

STATISTICAL SIMULATION OF WOOD DENSITY CHANGES DUE TO THERMAL TREATMENT IN CARBON DIOXIDE ATMOSPHERE

The aim of the present investigation was to detect changes, and make statistical simulation, of oak (*Quercus robur*) density, due to the influence of thermal modification parameters. The reduction of the density was recorded by High-Frequency Densitometry. To achieve a comparison of the initial density and the resulting density of thermal treatment, the examination was carried out on the same sample. As a result, an adequate regression model $\Delta\rho_0 = f(T, v, \tau)$ was obtained for dry wood density changes $\Delta\rho_0$ of oak with respect to temperature T , heating rate v , and hold-up time at treatment temperature τ . The obtained results made it possible to present visually plots of nonlinear influence of modification parameters (T, v, τ) on oak wood density changes during heat treatment in carbon dioxide atmosphere.

Key words: thermal modification, wood density, high-frequently densitometry, multiply regression, treatment temperature, hold-up time, heating rate, carbon dioxide.

Introduction. The thermal treatment of wood in CO_2 is an environmentally friendly method for wood protection. The progressive thermal degradation at high temperature leads to the reduction of hydroxyl groups. For this reason hygroscopicity, dimensional stability and durability are improved [2]. On the other hand, depending on the treatment parameters, such as maximum treatment temperature, heating rate, holding time at the maximum temperature and the medium in the atmosphere, the wood becomes more brittle, and its mechanical strength and technological properties are impaired [4]. These factors, as mentioned above, influence on the changes in dry wood density. Beside strength, hardness, calorific value and stability, a range of other physical, mechanical and technological properties of wood are determined by the density of the material [5]. In this view, it seems necessary to conduct a detailed analysis of wood density changes, write the dynamics and make statistical simulation of this process depending on the process parameters.

Materials and methods. The investigations were carried out on wood samples of one tree of the oak species (*Quercus robur*). Apience of sawn timber was planed and sawn into 6 specimens (20 x 20 x approx. 400 mm tangential x radial x longitudinal) in direction parallel to the grain. Then the middle part of each specimens were cut into 10 specimens with a dimension of 20 x 20 x 20 mm (tangential x radial x longitudinal) according to DIN 52180. The overall amount of 60 samples was placed in their original position in the board. In an upstream step of preparation all these “twin samples” were arranged in a special pattern and calibrated to achieve a high quality cross-section surface, for the measurement of density by HFD- Method. At the beginning, the wood samples were conditioned to a 7-8% equilibrium moisture content at the $20\pm 2^\circ\text{C}$ and 65% relative humidity according to ISO 554. On the one hand, equal conditions for the investigations have to be achieved, as well, as on the other hand, the moisture content of the material should be smaller than 10% before heat treatment to avoid a lot of splitting and colour differences, which may result.

In the next step, the HFD- Method was applied to measure the density of the identical specimen as mentioned above. The method is based on the propagation of continuous electromagnetic waves in a high-frequency (HF) transmitter-receiver link of an ex-

tremely small electrode system in direct contact with the wood surface that is to be investigated. The experimental setup is shown in Fig. 1. Subsequently, the specimen were treated in a temperature controlled heating unit of 5, 10 and 15 degrees per hour at three temperatures (180, 200 and 220°C) with duration of 0-2 hours at each temperature level. Each of six individual specimens were taken out when reaching and when holding the various temperature levels for 0-2 hours and replaced with dummy specimen.

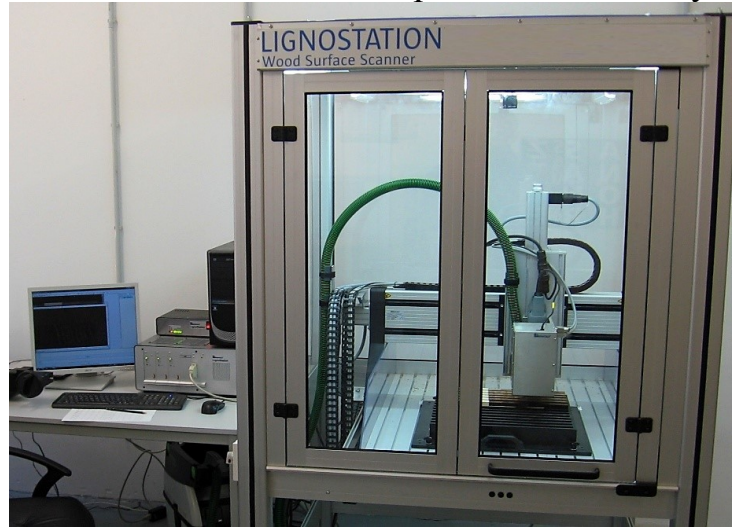


Fig. 1. Equipment for wood density measuring by HFD- method

Thus, for each temperature, the samples were removed and then remeasured with HFD-Method. The process conditions were applied and run under atmospheric pressure and in the presence of carbon dioxide. The choice of the wood specimens and materials, advanced description of the experimental setup for thermal modification in CO₂ and measurement results are given in [6].

Results and discussions. After statistical analysis of the results, blunders discarding and checking of normality distribution of the results, the choice of empirical model was carried out. By virtue of the fact that results obtained by using multiple linear regression is unsatisfying (derivable model is not adequate) the relationships among data are nonlinear and could be set out using multiple regression.

Since the central composite plans, including the second order rotatable plan, require coding factors on five levels (zero, upper and bottom levels, positive and negative values of star ($\pm\alpha$)) their use is not possible in our case, because the planning of the experiment did not provide α . Besides, using this plan often, we obtain the significant curvature of response surface for considered dependencies. It does not allow for adequate description of regression dependence, and requires division of the research region into two experiment, as is shown in [7]. An alternative, in this case, is using multiple regression. To obtain adequate regression model that describes experimental data, the multiple regression block of software package STATISTICA 10 was used [8-19]. Using this block with setting the procedure for evaluation of regression coefficients (step-by-step with inclusion) the three-order regression dependence emerged:

$$\begin{aligned} \Delta\rho_0 = & b_0 + b_1T + b_2\tau + b_3\nu + b_{12}T\tau + b_{13}T\nu + b_{23}\tau\nu + b_{112}T^2\tau + \\ & + b_{113}T^2\nu + b_{221}\tau^2T + b_{223}\tau^2\nu + b_{331}\nu^2T + b_{332}\nu^2\tau + b_{11}T^2 +, \quad (1) \\ & + b_{22}\tau^2 + b_{33}\nu^2 + b_{111}T^3 + b_{222}\tau^3 + b_{333}\nu^3 \end{aligned}$$

Values for significant coefficients of model (1) are presented in Table 1.

Table 1. Significant coefficients of multiple regression

Regression coefficients	Values	Student's regression t-criterion	
		calculated values	table values t _(0,05;5)
b ₀	-1605,178	58,81	1,98
b ₁	15,088	55,04	1,98
b ₁₁	-0,034	48,94	1,98
b ₁₁₂	-0,005	8,56	1,98
b ₃₃₁	-0,002	14,90	1,98
b ₂₂	21,476	10,08	1,98
b ₂	-255,716	11,87	1,98
b ₂₂₁	-0,127	11,93	1,98
b ₁₂	2,273	10,65	1,98
b ₃₃₃	0,010	13,69	1,98
b ₁₃	0,015	12,27	1,98
b ₃₃₂	-0,003	2,261	1,98

Coefficient of multiple determination (R^2) for equation (1) with coefficients from Table 1 equals $R^2 = 0,99990491$. Calculated and tabulated means for significant Student's t-criterion shown in tab. 1 also. Adequacy for equation of multiple regression in general is evaluated by Fisher's criterion; for our occurrence $F=14338,95$. As observed value of F-criterion exceeds the one from table, and significance level $p=0.000001$ the regression simulation (1) is adequate and highly significant. As previously mentioned, multiple regression assumes relationships between the variables in the equation, and a normal distribution of residuals (*observed minus predicted* values).

If these assumptions are violated, our final conclusion may not be accurate.

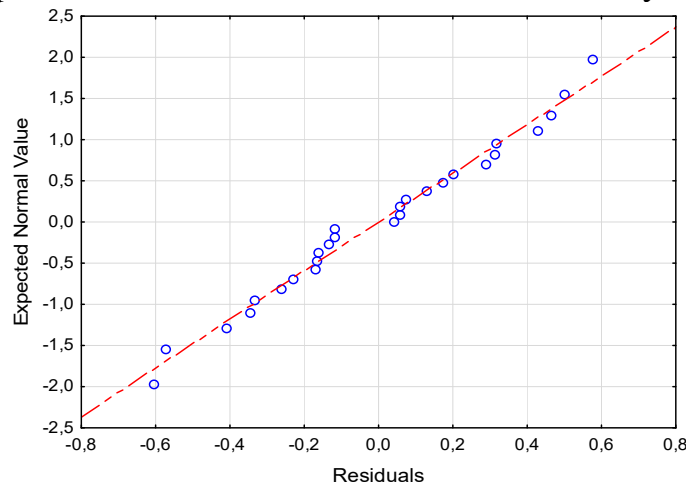


Fig. 2. Normal probability plot of residuals

The normal probability plot of residuals (Fig.2.) will give us an indication of whether gross violations of the assumptions have occurred. As can be seen from the graph, deviation of residuals points from line is inappreciable (all values fall very closely onto a straight line in the plot); it indicates normality of their distribution. Furthermore, this arrangement of residuals relatively the line attest sufficient compliance of the experimental results and the results obtained using the regression equation (2).

Adequate regression model that describes experimental data is as follows:

$$\begin{aligned}
 \Delta\rho_0 = & -1605,178 + 15,088 \cdot T - 255,716 \cdot \tau + 2,273 \cdot T\tau + \\
 & + 0,015 \cdot T\nu - 0,005 \cdot T^2\tau - 0,127\tau^2T - 0,002 \cdot \nu^2T - \\
 & - 0,003 \cdot \nu^2\tau - 0,034 \cdot T^2 + 21,476 \cdot \tau^2 + 0,010\nu^3
 \end{aligned} \tag{2}$$

To check the stability of regression coefficients residue vs. deleted residuals graph was built (Fig.3.). In view of insignificant differences in the values of received and deleted residues, within the confidence interval of 95%, we can state that the coefficients of regression are stable and do not depend on individual experiments.

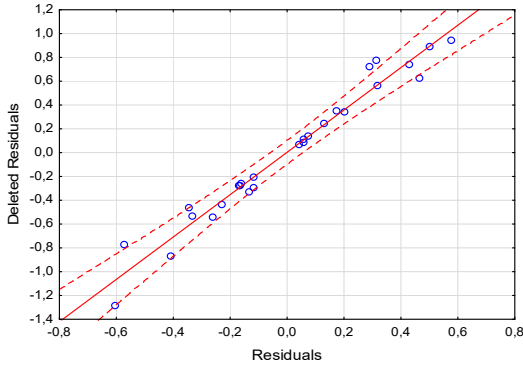


Fig. 3. Residuals vs. deleted residuals (confidence interval 95%)

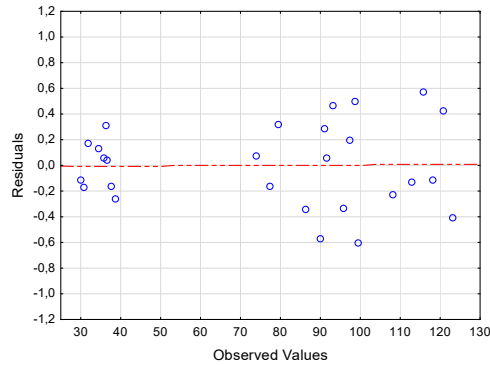


Fig. 4. Residuals and observed values

The relationship between observed values and residuals is shown on Fig. 4. The distribution of residues do not form a homogeneous region centered around the line. It indicates the existence of nonlinear relationship between the predictors and the response function, as evidenced by the obtained regression equation (2). Moreover, Fig. 4. detects absence of outliers or groups of observations that are over- or under-predicted, which also confirm the value of a "legitimate" correlation. Graphs for dependencies $\Delta\rho_0 = f(T, \nu, \tau)$ of response surface defined by equation (2), in case when one of three variable factors is stabilized, are shown in Fig. 5-6. The graphic dependencies analysis shows that the most significant effect on the density changes of oak has a temperature factor and hold-up time. As shown in Fig. 6, increasing of modification temperature from 180 to 200°C, leads to an increase in changes, at constant hold-up time and fixed heating rate, averaging from 5.4 to 17.6%. Effect of hold-up time at given temperature modification, albeit to a lesser degree, also leads to decrease in density (Fig. 5).

However, the influence of this factor is not so significant during prolonged hold time. On the other hand, increasing of heating rate from 5 to 15 °C/h to the desired temperature modification, slows down the rate of density decrease, although not significantly compared with the influence of two other factors.

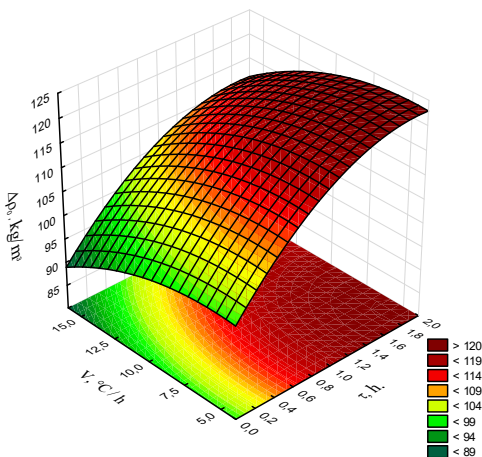


Fig. 5. Dependence of oak wood density changes from variations in heating rate, and hold-up time under the fixed temperature (180 °C) in CO₂ atmosphere

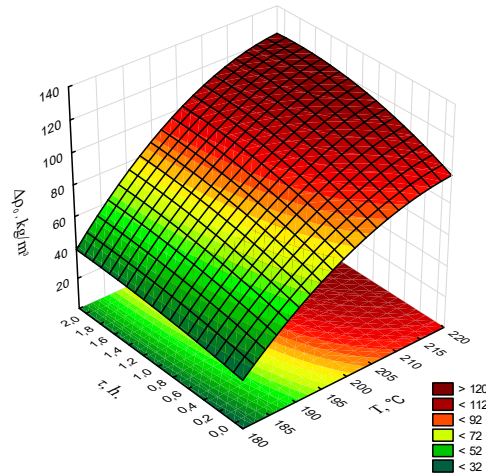


Fig. 6. Dependence of oak wood density changes from variations in temperature, and hold-up time under the fixed heating rate (5 °C/h.) in CO₂ atmosphere

Conclusions. Wood density changes in oak, due to thermal treatment in CO₂ atmosphere, was investigated. Statistical simulation of experimental data using multiply regression module was made. Consequently, has been obtained an adequate regression model $\Delta\rho_0 = f(T, v, \tau)$. Predicted results made it possible to show visual dependences of nonlinear influence of modification parameters (treatment temperature, heating rate, hold-up time) on oak wood density changes during heat modification in carbon dioxide.

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Статистичне моделювання впливу режимних параметрів процесу термічного модифікування деревини в середовищі вуглекислого газу на динаміку зміни її щільності

Досліджено зміну щільності деревини дуба в процесі термічного модифікування методом високочастотної денситометрії. З використання методу множинної регресії отримано математичні залежності зміни щільності деревини при її термічному обробленні в середовищі CO₂ та графічні залежності поверхні відклику рівнянь регресії.

Ключові слова: щільність деревини, термічне модифікування, статистична обробка, високочастотна денситометрія, множинна регресія.

UDC 674.8

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IDENTIFYING THE WORKING CONDITIONS OF FLAT DIE PELLET PRESS HOLES WITH PREDETERMINED GEOMETRICAL PARAMETERS

The method for determining the working capacity of flat die pellet press holes with predetermined geometrical parameters has been proposed.

Keywords: pellet press, hardwood pellet, flat die, hole geometry.

Introduction. The characteristics of a flat die in pellet press can determine its performance through the blank and effective thickness, the reliefs and the hole pattern [1]. The most important physical characteristics of a flat die are the blank thickness and the effective thickness. The blank thickness determines the overall strength of the flat die. The thicker the blank, the more it resists deflection caused by the rollers. Blank thickness should be increased instead of ligament thickness, especially in cases of repeated circumferential breakage.

Effective thickness is the length of the pellet chamber that will perform the pelletizing. Effective thickness governs the amount of work the flat die will perform on a material, increasing pellet quality. Changes in material necessitate changes in effective thickness due to changes in the materials coefficient of friction [1-7].

Reliefs were developed so that increased blank thickness could be used with applications that needed thinner effective thicknesses. Currently, there are two major types of reliefs: straight hole and tapered. Straight holes can be oversized, while tapered holes can be at different angles. Tapered reliefs are extended by adding a straight relief to it. These two reliefs' can be drilled in three methods: variable, non-variable, and special variable.

Standard is the normal pattern and the heavy one is used in high pressure, for hardwood waste. These patterns, when applied to various wood materials, will give different results. Every opportunity should be taken to get maximum hole count in a flat die as it increases the throughput and die life.

Research objectives – determination of movement condition of hardwood wastes, during compaction in a die hole with specific geometry.

For flat die standard hole to formulate efficiency condition by changing its basic geometrical parameters.