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Mathematical Prediction of Biofuel´s Mechanical Properties during Densification Process

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Abstract: *The main aim of this paper is to present the results of experimental research which was done on our department in laboratory conditions. Aim of the research study was to determine the effect and interaction of technological variables and material parameters during densification using mathematical modelling in order to determine the final biofuels quality and mathematical model. Also, the design of experiment (DOE), evaluation methodology for this experimental plan and research findings are presented in this paper. Experimental research dealt with the densification of selected Slovakian woods. The main goal of this experimental research was to obtain such results which can be used for mathematical models design. This statistical mathematical models based on the proposed experiments can be a very useful tool for final biofuels prediction before densification and thus the densification process can be improved and adjusted according to properties of raw material to be pressed. The useful practical output of this experimental research is designed mathematical model which describe the densification process at various adjusted levels of influencing variables. These models can be implemented into the densification machines control system and thus the final quality of biofuels can be adjusted and controlled during densification.*

Keywords: *Solid biofuel, Pressing temperature, Moisture content, Particle size, Pressing pressure, Mathematical model, DOE.*

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I. INTRODUCTION

One of the recovery possibilities for waste biomass raw materials is the production of solid biofuels. Densification as a technology for solid biofuels production is very interesting and itself complicated. During the densification process, many various variables influence this process and thus the final solid biofuel quality. Using a variety combination of influencing variables can improve the final quality of solid biofuels.

Raw biomass material variables influence, especially (a type of raw material, particle size, moisture content, compression pressure and pressing temperature) can be recognized during the production of solid biofuels. Their effect can be seen through the quality indicators; especially mentioned variables significantly influence the mechanical quality indicators of solid biofuels. On the base of our experiences and analyses, they can be divided into the following three groups - raw material parameters, technological parameters and structural parameters of pressing machine (Križan, 2015). Requirements from the biofuels production are showing that another important fact is considering the possibility for a variable change of pressed material (Križan et al., 2015; Holm et al., 2006).

Solid biofuels quality is given by EU standards (EN ISO, 2014) and is evaluated by mechanical and chemical-thermic indicators of quality (Nosek et al., 2016; Baláš et al., 2012). The input raw material needs to be treated for gaining the optimal particle size and the optimal moisture content level and we have to provide optimal technological parameters throughout the process of densification (Križan, 2015; Mani et al., 2006). Each type of raw material requires an independent approach. Each small change in the properties of the raw material can influence the final quality of the solid biofuels (Križan et al., 2015; Kaliyan et al., 2009) and also the pressing forces distribution along with the pressing chamber of densification machine. Different raw material properties cause different conditions during the densification process (Nielsen et al., 2009). Therefore is very important to determine the impact of each variable and their interaction and to quantify the effect of the variable on densification itself.

Knowledge about densification process in the base is a necessary condition for the development and engineering of densification machines with effective production of solid biofuels. The general purpose of this paper is to present the methodology for determining the relationship between technological and material variables and final quality of solid biofuels, which can be used for prediction and control of solid biofuels quality during densification. The submitted paper describes the methodology which consists of the design of experiment (DOE), measured data evaluation and processing, the design of a mathematical algorithm to a system for prediction and control of solid biofuels quality. The basic aim and intention are that the gained research findings from determination the relationship between technological and material variables and final quality of solid biofuels should be used for the design of the mathematical model and to build up the simple software for prediction and control the quality of the solid biofuel. Based on this objective it was necessary to design a series of experiment and their evaluation methodology that can allow determining the influencing variables effect. When the correct methodology and methods have used the increase of solid biofuels quality can be ensured. Initially, to evaluate if the biofuels fulfil the requirements established in the European Standards (EN ISO, 2014), physical characterization of each densified briquette was performed. Subsequently, to determine the impact of material and technological factors, all related parameters were measured, and the densification according to the designed experimental plan was executed. Finally, to better understand the effect of the material and technological variables, detailed data processing was done and mutual interactions of monitored variables were characterized.

II. EXPERIMENTAL PROCEDURE

1.1 Aim of experimental research

The main aim of our experimental research is to determine the effect and interaction of raw material variables and densification process variables on the final density of solid biofuels. The best way is to choose such variables which have during the densification process continuous characteristics. In our case, according to the recent published research (Križan et al., 2015; Križan et al., 2018) works and our experience determination of the mutual interaction between biofuels density (ρ), compression pressure (p), pressing temperature (T), particle size (L) and moisture content (w_r) of raw sawdust used for densification were chosen.

The raw material to be pressed had to be characterized, suitable experimental device and equipment had to be used and proper experimental design and evaluation and processing methods had to be used. Each type of raw material have different chemical composition and behaviour during the densification, there requires an independent approach. That means the experimental research had to be repeated with each type of used raw material. This approach allows designing separate mathematical model for each type of used raw material. These statistical mathematical models based on the proposed experiments can be used of the measured data processed and can be a very useful tool for final solid biofuels prediction before densification and thus the densification process can be improved and adjusted according to properties of raw material to be pressed.

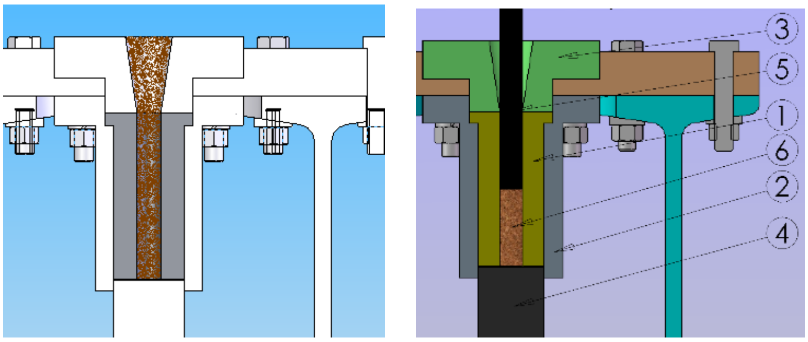
1.2 Properties of raw material

Experimental research in our laboratory conditions was carried out with widely known wooden, which are originating from south-western Slovakia. Softwoods and also hardwoods were chosen for this experiment and suitable raw material in sawdust form was obtained from wood processing company without bark. It was necessary to characterize the basic properties of each used raw material because particle size and moisture content of raw material affect the creation and value of binding forces between raw material particles, and thus the final physical properties of solid biofuel. The moisture content of sawdust before experimental research was measured by Kern MRS 120-3 balance. This measurement was based on heating the material (gravimetric method of moisture content measuring) (EN ISO, 2015) at 105 ± 2°C until a constant weight was achieved. Initially, the particle size distribution was analysed by Retsch Vibrating Sieve Equipment AS 200.

1.3 Experimental device

Experimental research was realized for determination of technological and material variables effect on final biofuels quality during densification. Briquettes quality, as a final output of the densification process, was evaluated by its particle density (Križan et al., 2014; Križan et al., 2011). Briquettes were produced by a vertical hydraulic press (see Figure 1) which was supplemented by the experimental pressing device. This equipment is representing vertical single-pressing densification. The experimental pressing device consists of a base frame, a cylindrical pressing chamber with 20 mm diameter die, a heating device with a temperature sensor for temperature control and the backpressure plug. When the temperature into the chamber was reached the raw material was fed into the chamber and pressing by hydraulic piston was performed. The hydraulic press allowed setting the pressing pressure in the range from 31 MPa to 318 MPa.

Figure1. Cross-view of the experimental pressing device and pressing phases (1-pressing chamber; 2-pressing chamber flange; 3-startup chamber; 4-counter pressure plug; 5-pressing piston; 6-pressed raw material).



1.4 Experiment conditions and experiment design

As inputs variables are necessary to choose those variables where the effect on outputs is expected. This may not be clear at the beginning of the experiment and then is needed to realize a screening experiment. With respect to the complexity of the presented process and the number of considered variables, 2 parameters of the process itself and 2 main material parameters, we have used the second-order nonlinear model. Second-order models are used if the models of first-order do not describe the examined object sufficiently (Wortmann, 2001). Compared to the model of first-order, the experiment with the second-order model is more complex, involve fact on more levels and therefore also a higher number of measurements. Each variable within the second-order experiment must be changed on at least three levels. Currently, there is a high number of second-order proposals based on various criteria and presenting various characteristics. If we consider a system that has 4 parameters, the output variable can be considered as a function with 4 variables $\rho = f(p, T, w_r, L)$. Then we will consider this function in shape:

Equation 1: Mathematical interpretation of second-order model with 4 variables

$$\rho = b_0 + b_1.p + b_2.p^2 + b_3.T + b_4.T^2 + b_5.w_r + b_6.w_r^2 + b_7.L + b_8.L^2$$

where

b_i are regression parameters,

p, T, w_r, L are controllable variables.

The second-order model was chosen because these are used in the models of first-order do not describe the examined object sufficiently (Wortmann, 2001; Ryan, 2011). Compared to the model of first-order, the experiment with the second-order model is more complex, involve fact on more levels and therefore also a higher number of measurements. Each variable within the second-order experiment must be changed on at least three levels (Ryan, 2011; Schmidt et al., 1994). Currently, there is a high number of second-order proposals based on various criteria and presenting various characteristics. Densification process is often dependent on a relatively large number of variables. It is practically impossible to test every combination of variables during process development in order to determine the relevant correlations between the individual variables. DOE uses a minimal number of experiments to provide an empirical process model for the interrelationship between the control and disturbance variables in the process and the resulting product and process characteristics (Schmidt et al., 1994). Fractional factorial designs (screening designs) provide the possibility of significantly reducing the number of experiments. Results from screening designs can also be transferred to a subsequent series of experiments with less investigated factors, known as response surface designs (Schmidt et al., 1994; Duncan, 1986; Wonnacott et al., 1993). Response surface designs are used to determine and then optimize non-linear interrelationships (Wonnacott et al., 1993). In the following Table 1, the chosen levels for each variable (raw material and process parameters) can be seen. Mentioned monitored variables (outputs) according to the technical standards and experimental device possibilities should be chosen. Outputs should represent quality indicators and these variables should have also a function of comparative criterions. For the experimental research particle sizes up to 0.5 mm, up to 1.0 mm, up to 2.0 mm, up to 4.0 mm and more than 4.0 mm was chosen according to our possibilities. Levels of raw material moisture content on the base of our professional practice experiences were chosen. Raw material parameters were achieved by disintegration, drying and separation equipment. Levels of compression pressure and pressing temperature according to the experimental device possibilities were chosen. Experiments were evaluated and selected influencing parameters were optimized to achieve the optimum value for the best quality of the solid biofuel.

In our case, more than 2 levels of variables were chosen, than we can use the Central Composite Design (CCD) (Schmidt, 1994). A second but also a very important reason for choosing of CCD in our case is volition to use surface response method, where 3 levels of variables are a necessary condition for surface response modelling. The surface response method is a tool to investigate the response of a variable to changes in a set of design or explanatory variables and helps to find the optimal method for the response as a measurable output of our interest (Wortmann, 2001; Ryan, 2011). The base of our CCD plan is a two-level full factorial plan which is complemented by central and axial points. Value of number α and a number of central points on the base of other requirements (orthogonally, rotatability, number of variables, etc.), (Schmidt, 1994) related the CCD plan have to be calculated.

A set number of samples for testing will be produced according to the designed experimental plan. The testing and sample's production process should be randomized order to avoid the effects of systematic errors or we can increase the number of replications for a given set of variables. The raw material for the experiment has to be treated. Wood sawdust has to be separate for obtaining the given particle sizes and also have to be prepared for achieving the given moisture content level. In the final, the outputs variables were measured by appropriate measuring equipment for final particle density obtaining. The values of measured parameters can be calculated as ρ_1, ρ_2, ..., ρ_j, where j = 1, 2, ..., r (in our case, r = 7), for experiment 1 - 16. When evaluating, only measured values which characterize the core of the experiment for the central composite design 2^4 = 16 settings were used. From these measured set of values, the selection averages are calculated. According to the experimental plan for each experiment setting at least 7 briquettes with approx. 20 mm diameter was produced. This number was chosen according to requirements of standard mathematical and statistical methods for experiments evaluation (Križan, 2015).

Table1 Input controllable variables of the process (Križan, 2015; Schmidt, 1994).

Levels related to CCD plan	Variables Compression pressure *p* (MPa)	Pressing temperature *T* (°C)	Particle size *L* (mm)	Moisture content w_r (%)
-α	63	55	0 – 0.5	5
-1	95	85	0.5 – 1.0	8
0	127	100	1.0 – 2.0	10
1	159	115	2.0 – 4.0	12
α	191	130	4.0 <	15

Initial information about the effect of a particular variable is obtained mostly with the aid of the multi-variation diagram. Evaluating the divergence of averages in groups makes it possible to use procedures derived from the control chart – average analysis (ANOM) or variance analysis (ANOVA) (Wortmann, 2001). Results of the experiment realized according to designed experimental plan also surface response can be used. The surface response will represent a group of points which forms a continuous surface when each axe in orthogonal view represent variables which influence the process and also the output variable (Wortmann, 2001; Ryan, 2011). Influence of two input variables is related to the third axes, which mainly represents the output variable. To we know to create a surface response, it is necessary to have a "prescription" under which the points in a coordinate system will bring out, in other words, a mathematical model that gives us a three-dimensional function displayed as the surface response will create. In nowadays for the creation of mathematical models are various mathematical - statistics software widely used, which enable comfortable testing of hypothesis, testing of the suitability of obtained data (values) and testing of model quality itself. With mathematical - statistics software can be generated many mathematical models, which described the process with some precision and can be chosen that model which suits us from variables composition and required precision points of view. We can find the optimal mathematical model from described points of view. Briquettes particle density was calculated by means of the ratio between briquettes weight and its volume. The weight, length and diameter of each briquette were measured by a digital calliper and an electronic balance. The volume of briquettes was calculated as the volume of a cylinder with dimensions (length and diameter), according to EN ISO 17829 (EN ISO, 2016). The average density was calculated for each sample briquettes in each experiment setting. These sets of values with using proper mathematical-statistical tools and methods were used for the development of a mathematical algorithm for each raw material.

III. RESULTS AND DISCUSSIONS

In this experimental study sawdust of pine tree (*Pinus sylvestris*), sawdust of oak tree (*Quercus robur*) and sawdust of acacia tree (*Robinia pseudoacacia L.*) was used. Densification of three chosen sawdust was executed gradually in order to achieve correct results. The degree of influence of a variable is defined as the magnitude of response change caused by a change in the factor level. The magnitude of the main effects and their interactions were estimated. Figure 3, Figure 4 and Figure 5 illustrate the effect of differences.

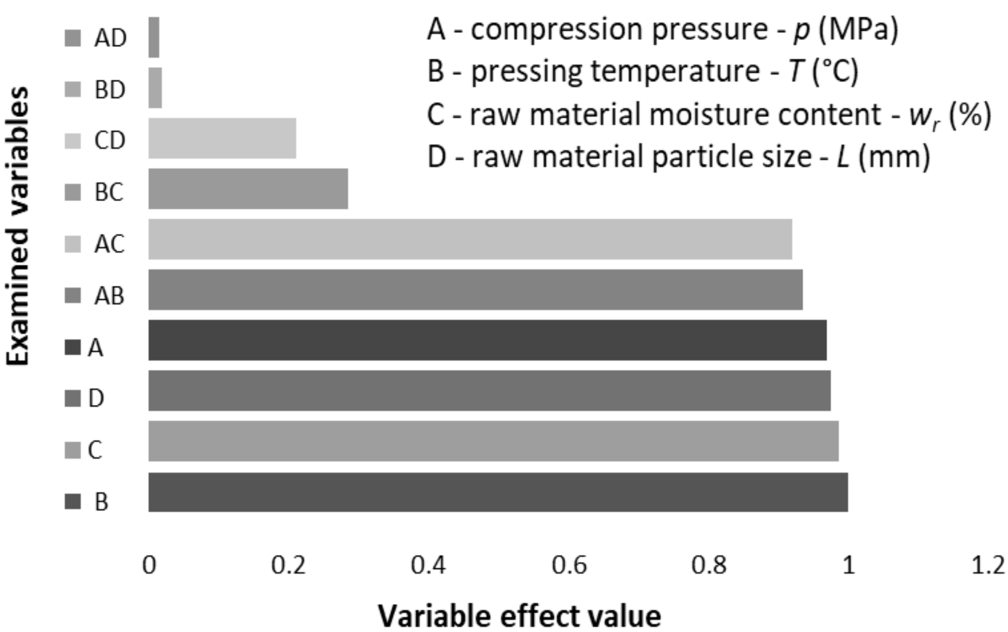
3.1. Densification of acacia tree sawdust

Figure 2 shown the samples from acacia wood sawdust which were produced during experimental research. You can see that different conditions caused the (optically) different final quality of briquettes. For a closer determination, not only of the effect of each variable but also of their mutual interactions, a variable effect method was used. The degree of influence of a variable on an observed variable is known as the variable effect, which is defined as the magnitude of response change caused by a change in the variable level.

Figure2. Samples – briquettes from acacia wood produced during the experiment

According to the design matrix, the magnitude of the main effects and their interaction were estimated. Estimated (calculated) values of each of the effects are shown in Figure 3 and also this figure illustrates the effect differences. We can see that the most influencing variable for this acacia wood is pressing temperature, following by moisture content, particle size and compression pressure with interaction with pressing temperature and moisture content.

Figure3. Diagram of variables and their interaction effect for acacia tree



We were able to design the mathematical model with the help of software SAS and JMP 8 and also we were able to calculate or estimate the regression parameters values (Schmidt, 1994). The final designed forms of mathematical models for three investigated raw materials you can see in following relation 2, where the "ρ" is biofuel particle density. The final form of a mathematical model which describes relations between influencing variables and final particle density during densification of acacia tree sawdust.

Equation 2: Mathematical model for acacia tree sawdust densification

$$\rho = 1.07608 - 0.00115745 * T - 0,027793 * w_r - 0,0147938 * L + 0,000576255 * p$$

This equation 2 is the final mathematical model which describes the behaviour of acacia wood sawdust during densification from the final briquettes density point of view. Have to write that designed mathematical model is valid only for acacia wood sawdust and in the following range of examined variables: pressure 95 – 159 MPa, temperature 85 – 115 °C, moisture content 8 – 12 % and particle size 1- 4 mm.

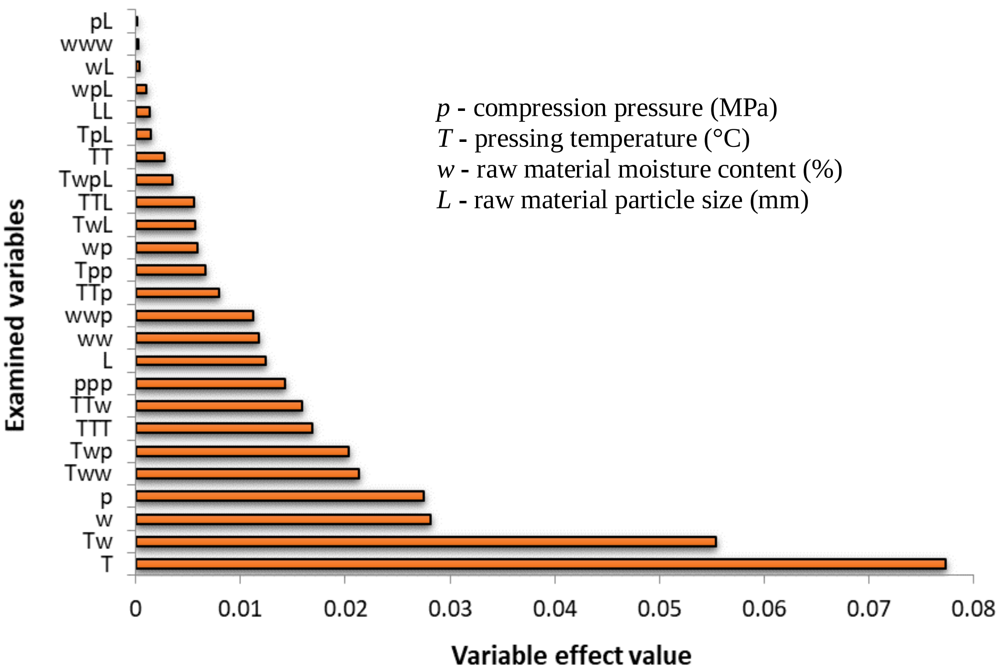
3.2. Densification of pine and oak wood sawdust

A similar methodology was used also at experimental densification of pine wood sawdust and also oak wood sawdust. Method of parameters effect (see Figure 4) determined that the biggest effect has pressing temperature and material moisture. Also, their interaction has a very significant effect on final briquette density. This is a very important and helpful result. The final form of a mathematical model which describes relations between influencing variables and final particle density during densification of pine sawdust.

Equation 3: Mathematical model for pine sawdust densification

$$\rho = 0.832962 + 0.00064563*p + 0.004026*T - 0.021436*w_r - 0.012259*L - 0.000045*(p - 130.2)*(T - 103.5) + 0.000249*(p - 130.2)*(w_r - 9.83333) + 0.001329*(T - 103.5)*(w_r - 9.83333) - 0.000022*(p - 130.2)*(T - 103.5)*(w_r - 9.83333) - 0.000005*(p - 130.2)*(T - 103.5)*(w_r - 9.83333)*(L - 2.03333) + 0.001707*(w_r - 9.83333)*(w_r - 9.83333)$$

Figure4. Individual variables effect (Parrett's effect diagram) for pine sawdust



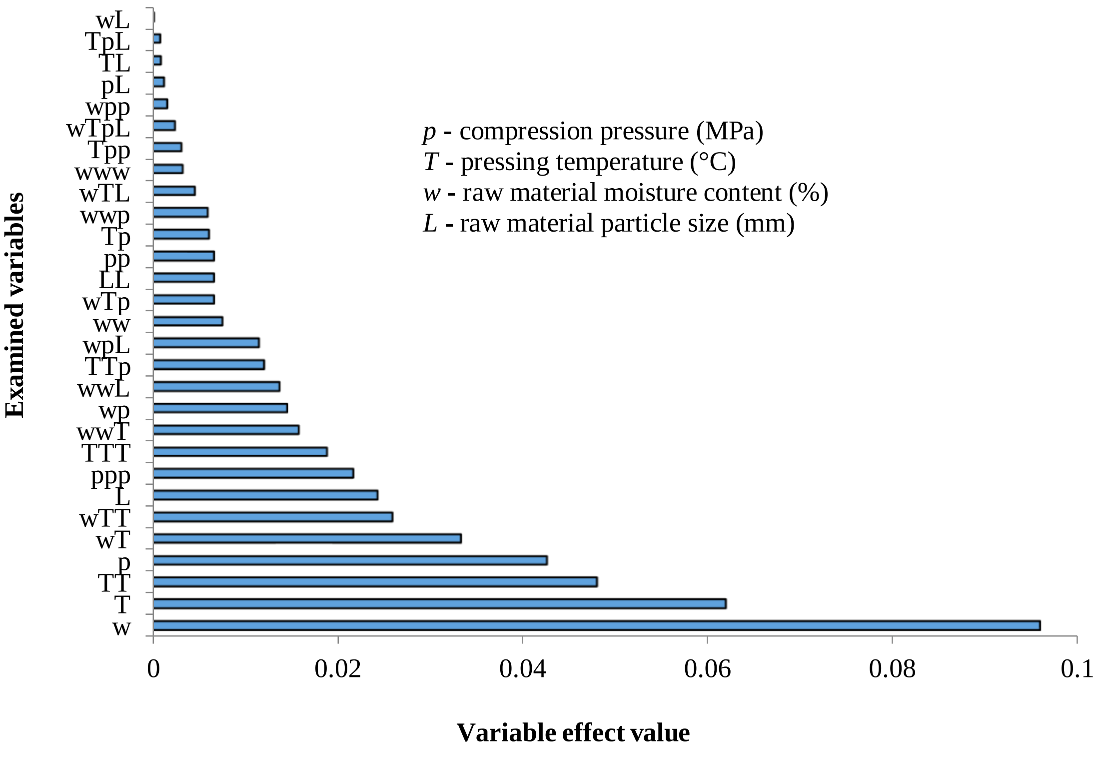
This equation 3 is the final mathematical model which describes the behaviour of pine sawdust during densification from the final briquettes density point of view. Have to write that designed mathematical model is valid only for pine sawdust and in the following range of examined variables: pressure 63 – 191 MPa, temperature 55 – 130 °C, moisture content 5 – 15 % and particle size 0.5 - 4 mm. With comparing with the model for acacia wood sawdust the range of examined variables is extended. The reason for the range extension was to determine the effect and interaction on a wide area. According to the extension also the design of the experiment was modified, a number of settings very increased to 30 settings. Also, the simple comparison of measured density values and calculated density values for pine sawdust confirmed that our experiment and measured data processing were correct and the difference between measured value and the calculated value when the mathematical model is used is under 5%.

Method of parameters effect (see Figure 5) at oak wood sawdust determined that the biggest effect have the raw material moisture content followed by pressing temperature and compression pressure. Also, other interactions have a very significant effect on final briquette density. This is a very important and helpful result. Different chemical composition and raw material properties proved the different conditions during densification. This fact proves the comparison of results on Figure 4 and Figure 5, where the effect of examined parameters at pine and oak sawdust densification are displayed. Effects of parameters and also of parameters interaction is not the same. Here you can see why is important to know the material and its behaviour during densification. The final form of a mathematical model which describes relations between influencing variables and final particle density during densification of oak sawdust.

Equation 4: Mathematical model for pine sawdust densification

$$\rho = 0.141992 + 0.001122*p + 0.005961*T - 0.039024*w_r + 0.000947*(T - 103.5)*(w_r - 9.83333) + 0.157317*L - 0.000004*(p - 130.2)*(T - 103.5)*(w_r - 9.83333)*(L - 2.03333) - 0.000003*(T - 103.5)*(T - 103.5)*(T - 103.5) + 0.028938*(L - 2.03333)*(L - 2.03333) - 0.063987*(L - 2.03333)*(L - 2.03333)*(L - 2.03333)$$

Figure5. Individual variables effect (Parrett's effect diagram) for oak sawdust



This equation 4 is the final mathematical model which describes the behaviour of oak sawdust during densification from the final briquettes density point of view. Have to write that designed mathematical model is valid only for oak sawdust and in the following range of examined variables: pressure 63 – 191 MPa, temperature 55 – 130 °C, moisture content 5 – 15 % and particle size 0.5 - 4 mm. A simple comparison of measured density values and calculated density values for oak sawdust confirmed that our experiment and measured data processing were correct.

The designed mathematical models can be used and can be operated in two basic ways - modes. In the first mode, the values of the examined variables (*p, T,* w_r*, L*) are configured, and the model calculates the estimation of the resulting particle density values after the analysis. In the second mode, the application functions in a reversed manner. The particle density values of the briquette, along with three of the examined variables, are set. The application will calculate the estimate for the remaining variable after the analysis. The mathematical model can be a useful tool for estimating the quality of briquettes according to the configured variables or for optimizing the parameters of pressing machine, pressing chambers, etc. The designed mathematical models can be used also in the control of densification machine during operation, model like a control tool inside the control system of the machine. When the influencing parameters are controlled - managed, it will be possible to optimize the entire densification process for a different kind of input raw material and thus to design an optimized machine construction for each customer, depending on its requirements. Optimization will allow you to set up process parameters so that the production quality achieved meets standard limits at the lowest operating and investment costs.

IV. RESULTS AND DISCUSSIONS

Design of experimental plan, a methodology for evaluation of experimental research and research findings regarding the effect of the important variable during densification of pine sawdust, oak sawdust and acacia tree sawdust was presented in this paper. However, the design methodology can be and should be used for each raw material. Authors presented the process and methodology of DOE an evaluation process when the mathematical model has to be the final result. Presented mathematical-statistic methods are widely known, but the usage and application in this area are not usual. Presented research findings relate to the effect of material and densification process variables determination on final solid biofuels particle density. With the application of mathematical and statistical methods, is possible to design a mathematical model of single-axis densification of raw sawdust. The mathematical model can be implemented as a basic algorithm into software which is very easily usable for the audience.

V. CONCLUSION

In this paper, we would like to present the developed mathematical models which describes the influencing variables interaction and influencing variables effect on final biofuels quality. The prediction and control system works with research findings regarding the effect of compression pressure, pressing temperature, particle size and moisture content. According to the designed experimental plan the mathematical models for different woodens were developed which describes a related variables interaction and variables effect on final biofuels quality. This mathematical model can be useful at optimizing of the densification process related to an input raw material. The main conclusions that can be drawn from this study are as follows:

• Applicability of mathematical and statistical methods was proven.
• All of the investigated input variables have an effect on monitored outputs.
• Effect of examined variables was different for each pressed raw material, kind of raw material effect was proven.
• The final solid biofuels particle density can be increased by a sensitive levels selection of influencing variables.
• The designed mathematical model can be used for prediction of solid biofuels density and also at construction design and engineering of densification machines.

Conflict of interest

There is no conflict to disclose.

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