Experimental Study of Impregnation Birch and Aspen Samples

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An experimental study of wood impregnation was implemented by applying centrifugal methods. The impregnants were a 10% aqueous solution of potassium chloride and a 2% aqueous solution of borax. Birch (*Betula pendula*) and aspen (*Populus tremula*) wood samples in different moisture content were tested. The impregnation time in the centrifugal device were 30 seconds repeated 21 times, and the samples were measured after every 30 seconds. The experimental results were fitted to a nonlinear filtration law, which indicated that the centrifugal wood impregnation was dependent on wood species, wood moisture, rotational speed, and radius. Determination of rotational speed and centrifuge radius for impregnating aspen and birch at varying lengths and humidity under conditions of the nonlinear impregnant filtration law can be done using the example charts that were developed and presented in this study.

Key words: Wood impregnation; Centrifugal force impregnation; Aspen; Birch

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INTRODUCTION

Wood impregnation of soft-leaved species by different chemical compounds is widely used in industry as a way to improve its operational characteristics. Various methods for wood impregnation are known. Of particular interest is a method of centrifugal force field impregnation. This is caused by the two following facts studied before (Patyakin *et al.* 1990): the high filling with impregnant liquid percentage and the almost uniform impregnant liquid distribution within processed wood samples. The high speed of the impregnation during centrifugation is caused by the strong gradient of the impregnant's hydraulic pressure, which takes place because of the centrifugal acceleration distribution. The longer the distance from the sample's cross section to the rotation center, the higher the centrifugal acceleration at this section is; distribution of the hydraulic pressure along the sample is quadratic (Grigorev *et al.* 2013) . Thus, the pressure's gradient "pushes" the liquid into the inner space of the sample alongside the fibers from the side, which is immersed into the impregnant, toward the rotation center. The second side of the sample is normally set free of the liquid in order to avoid counter-pressure of the impregnant effect, which would have taken place in the case of two immersed sides of the sample. In the studied process, the liquid impregnates the sample from the immersed side mainly along the fibers. Previously it has been demonstrated that permeability of wood samples from the sides that are parallel to the fiber direction is low compared to the sample permeability from the sides that are perpendicular to the fiber direction (Patyakin *et al.* 1990). This is why the impregnation speed from the sides parallel to the fibers is also low (unlike autoclave or diffusive impregnation processes). The essence of this technique is expounded in this article.

A considerable amount of scientific work has been devoted to the study and improvement of the centrifugal wood impregnation process. Technical solutions for balancing centrifuge platforms, allowing impregnation of long (up to several meters) wood samples, for example, (Birman *et al.* 2010; Grigoriev *et al.* 2013a), have already been developed. However, further improvement of the centrifugal wood impregnation process requires additional research. Many of the current impregnation wood models dealing with centrifugal forces describe the kinetics of the centrifugal impregnation from the perspective of the linear Darcy filtration law, which means that the impregnant pressure is considered to be directly proportional to the impregnation front speed. In practice, however, this is not true for all parameters of the impregnation process and not for all properties of impregnants (Patyakin *et al.* 1990). Thus this requires more experimental evaluation.

Initially there have been some established models, based on linear Darcy's filtration law, for description of the kinetics of centrifugal impregnation (Patyakin *et al*. 1990). It also has been mentioned that the counter-pressure of the trapped air in some cases breaks the linear dependence between the impregnant hydraulic pressure and the impregnation front speed (Patyakin *et al*. 1990). The cited authors suggested using non-linear dependences between the hydraulic pressure and the speed of impregnation, *e.g.* as done by Aravin and Numerov 1953, who found general dependences for non-deformable media permeability for gases and liquids. This theoretical suggestion is based on the results of Chudinov (1984). But neither exact solution nor numeric for practical calculations have been provided.

A discussion of a theoretical model for centrifugal impregnation of wood is provided by Grigoriev *et al.* (2013). The model is based on the dependence, which discounts the rate regularity deviation between liquid filtration speed of wood sample and the Darcy ratio result (hereinafter – for non-linear filtering laws), for which the impregnation front speed is given by Eq. 1,

$$v = f\left(K, n\right)\left[-\frac{dH\left(z\right)}{dz}\right]^{n} \tag{1}$$

where z is the position of impregnation front measured from the sample butt dipped in the impregnant; $H(z)$ is the impregnant pressure; n is a parameter in the nonlinear filtration law, the value of which is determined by the impregnated wood species as well as by other centrifugal impregnation process parameters; K is the linear coefficient of filtration, which is dependent on the wood species; and $f(K, n)$ is a function of K and n filtration characteristics.

It can be seen from Eq. 1 that with changes in the $f(K,n)$ function and n, the speed v is also changed in value, even when the value of $H(z)$ remains constant. Thus, in this model, the empirical K and n parameters are involved to describe permeability of the samples being impregnated caused by the internal flow resistance or the forces caused by the liquid and the wood interaction.

However, the application of the proposed model in practice requires additional experimental data. For practical use of the model Eq. 1, it is necessary to study the K and *n* parameters numeric regarding the parameters of wood samples and the centrifugal impregnation process.

The major aim of this research is the derivation of experimental coefficient values from the nonlinear relation of the centrifugal wood impregnation front speed.

EXPERIMENTAL

This research was conducted by the Department of Technology of Timber Enterprises in their laboratory at St. Petersburg State Forest Technical University in Russia. The controlled independent variable were: the wood species used for the experiment aspen (*Populus tremula*) and birch (*Betula pendula*), the centrifuge platform radius *s* (0.5 m), the geometric parameters of the experimental samples (dimensions of 0.045 x 0.045 m, length 0.33 m), the temperature of the impregnants used in the experiment (20 °C), and the impregnation liquid level *h* in the metal cylinder reservoir with the experimental samples (0.3 m). The impregnants used for this research were: 10% aqueous solution of potassium chloride (density ρ = 1063 kg/m^3) and 2% aqueous solution of borax (density ρ = 1024 kg/m^3). Both liquids were controlled with an areometer.

The schematics of the experimental centrifuge used for this research are shown in Figs. 1 and 2.

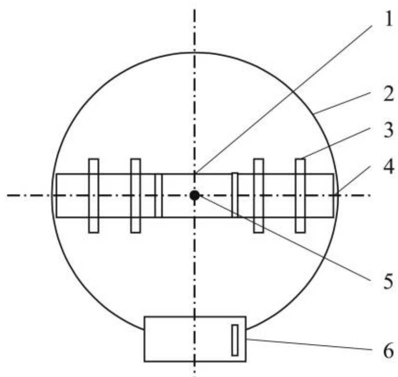

Fig. 1. Scheme of the experimental installation: 1 - carousel; 2 - installation body; 3 - clamps; 4 - impregnating bath, 5 - rotation axis; 6 - control desk

The moisture content of the wood samples *W* ranged from 10 to 70% (10%, 40%, and 70%). The rotary speed of the centrifuge platform *N* ranged from 450 to 750 rev/min (450 rev/min, 600 rev/min, and 750 rev/min), while the total impregnation time was 630 seconds. Thus, the matrix of experimental studies included 9 independent combinations of the moisture content and the rotary speed, which is enough to obtain two-parametrical nonlinear models, because the number of sets, 9, exceeds the number of estimated parameters, which may be 8 at maximum.

After mass and moisture of a sample were determined, an experimental sample was placed in the impregnating bath, fixing it in using a corset (Fig. 1). The impregnating bath was filled with impregnant to the level of 300 mm (impregnating liquid level *h* was set lower than that that of the wood sample in order to provide sample's impregnation from only immersed side. Otherwise counter-pressure of the impregnant may occur) and hermetically closed with a lid (which is not shown in Fig. 2).

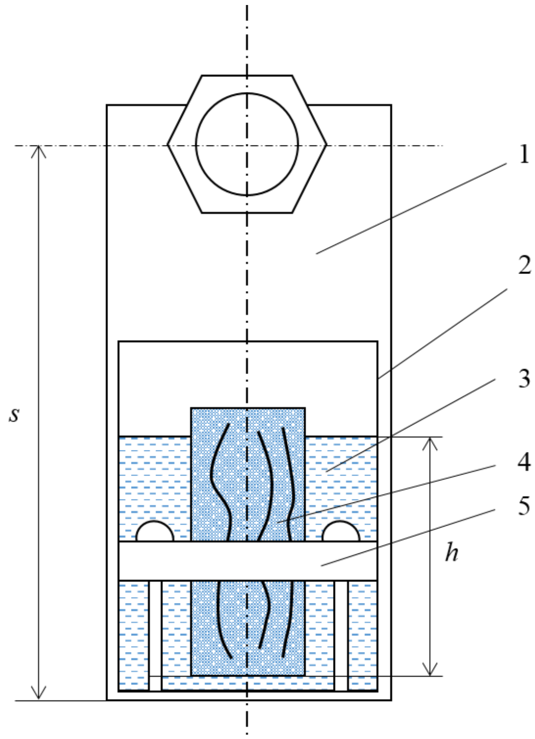

Fig. 2. Scheme of sample placing in bath: 1 - carousel; 2 - impregnating bath; 3 - impregnant; 4 - wood sample; 5 – clamps

The wood sample was fixed on the centrifuge platform with clamps (Fig. 1). The gear was then turned on and, after reaching the steady rotary speed (average of 4 to 5 seconds) the sample was impregnated for 30 seconds (rotation in the horizontal plane). Then it was removed from the bath and weighed; after weighing, the sample again was placed in the bath and impregnated for an additional 30 seconds at the steady rotary speed. The impregnation and weighing of the samples were performed before the total impregnation time in steady rotary speed reached 630 seconds, which means a total of 21 impregnations and weighting cycles. This procedure was repeated five times.

Using the obtained average data on the weight of impregnate that was absorbed by the sample Δm, and using the least-squares statistical method, the regression equation was formulated as follows (this two-parametric dependence can be obtained from 21 measurements, because the number of measurements is higher than the number of unknown parameters),

$$\Delta m = A_0 t^{A_1} \tag{2}$$

where A_0 and A_1 are the coefficients of the regression equation.

Next, using the A_1 average values from Eq. 2 and employing the dependence model developed by Grigoriev *et al.* [2013], the parameter value of filtration law *n* was determined by using the following formula,

$$n = 0.116 + \left[-2.39\left(\frac{h}{s}\right)^2 + 0.021\left(\frac{h}{s}\right) + 12 \right]\left[-0.00232A_1^2 + 0.0665A_1 - 0.072 \right] \tag{3}$$

where n is the parameter of nonlinear filtration law, h is the impregnation liquid level, s is centrifuge platform radius, and A_1 is a coefficient mentioned above.

After calculating the parameter n from equation (3) and combining them with the initials (W and N), the regression equations were determined as follows,

aspen, 10% aqueous solution potassium chloride:	$n = 1.539P^{0.0619}W^{-0.0958}$	(4)
aspen, 2% aqueous solution borax	$n = 1.426P^{0.0811}W^{-0.1022}$	(5)
birch, 10% aqueous solution potassium chloride:	$n = 1.567P^{0.0625}W^{-0.0935}$	(6)
birch, 2% aqueous solution borax	$n = 1.452P^{0.078}W^{-0.1002}$	(7)

where

$$P = 10^{-5} \cdot \left(\frac{\pi N}{30}\right)^2 \rho \cdot h \cdot (2s - h)$$

is the impregnant hydraulic pressure at the level of the sample's immersed end and W is the moisture content of the sample.

Then the function $f(K, n)$ was defined in the expression for centrifugal impregnation front speed (Eq. 1). Depending on the impregnation front position a_τ, the formula for determining impregnation time τ is of the form,

$$\tau = \int_0^{a_\tau} \frac{1}{f(K,n)\left[\frac{1}{2a}\rho\omega^2(h-a)(2s-a-h)\right]^n} da \tag{8}$$

where ω is the angular speed of centrifuge platform.

Then with known parameter value n, the numerical value of the function $f(K, n)$ can be determined as:

$$f(K,n) = \frac{\int_0^{a_\tau} \frac{1}{\left[\frac{1}{2a}\rho\omega^2(h-a)(2s-a-h)\right]^n} da}{\tau} \tag{9}$$

Calculations carried out with Eq. 9 employ the parameter value n obtained from the regression relationship (3). The values s and h in the calculations are constant (0.5 and 0.3 m, respectively), and the impregnant density and the angular speed of the centrifuge platform during the experiment were considered in processing the results.

Determining the value a_τ is based on the assumption of equitability of the impregnant inside the wood sample. The results which were made earlier in Grigoriev *et al*. (2013), established the degree of fill Q in free space inside wood after centrifugal impregnation (77% and 72% for aspen and birch, respectively), then:

$$a_x = \frac{QC\rho}{F\rho}\Delta m \tag{10}$$

In this expression C is the sample porosity at a given humidity, ρ is the sample density, ρ is the wood density, and F is the sample cross-sectional area.

Calculations using Eq. 9 were carried out for all measured values of mass increase in each experiment, and then the arithmetic mean value was determined after the numerical integration of (9) was figured for each experiment for each wood species and impregnant. The regression relationship was then deduced as,

$$f(K,n) = Kn^{C_0} \tag{11}$$

where K is a linear coefficient of impregnant filtration law and C_0 is a constant numerical coefficient.

After processing the data on the mass of absorbed impregnant, the parameters of the nonlinear impregnant filtration law were determined following the regression equations that were developed and are presented below:

aspen, 10% aqueous solution potassium chloride
$$f(K,n) = K \cdot n^{-15.2}, K = 3.177 \cdot 10^{-10} \tag{12}$$

aspen, 2% aqueous solution borax
$$f(K,n) = K \cdot n^{-13.93}, K = 2.831 \cdot 10^{-10} \tag{13}$$

birch, 10% aqueous solution potassium chloride
$$f(K,n) = K \cdot n^{-15.56}, K = 2.934 \cdot 10^{-10} \tag{14}$$

birch, 2% aqueous solution borax
$$f(K,n) = K \cdot n^{-14.35}, K = 2.566 \cdot 10^{-10} \tag{15}$$

RESULTS AND DISCUSSION

Table 1 presents a summary of the results of data from the repeated experiments. Statistical results from the different equation are presented, indicating the adequacy of the formulated models by their coefficients of determination (Table 1).

Table 1. Summary Results from Regression Equations (4) to (7), and (12) to (15)

Equation number	Cochran's test, G_p	R^2	Fisher's test, F_{calc}	Equation number	R^2
(4)	0.1755	0.91	1.56	(12)	0.98
(5)	0.1401	0.92	1.39	(13)	0.99
(6)	0.1539	0.90	1.73	(14)	0.99
(7)	0.1660	0.91	1.54	(15)	0.98

Table 1 shows that the statistical results on the experiment adequacy and coefficient of determination values were acceptable. These allow taking the derived dependence as impregnation time for the aspen and birch wood with the liquids or impregnants used. After substituting the applicable expressions (4) to (7), (12) to (15) into the equation for the infiltration time (8), and assuming the hypotheses of the nonlinear impregnant filtration law, the following relationships were derived for aspen and birch,

aspen, 10% aqueous solution potassium chloride
$$\tau=\int_0^{a_i} 2.238\cdot 10^8\,\frac{Z_1^{15.2}}{X^{0.840Z_1}}\,da,\ Z_1=\frac{Y^{0.0619}}{W^{0.0958}} \tag{16}$$

aspen, 2% aqueous solution borax
$$\tau=\int_0^{a_i} 7.88\cdot 10^6\,\frac{Z_2^{13.93}}{X^{0.645Z_2}}\,da,\ Z_2=\frac{Y^{0.0811}}{W^{0.1022}} \tag{17}$$

birch, 10% aqueous solution potassium chloride
$$\tau=\int_0^{a_i} 2.74\cdot 10^8\,\frac{Z_4^{15.56}}{X^{0.85Z_4}}\,da,\ Z_4=\frac{Y^{0.0625}}{W^{0.0935}} \tag{18}$$

birch, 2% aqueous solution borax
$$\tau=\int_0^{a_i} 1.45\cdot 10^7\,\frac{Z_5^{14.35}}{X^{0.677Z_5}}\,da,\ Z_5=\frac{Y^{0.078}}{W^{0.1002}} \tag{19}$$

where $X=\frac{5.83N^2(h-a)(2s-a-h)}{a}$ $Y=N^2h(2s-h)$.

Here it should be noted that the parameters K and n (the last one is determined not only by the species and the liquid type, but also by the hydraulic pressure, see Eq. 4-7, which helps to take into account the driving force acting on the fluid in the wood matrix accurately) are obtained for the exact pairs "wood species + liquid" and, thus, we suppose them to be a sort of "integral" pairs' properties in the centrifugal impregnation process.

We consider the determined parameters K and n to be a sort of "integral" of empirical characteristics for the samples permeability, because their numeric value affects the centrifugal impregnation front speed and, thus, the impregnation time, which is seen in Eq. 1. The fact that the results obtained were replicable and consistent in all the experiments, as indicated by Cochran's test (Table 1), gives support for this conclusion.

Since the liquids used for impregnation were 10% aqueous solution of potassium chloride and 2% aqueous solution of borax (non-replete solutions), we have neglected crystallization, dissolution, or probable dispersion of the actives in the wood matrix and considered the actives distribution to be uniform in the samples' inner space. Uniform distribution of impregnants has also been reported (Patyakin *et al*. 1990). Thus, for further research it seems necessary to study the actives distribution when impregnating wood samples with saturated solutions.

Using the obtained dependency and diagrams that were developed from the experiment data, rational parameters definition of work and equipment design value for impregnation does not meet with difficulties.

Figure 3 shows charts that can be used to calculate impregnation time for wood samples using the formulas derived earlier and considering alternative centrifuge rotary speeds and platform radius. In these charts, the example moisture was 10%, length of wood sample was 2 m, and the impregnant level in the cylinder was 2 m. However, the centrifugal impregnation topic is not a widely developed technique and only few scientific results are available for comparison.

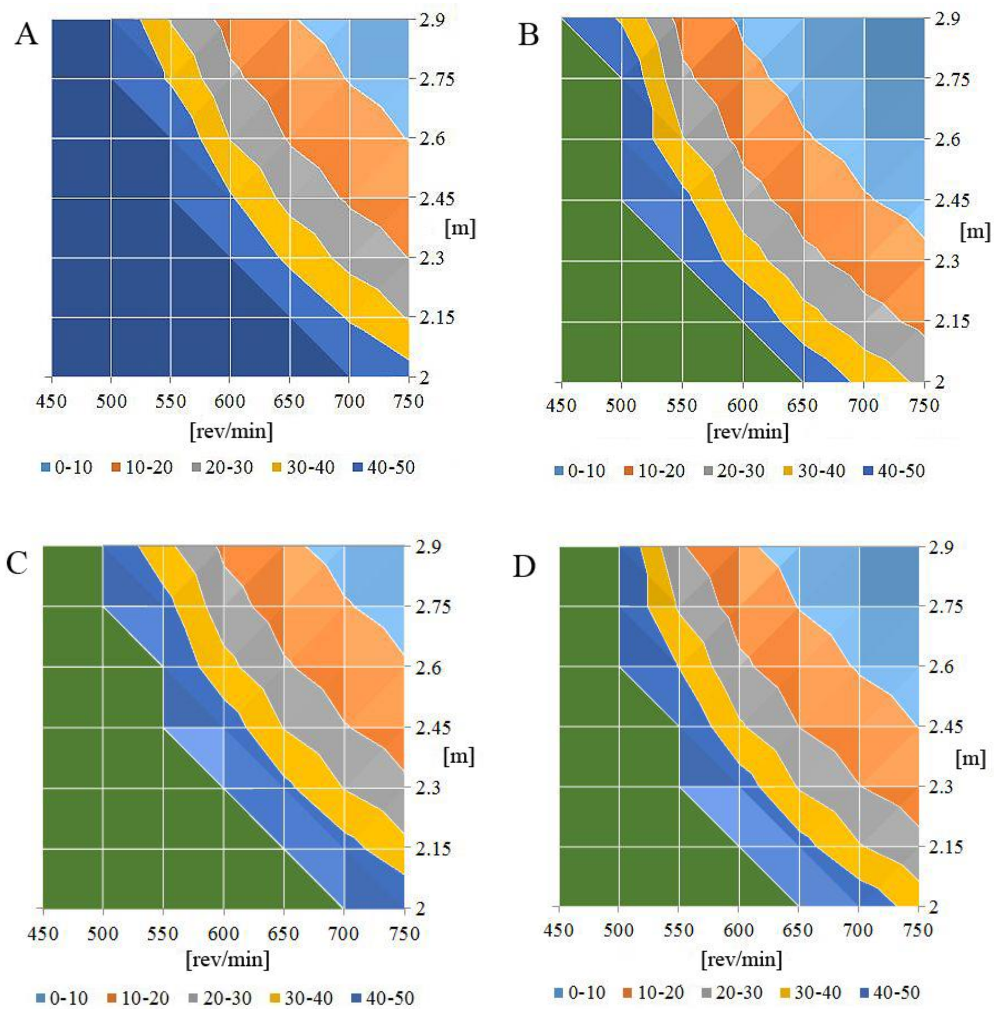

Fig. 3. Impregnation time of aspen and birch using two types of impregnants (A - aspen, potassium chloride B - aspen, borax , C - birch, potassium chloride , D - birch, borax)

For example, if you need to impregnate a 2 m length aspen wood unit with 95% borax in less than 20 min, the diagram in Fig. 3 *B* then determines that at a speed of 550 rev/min, the required radius of the platform is 2.8 m. If with the same platform radius, one were to increase the speed up to 650 rev/min, then the desired impregnation condition will be realized in 10 min. If one increases the speed up to 750 rev/min, then the centrifuge platform radius of 2.25 m is enough (impregnation will takes 20 min); similarly *ceteris paribus*, increasing the platform radius to 2.55 m will reduce impregnation time to 10 min.

CONCLUSIONS

1. It is experimentally established that the parameters of nonlinear filtration law with centrifugal wood impregnation are dependent on wood species, wood moisture, and impregnant type; dependences are expressed by the formulas (4) through (7) and (12) through (15).
2. Value determination of the nonlinear parameter for the impregnant filtration law was replicable and consistent in all the experiments, as indicated by Cochran's test (Table

1). This test confirms the adequacy of using previously formulated (Grigoriev *et al.* 2013b) methodology of defining the parameters of the nonlinear impregnant filtration law by means of an experimental curve of sample saturation.

3. Determination of rational speed and centrifuge radius for impregnating aspen (*Populus tremula*) and birch (*Betula pendula*) at varying lengths and humidity under conditions of the nonlinear impregnant filtration law can be done using the example charts that were developed and presented in Fig. 3.
4. The experimental data derived from this research can be used in practice for determining the time mode for aspen and birch using the centrifugal impregnation method.

ACKNOWLEDGEMENT

This work has been partially supported by the Ministry of Education and Science of Russian Federation (project № 2014/181 1576 “Scientific bases of sustainable forest harvesting and woodworking processes” and the Environment conscious energy efficient building TAMOP-4.2.2.A–11/1/KONV-2012-0068 project sponsored by the EU and European Social Foundation.

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Article submitted: August 1, 2014; Peer review completed: September 2, 2104; Revised version received: September 9, 2014; Second revision received and accepted: September 25, 2014; Published: October 6, 2014.