completely non-porous, and so a cubic metre of solid wood would weigh roughly 1500 kgm^{-3} for all species but wood is surely not comprised of 100% cell wall material as it contains air pockets in the cell lumina [5]. The oven-dry density of any wood species is a direct reflection of the amount of space occupied by the wood tissue [6]. Therefore, a value of 1500 kgm^{-3} is a reasonable approximation for the density of oven-dry wood tissue. However, actual value may vary slightly between species and between earlywood and latewood as the proportion of the cell wall constituents varies [5].

Density is primarily determined by the amount of cell wall material in relation to the voids [7], so the density determinations are influenced by the amount of moisture in the wood [8]. Importantly, cell wall substance adsorbs and desorbs moisture from and to the environment since wood is a hygroscopic material. A consequence of this is that cell wall shrinks and swells, thus the relative proportions of cell wall substance and pore space vary greatly, therefore, some variation in values is expected on the basic density which tends to vary significantly between species, within species, between trees, and also within single trees [8; 9]. The weight and density of wood varies between species caused by differences in the ratio of cell wall to air spaces [5]. Differences in density and void volume arise simply from differences in the anatomy of the wood modified by the effect of extractives, and is also derived from anatomical differences such as in cell types and quantitative distribution, thickness of cell walls and size of cell cavities [4].

Mass of a block is readily determined by weighing but the measurement of the volume of a specimen presents more problems. It is possible to measure the linear dimensions of a regular shape piece of wood cut from a plank, and then calculate its volume. But this is rarely convenient, and might be impractical if only a tiny sample is available, e.g. archaeological specimen. It is totally impossible to cut and measure a piece of cell wall. Luckily however, there are a variety of other ways; volume can be measured so these results in measured with different density techniques available such as densitometry [11; 12], gravimetry [13; 14], pycnometry [15], dot-count [16], microphotometer [17], maximum moisture content method [18], and the archimedian method [19].

The purpose of the present work was to compare the effects of longitudinal length (5 and 10 mm) and saturation time (5 and 10 days) for density determination of Sitka spruce (*Picea sitchensis* (Bong.) Carr.) by the maximum moisture content method, and to find out the most reliable experimental conditions for more precise density determination. Although, this study has been carried out by using the one of the softwood species, the experimental findings would also be subject to effective for other softwood species.

2. MATERIALS AND METHODS

2.1. Method: Theoretical

A useful way of determining the volume of an irregular block is by recourse to the known consistent value for the density of cell wall material which is 1.53 gcm^{-3} . The method of determining basic density based on this is known as the maximum moisture content method and has been evaluated by Smith [18], and he derived the following formula (Equation 1) which only two weighings are required to determine the density of wood, i.e. the weight after drying until constant weight (at $103 \pm 2 \text{ }^\circ\text{C}$) divided by the green volume which is measured by the water displacement method [20].

$$d = [\text{Mo} / ((\text{Mo} / \text{G}) + (\text{Ms} - \text{Mo}))] \times 1000 \quad (\text{Equation 1})$$

where (in Equation 1): d is the density of wood sample (kgm^{-3}), Mo is oven-dry weight of sample (g), Ms is weight of sample saturated with deionised water (g), G : density of cell wall substance (1.53 gcm^{-3}) [7; 21].

In this method, before measuring the oven-dried weight, wood must fully be saturated by water to measure the green volume according to the Archimedes principle which was stated that a body immersed in a fluid experiences an upthrust equal to the weight of the fluid displaced. Thus a completely saturated wood block displaces an amount of water which equals its own volume [21].

2.2. Method: Experimental

The air-dried defect free Sitka spruce (*Picea sitchensis* (Bong.) Carr.) sapwood was selected, from which a stake initially $200 \times 25 \times 25 \text{ mm}$ was sized by silk cutting, and its moisture content was nominated approximately 12 percent by conditioning process in a constant temperature and humidity room set at $20 \text{ }^\circ\text{C}$ and 65% relative humidity. As shown in Figure 1, this stake was then cut along its length into 12 blocks of 10 mm in longitudinal length and another 12 of 5 mm. Thereafter, the first 6 blocks of 10 mm and 5 mm were labelled "5 days" to be placed in the desiccator and the other 6 blocks of each length group were marked as "10 days".

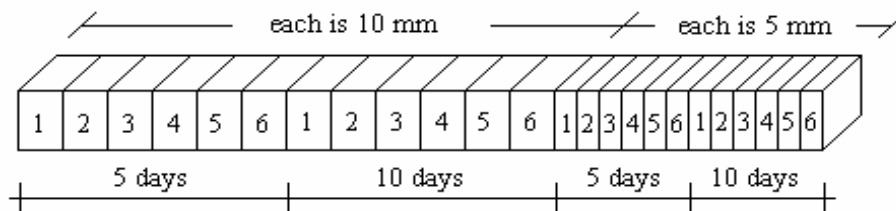


Figure 1. Preparation of the experimental blocks of Sitka spruce according to the two different types of longitudinal length (10 and 5 mm) and of saturation time (5 and 10 days).

Loose fibres were removed with a razor blade, and to obtain actual volume and mass, each experimental block was dimensionally measured with a micrometer and was weighed by a scale. As shown in Figure 2, the blocks (1) were then placed into a vacuum desiccator (2) according to their saturation time, held down 20 mm below deionised water (3) with a wire gauze (4). A fungicide of 25 ml was added to the water. After, the application of vacuum of -80 kPa (600 mmHg) was pulled out for 12 hours, the desiccators were kept closed for 12 hours. This vacuum impregnation procedure was continuously performed for 5 and 10 days. After having finished the soaking time under vacuum, the experimental blocks were assumed to be saturated.

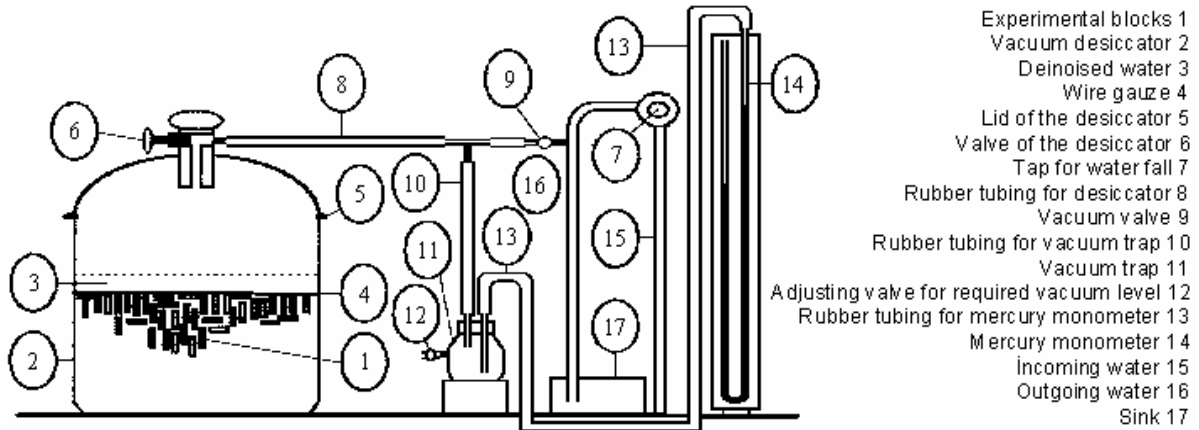


Figure 2. A diagram showing the vacuum apparatus for saturating the samples with deionised water.

The blocks were then individually weighed in a beaker of deionised water to determine the green volume as follows (Figure 3): Firstly, a beaker (1) containing distilled water (2) was put on the pan (3) of the balance. Next, a fine needle (4) was held partly submerged in the water with a clamp (5, 6) and the balance (8) was tared. Thereafter, the needle was taken out, and an experimental block (7), surface dried on tissue paper, was suspended onto the needle. The block was then submerged into the water so that the block was not in contact with the sides or bottom of the beaker. Afterwards, the weight of the block volume was directly read and recorded on a four digit automatic balance. Thus, the saturated block displaced an amount of water which was equal to its own volume in cubic centimeters [5; 20].

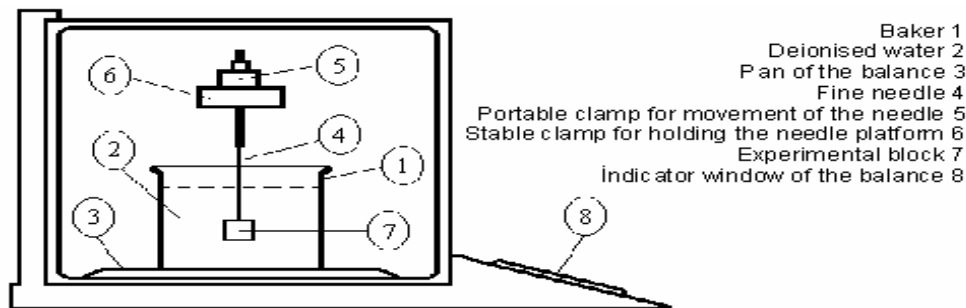


Figure 3. Recording the weight of the displaced volume of water during the measurement of basic density.

Subsequently, all the blocks were dried in an oven at 103 ± 2 °C for 24 hours. After having dried, the experimental blocks were cooled in a desiccator charged with granulated silica and reweighed. After the measurement of the saturated and dried weights, density was calculated for each block by Equation 1.

Since the maximum moisture content method gives lower variation only when the experimental blocks are properly saturated, a further assessment was made in relation to the maximum theoretical possible uptake of each block based on void volume. This takes into account the density. The amount of space available in each block was calculated as an estimation of the maximum volume of deionised water which could be absorbed by wood in a given block volume [22]. This was calculated from Equation 2.

$$VVF = [((M_s - M_g) / V_{blk}) / 1000] / [(1 - (d / 1500)) \times 1000] \quad (\text{Equation 2})$$

where (in Equation 2): VVF is the percentage of void volume filled (%), M_s is weight of sample saturated with deionised water (g), M_g is green weight (before saturation) of sample (g), V_{blk} is stereometric block volume of the experimental sample, d is the density of wood sample (kgm^{-3}) [23].

2.3. Statistical Analysis

All statistical analysis was conducted using the statistical package MINITAB, version 10.51 (Minitab Inc. Minitab for Windows, 1995). Density and the percentage of void volume filled (VVF) data were tested for normality using the Ryan-Joiner test (Minitab 10.51). The normal scores of the data were compared with the experimental data by correlation analyses. The hypothesis of normality was rejected if the calculated

correlation coefficient was below a critical value obtained from tables. Minitab was also used for analysis of variance (ANOVA) and Tukey pairwise comparison tests were performed to identify significant differences between treatment means. Balanced analyses of variance (Balanced ANOVA) was also used to examine the relative importance of the factors influencing both density and the percentage of VVF with all possible interactions [24, 25].

3. RESULTS AND DISCUSSION

The experimental results showed that there was greatest effect of both specimen length and saturation time on void volume filled (VVF) in the longitudinal penetration by deionised water since the VVF was higher in the shorter length specimens with the longer saturation time for water. As is shown in Table 1, the VVF increased from 95% to 100%, as specimen lengths decreased from 10 mm to 5 mm on longitudinal penetration with increasing of the saturation time from 5 days to 10 days. It could also be seen from Table 1 that an increase in density from 428 kgm⁻³ to 437 kgm⁻³ was manifested by a corresponding decrease in VVF from 100% to 95%.

Table 1. Means of the percentage of void volume filled and wood density, and pairwise differences of means between each experimental (length/time) group.

| Longitudinal Length (mm) | Saturation Time (day) | Void Volume Filled (%) | Wood Density (kgm ⁻³) |
|-----------------------------|--------------------------|---------------------------|--------------------------------------|
| 10 | 5 | 95 (1.67) a | 437 (11.88) a |
| 10 | 10 | 96 (0.29) b | 434 (1.57) a |
| 5 | 5 | 97 (0.87) c | 430 (3.60) a |
| 5 | 10 | 100 (0.18) d | 428 (0.61) a |

Values (of void volume filled and of wood density, in Table 1) are means of 6 replicates in each experimental group, and figures in parenthesis are standard deviations. Means within a given column followed by the same letter are not significantly different from each other by Tukey Pairwise Comparison Test at $p < 0.05$ level.

To put it precisely, the first six experimental blocks of 10 mm in longitudinal length which were placed in the vacuum desiccator to saturate by water for 5 days are referred as LT-105 and its last six blocks that were immersed for 10 days as LT-1010, while the first six blocks of 5 mm in longitudinal length which were kept closed in desiccator for 5 days are described as LT-55 and its last six blocks that were saturated for 10 days as LT-510. The ranges of density and VVF values were very close to each other in the blocks of LT-510 with the lowest standard deviations (SD: 0.61 and 0.18), and they were also the most saturated by the deionised water as much as 100% according to the other experimental blocks. Conversely, the actual values of both density and VVF created quite large range between each other in the blocks of LT-105 with the highest standard deviations (SD: 11.88 and 1.67), and these experimental blocks were the least saturated by water as much as 95% than all the other samples. Although the ranges of density and VVF were also narrow in the blocks of LT-1010 with the low standard deviation (SD: 1.57 and 0.29), the mean of the actual values of VVF (96%) was the third in the standing order. These considered ranges were a little wider in the block of LT-55 with the high standard deviation (SD: 3.60 and 0.87), besides its VVF (97%) was the second according to the mean values.

The experimental data of density and the percentage of VVF were also analysed by balanced analysis of variance according to length, time, and length x time. Table 2 shows the results in the form of an analysis of variance according to longitudinal length and saturation time for both density and the percentage of void volume filled (VVF). There was significant effect of longitudinal length on both density ($p=0.023$) and VVF ($p=0.000$). The effect of saturation time was highly significant on VVF ($p=0.000$), although there was no effect of that on density ($p=0.394$). Besides, the interaction between longitudinal length and saturation time was not significant in either density ($p=0.783$) or VVF ($p=0.326$).

Table 2. Analysis of variance for the percentage of void volume filled (VVF) and density indicating significant effects ($p < 0.050$) for length (the longitudinal length of the test block: mm), time (the saturation time under vacuum: day), and length x time.

| Parameter | Source | DF | SS | MS | F | p | |
|-----------|---------------|----|---------|--------|-------|-------|-----|
| VVF | Length | 1 | 65.565 | 62.565 | 68.15 | 0.000 | *** |
| | Time | 1 | 23.661 | 23.661 | 25.77 | 0.000 | *** |
| | Length x Time | 1 | 0.932 | 0.932 | 1.02 | 0.326 | NS |
| | Error | 20 | 18.360 | 0.918 | | | |
| | Total | 23 | 105.519 | | | | |
| Density | Length | 1 | 239.65 | 239.65 | 6.11 | 0.023 | * |
| | Time | 1 | 29.79 | 29.79 | 0.76 | 0.394 | NS |
| | Length x Time | 1 | 3.05 | 3.05 | 0.08 | 0.783 | NS |
| | Error | 20 | 784.58 | 39.23 | | | |
| | Total | 23 | 1057.08 | | | | |

Source: source of variation, DF: degrees of freedom, SS: sum of squares, MS: mean of square, NS: not significant, * significant at 95% level, *** significant at 99.9% level.

The actual results of the percentage of void volume filled (VVF) and density are shown in Figure 4 for comparison of changes in both data across the original locations of the experimental blocks from first block to sixth block of each group of the longitudinal length (10 and 5 mm) and the saturation time (5 and 10 days).

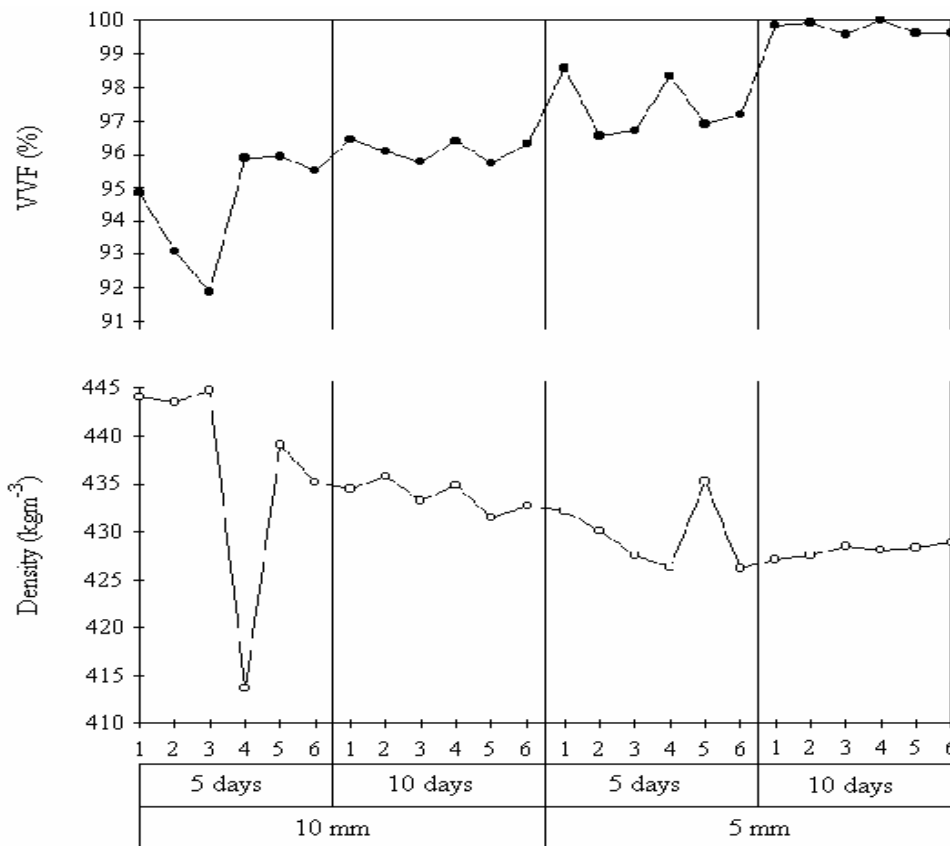


Figure 4. Comparison of changes in the percentage of void volume filled (VVF) and wood density for two different types of longitudinal length and saturation time.

In Figure 4: the first 12 blocks of 24 have 10 mm length, the others have 5 mm; and the first 6 blocks of each length group were placed in the desiccator for 5 days while the last 6 were left for 10 days.

Although Figure 4 revealed variability in VVF, the general trend was that shorter length blocks were saturated rapidly compared to longer blocks. The voids in the shorter length blocks were filled rapidly within short period of saturation time. The longer blocks were less saturated but if the period was prolonged better

saturation of water were likely to achieve. It can be inferred from the experimental results that the longitudinal penetration of deionised water in shorter length specimens was obviously greater than the longer length specimens under the same condition of the saturation process. As the period of vacuum increased, more deionised water were driven into the wood capillary system where the deionised water flowed more effectively from one tracheid lumen to another via unspirated bordered pits. This resulted in complete saturation or the more fillings of the void volumes along the grain as the period of vacuum increased. However, if voids were occupied by moisture or extractive or blockages of flow pathways by resins, the flow of the deionised water would not be possible even by doubling the period of vacuum increase [26].

4. CONCLUSIONS

It was found that the longitudinal uptake of deionised water along the wood capillary system decreased with increasing specimen length. The shorter length flow paths were saturated more effectively than the longer length flow paths. That is, more void space was filled and the higher retentions (the percentage of void volume filled) were achieved in shorter length specimens compared to longer length specimens.

The obvious reason for much more penetration of deionised water for shorter length specimens compared to longer ones could be due to the lower number of tracheids in the longitudinal direction and due to the longer period of vacuum application. Therefore, the longitudinal penetration of deionised water were very much dependent on the period of vacuum, availability of void volume. When the vacuum application increased the penetration of the deionised water increased as seen in this experiment. The period of vacuum was the important factor which determined amount of deionised water retained in the test blocks.

According to Olesen [20], the maximum moisture content method to determine wood density gives lower variation only when the experimental blocks are properly saturated. This statement fit the findings of the present study. In this case, it has been observed that the most saturated experimental blocks by the deionised water were the blocks of LT-510 (5 mm in longitudinal length, and 10 days saturation time) as much as 100%, and the ranges of wood density and the percentage of void volume filled (VVF) values were very close to each other in these blocks with the lowest standard deviations (SD: 0.61 and 0.18). Hence, the short block length (5 mm in longitudinal direction) and the longer saturation time (10 days) can be suggested to use as to be the most reliable conditions to determine wood density of Sitka spruce (*Picea sitchensis* (Bong.) Carr.).

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