

Peter Križan

The Densification Process of Wood Waste

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Managing Editor: Elisa Capello

Language Editor: Mary Boyd

Published by De Gruyter Open Ltd, Warsaw/Berlin
Part of Walter de Gruyter GmbH, Berlin/Munich/Boston



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ISBN 978-3-11-044001-0
e-ISBN 978-3-11-044002-7

Bibliographic information published by the Deutsche Nationalbibliothek
The Deutsche Nationalbibliothek lists this publication in the Deutsche Nationalbibliografie; detailed bibliographic data are available in the Internet at <http://dnb.dnb.de>.

Managing Editor: Elisa Capello
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www.degruyteropen.com

Cover illustration: © Peter Križan

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This monograph is dedicated to research in the densification processes of saw dust from pine and its mathematical description for uniaxial pressing into compact briquettes. This publication shows an experimental strategy to determine the effects of individual parameters on the resulting density of the formations. This publication also includes the construction of an experimental pressing stand that measures the effects of the technological, material, and structural parameters on the resulting density of the briquettes. This work specifies parameters and identifies their effects on the quality of the briquettes. These effects were experimentally verified and described through a mathematical model. The mathematical model serves to predict the density of briquettes for some predefined pressing conditions. The pressing conditions include the temperature, compaction pressure, humidity, and size of raw material fractions. This mathematical model can aid in the design of pressing machines. For the simplified and clearer use of the mathematical model, application software was developed for the optimization of technological parameters and their resulting effect on the quality of the briquette. The monograph also provides the results of experiments performed by the author on different types of wood biomass. These results demonstrate the dependence of parameters effecting the final quality of the briquettes and their resulting influence on the densification process itself. This publication also defines the level of importance of the results in terms of optimization of the pressing chamber.

Approved by the scientific council of the Faculty of Mechanical Engineering, STU in Bratislava based on the editorial plan 25.02.2014

Abbreviations

A	material constant giving the elastic properties of the pressed material
a	radius of the lower interface of the spherical shelter where the plastic deformation within the pressing chamber is concentrated (mm)
A_3	work of the active pressure on the upper interface where plastic deformation is concentrated (J)
a_A	equivalent diameter of the particles (circular) (mm)
A_D	deformation work for shape change (J)
A_f	work of the contact friction on the conical contact area (J)
A_i	sensitivity coefficient for i
AIC	Akaike Information Criterium
AICc	Akaike Information Criterium of the second degree of correction
A_k	coefficient calculated based on experimental results of the characteristic shape and curve.
AR	Pellet abrasion (%)
a_V	equivalent particle diameter (balls) (mm)
B	material constant, which describes the content of volatile substances and cellulose in the material
b	radius of the upper interface of the spherical shelter where plastic deformation in the pressing chamber is concentrated (mm)
C	compressibility factor
D_1	diameter of the filling chamber (mm)
D_2	pressing chamber diameter (mm)
D_3	diameter of the extrusion chamber (mm)
D_4	diameter of the calibration part of the pressing chamber (mm)
DWM	disintegrated wood mass
D_k	opening diameter of the pressing chamber (mm)
d_n	briquette diameter (mm)
D_N	normalized briquette diameter for biofuels (mm)
D_p	mechanical resistance of pellet (%)
E	material modulus of elasticity (Pa, MPa)
E'_0	volume modulus of deformation (Pa, MPa)
E_0	volume modulus of elasticity (Pa, MPa)
e_0	initial porosity
E_L	modulus of elasticity in the direction of fibres (Pa, MPa)
E_R	modulus of elasticity in the radial direction of fibres (Pa, MPa)
E_v	modulus of elasticity in the vertical directions of fibres (Pa, MPa)
F	ratio of the average square
f_c	strength of briquette (Pa, MPa)
F_m	pressing force on the stopper (N)
F_{max}	maximum force applied to the briquette when splitting (N)

F_N	normal force (N)
F_μ	friction force (N)
G	ratio of elasticity in the vertical and radial directions of the fiber bundles
H	length of the compressed briquette in the chamber (mm)
HL	hygroscopic limit
H_0	hypothesis
i	number of measurement settings
j	number of pressings for a single setting i.e. number of repetitions
k	number of factors examined
K_0	coefficient of lateral pressure
k_m	number of model parameters
kp	coefficient related to pressure
$LCWS$	limit of cell wall saturation
L	fraction size of input material (mm)
l_4	length of the calibration part of the pressing chamber (mm)
l_k	length of the pressing chamber (mm)
L_k	overall length of the pressing chamber (mm)
L_N	length of briquette according to standards for biofuels (mm)
l_n	length of briquette (mm)
L_p	maximum probable function for the estimated model
l_z	length of the compressed briquette column (mm)
m	weight of loose particles (kg, g)
m, f, n	points defining the upper interface where plastic deformation within the pressing chamber is concentrated
m', f', n'	points defining the lower interface where plastic deformation within the pressing chamber is concentrated
m_0	absolute dry weight of wood (kg, g)
m_A	weight of pellets after abrasion test (kg, g)
m_{Ap}	weight of pellets before sieving and testing (kg, g)
m_E	weight of pellets before abrasion test (kg, g)
m_{Ep}	weight of pellets after sieving and testing (kg, g)
m_n	weight of briquette (kg, g)
MS_A	mean square (regressive) (kg.dm ³)
MS_R	mean square (residual) (kg.dm ³)
m_v	weight of moisture in wood (kg, g)
m_w	weight of wood with a humidity of w (kg, g)
n, n'	exponent expressing the intensity of structure change
PFS	point of fibre saturation
p	pressing pressure (Pa, MPa)
P	calculated level of significance
p_G	backpressure in the press housing (Pa, MPa)
p_i	maximum pressure in the i^{th} measurement (Pa, MPa)

p_k	axial piston pressure (Pa, MPa)
p_m	axial pressure on stopper (Pa, MPa)
p_{m0}	initial pressing pressure (Pa, MPa)
p_N	normal (radial) pressure (Pa, MPa)
P_p	probability (%)
p_r	radial pressure (Pa, MPa)
$p_{r,r}$	residual radial pressure (Pa, MPa)
r	radius of the pressing chamber (opening) (mm)
R^2	coefficient of multiple determinations
R^2_{adj}	modified coefficient of multiple determination
RMSE	standard deviation of the residuals
S_2	cross-sectional area of the pressing chamber output (mm ²)
s^2_ρ	sample variance (kg.dm ⁻³)
S_4	cross sectional area of the pressing chambers calibration (mm ²)
$S_{\tilde{c}}$	area of the actual particle (mm ²)
S_k	element cross section (mm ²)
S_p	equivalent circular area (mm ²)
S_r	element side shell area (mm ²)
SS_A	sum of the squares between groups (regressive) (kg.dm ⁻³)
SS_R	sum of the squares within groups (residual) (kg.dm ⁻³)
SS_T	overall sum of squares (kg.dm ⁻³)
T	pressing temperature (°C)
T_k	distance between piston and lower end of the pressing chamber (mm)
U	extended uncertainty (kg.dm ⁻³)
u	standard uncertainty (kg.dm ⁻³)
$u(x)$	estimation of the standard deviation (kg.dm ⁻³)
$U(\rho)$	covariance matrix
u_A	type A standard uncertainty (kg.dm ⁻³)
u_B	type B standard uncertainty (kg.dm ⁻³)
u_C	overall (combined) standard uncertainty (kg.dm ⁻³)
u_ρ	standard uncertainty for ρ (kg.dm ⁻³)
V_0	initial volume – before pressing (m ³ , mm ³)
$V_{\tilde{c}}$	real volume of particle (m ³ , mm ³)
V_g	equivalent volume of a sphere (m ³ , mm ³)
V_n	briquette volume (kg.m ⁻³)
v_p	piston speed (m.s ⁻¹)
V_S	volume of the bulk material after pouring (m ³ , mm ³)
w_a	absolute humidity (%)
w_r	relative humidity (%)
x	size of particles (mm)
Z_i	source of z_i
z_{imax}	maximum deviation from the nominal value of z_i

α	level of significance (%)
β_j	regressive parameter for j
β_s	compressibility of shredded wood ($\text{m}^2.\text{N}^{-1}$)
$\hat{\beta}$	vector estimates of unknown polynomial parameters
γ	conical angle of the pressing chamber ($^\circ$)
δ_{dov}	maximum permissible deviation of the measuring equipment
$\delta_{dov} p_1$	maximum permissible deviation of calliper 1 (mm)
$\delta_{dov} p_2$	maximum permissible deviation of calliper 2 (mm)
δ_{dov}^V	maximum permissible deviation of digital scale (g)
Δe	change in porosity
Δy	compression of briquette after maximum obtained applied force (mm)
ε	relative deformation
ε_a	axis (axial) displacement
ε_i	measurement error
ε_r	axis (radial) deformation
ε_v	volumetric deformation (Pa, MPa)
λ	horizontal pressing ratio
μ	coefficient of friction for coupling
ν_{LR}	Poisson ratio in the longitudinal direction of fibre bundles
ν_{RL}	Poisson ratio in the radial direction of fibre bundles
ρ	density of wood (kg.m^{-3})
ρ_o	density of absolutely dry wood (kg.m^{-3})
ρ_n, ρ_N	density of the briquette (kg.m^{-3})
ρ_s	bulk density (kg.m^{-3})
σ_o	initial stress (Pa, MPa)
σ_a	axis (axial) stress (Pa, MPa)
σ_{Br}	strength of briquettes when splitting (N.mm^{-1})
σ_{Bt}	compressive strength of briquette (Pa, MPa)
σ_k	yield strength (Pa, MPa)
σ_m	axial stress on the plug (Pa, MPa)
σ_p	briquette strength in compression (Pa, MPa)
σ_r	radial stress (Pa, MPa)
$\sigma_{r,r}$	residual radial stress (Pa, MPa)
σ_s	center stress (Pa, MPa)
σ_t	tensile strength (Pa, MPa)

Indexes and other abbreviations are explained in the text

Foreword

The Institute of Manufacturing Systems, Environmental Technology, and Quality Management (IMSETQM) at the Faculty of Mechanical Engineering, SUT in Bratislava, has been involved in several projects and problems focused on the recovery of wood waste through densification into a biofuel. Some projects were focused on the complete design of production lines on the principles of waste densification, whereas other projects dealt with the development of pressing machines – pelleting or briquetting presses. The successful operation of a production line for briquettes, as well as for the design/dimensioning of new pressing machines, depends on robust knowledge of densification technologies and pressing parameters and their effects on the quality of the resulting briquette/pellet. The quality of the final product is greatly affected by the state and quality of the raw material before the pressing process. We know that the raw material should be dry and disintegrated. However, what are the optimal values, for example, of the raw material water content and fraction size? How do they affect the quality of the briquette and what are the dependencies between them?

Ever-increasing demand to develop self-sufficient energy sources creates increasing problems even in the area of wood waste densification that must be quickly solved. The main goal of this monograph is to provide a bank of information in the area of wood waste recovery through densification and endeavour on the detailed description of the densification process to create a mathematical model that accurately describes this process. This mathematical model will serve to predict the resulting density of the briquette given specific pressing conditions, such as: pressing temperature, pressure, water content, and size of the input fractions. The mathematical model can also serve as a guideline when sizing the pressing machine.

The scientific monograph *The Densification Process of Wood Waste* originated based on long-term experience in the areas of experimental research of densification processes for various types of biomass, as well as experiences obtained through the solution of several real world projects. This monograph is primarily intended for students studying for their masters (Ing.) and doctorate (PhD.) degrees in university. The submitted monograph offers students interested in manufacturing and environmental technology, providing a complete image on the effects of important parameters in the densification of wood biomass. The monograph can also be useful for professionals who deal with the production of solid high-grade biofuels (pellets and briquettes), the design of machines for the production of solid Biofuels, and experimental research in the area of densification.

In terms of the research process at (IMSETQM) for pressing wood waste, I realized that it is very important to analyze the following: the pressing process, processes occurring in the pressing chamber, and the quantification of factors effecting the resulting quality of the briquette. Therefore, this monograph is divided into chapters which are related to one another. Originally, I intended to solve these tasks for multiple material types. However, throughout my research, I discovered that problems in this

area are very broad and it is necessary instead to concentrate on a specific type of material. Thus, I decided to focus on pine chips for initial research.

The introduction describes biomass and its advantages and then focuses specifically on the composition of dendromass and the energy potential of waste produced from this material. The following chapter is dedicated to the analysis of the densification process, specification of parameters affecting this process, and the quantification of their meaning. The contents of this chapter also include the analysis of particular substances, effects occurring in the pressing chamber during densification, existing mathematic descriptions of the densification process, and quality of briquette. One of the chapters is devoted to the description of the experimental pressing machine, which allows for geometrical variability of the pressing chamber, tool, and parameters such as force and temperature. In this chapter, the multifaceted experiment is explained to verify the effects of analyzed parameters, describe the methodology for evaluating measured data, and propose a mathematical model describing the densification process. This model describes the effects of the analyzed parameters and their mutual reactions and is the basis for the development of software for the easy optimization of technological parameters in the pressing chamber. This model is thus a predictor of briquette quality as a result of the input parameters. Following the chapters discussing the experimental research of monitored parameters and their effects in the densification process of pine sawdust, the results of the experiments with various types of wood biomass are documented. In the closing chapter of this monograph, the significance of the experiments with respect to the design optimization of the pressing chamber is discussed.

The motivation of this monograph is to gather a bank of knowledge and experiences dealing with the densification process of wood waste and share these experiences and results undertaken at the IMSETQM.

For their comments and suggestions to this monograph, I would like to express my gratitude to the reviewers as well as my friends and colleagues Prof. Ľubomír Šooš, PhD. and MSc. Miloš Matúš, who have dealt with the problems in densification. Furthermore, I express my gratitude to Prof. Rudolf Palenčár, PhD. and MSc. Michal Svátek, PhD. for their valuable suggestions and comments regarding the preparation and interpretation of experiments. I would also like to thank my working colleague at IMSETQM, whom I have worked together with in various projects regarding the densification of biomass, the proposal of technology for production lines, and machines in the mechanical manipulation of biomass.

Finally, I would like to thank my fiancée Lydka and parents for their continued support, understanding, and patience throughout my creative process.

1 Introduction

Since ancient times, nature has been considered a source of inexhaustible raw materials and energy. Nature has created energy sources throughout millions of years. However, in recent times, due to a rapid rate of use and recovery, its resources are now limited to only several decades (crude oil, natural gas) or in some special cases, centuries (coal). Unlike fossil fuels, renewable sources of energy are readily available, but we need to begin using them more appropriately. According to EU Directive (2001), renewable sources of energy can be defined as a renewable, non-fossil, source of energy, i.e. wind, solar, geothermal, wave/tidal, water, biomass, waste gas, sewage treatment plant gases, and biogas energy.

Since the dawn of time, the Sun has been an essential source of renewable energy that humankind depended on. Solar energy provides everything we require as an energy source, naturally, cleanly, and without any risk. Heat for our houses, electric power for appliances, fuel for our vehicles – all of these can be produced from solar energy originating from biomass, wind, or water energy. Several experts claim that renewable sources of energy do not fully substitute classic energy sources and are considered as supplementary sources. However, how much could these supplementary sources contribute to overall energy consumption? The answer is only a matter of our willingness to support the use of these alternative sources given the fact that the amount of solar energy falling on Earth is much greater than current human consumption. For Slovakia alone, the difference is up to 200 fold (Ľapák & et al., 2008). Slovakia is a country in which the use of renewable sources of energy, maybe with the exception of water energy, is considerably under valued. However, I believe that the economic, environmental, energetic, and social advantages of using alternative energy sources will be fully utilized in the near future.

Biomass is, in essence, conserved solar energy that plants, through photosynthesis, store as highly energetic organic matter. During photosynthesis, plants absorb solar energy and subsequently convert it to biomass. Solar energy stored in biomass can be a useful source of energy – heat, electric power, or fuel for motor vehicles. This energy can be provided through many means, e.g. burning, fermentation, anaerobic digestion, and so on (Basu, 2010). Wood, straw, or specially grown plants with short rotation cycles show great potential in these areas.

The process of photosynthesis in an uninterrupted ecosystem functions according to Figure 1.1. Water and minerals ascend by means of xylem cells through zone (B) to the treetop. Part of the water participates in assimilation, but the majority evaporates from the tree leaves and needles into the surrounding air. Assimilation takes place during the vegetation period in sunny conditions. The product of assimilation descends via the phloem cells (L) and participates in the growth of the meristematic tissues, and example of which is the Cambium (K). Also, part of the product is stored in the cortex of the roots, trunk, and treetop.



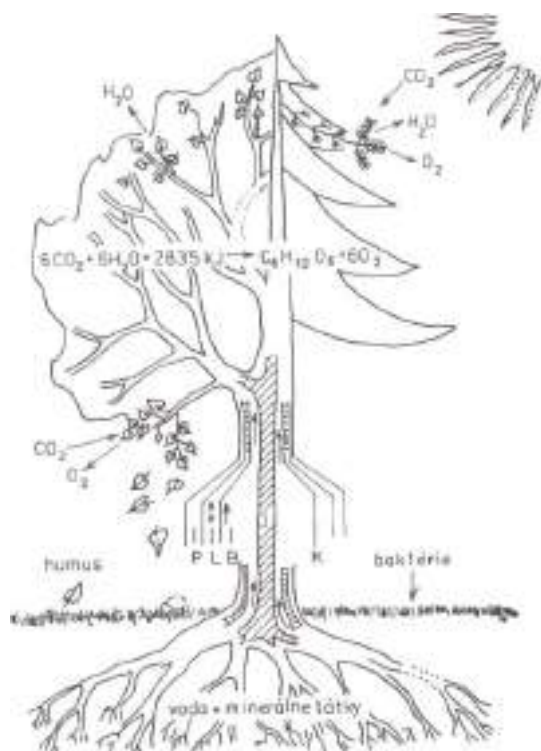


Figure 1.1: Parts of a tree, their physiological. Functions, and process of photosynthesis: B – xylem; L – phloem; K – Cambium; P – Periderm or Bark; J – Core (Požgaj, Chovanec, Kurjatko,&Babiak, 1997).

Biomass from wood is one of the most universal renewable sources of energy on Earth. It provides nutrition, and several products are made from it (paper, drugs, chemicals etc...), it is used as a building material, and its energy potential is used in the form of a fuel.

Burning this biomass yields chemically stored energy. Oxygen combines with carbon within the plant, resulting in the production of carbon dioxide and water. This process is a cycle since the carbon dioxide and water produced when burning wood are necessary substances for the growth of new biomass. It is worth mentioning that when we burn wood biomass, we only produce as much carbon dioxide that the plant needed in the first place to grow. This is not the case with the combustion of classic fossil fuels.

For this reason, we can appreciate the importance of energy from wood biomass in Slovakia as well. As a country where more than 90% of land consists of forest and farmland, the use of biomass has great potential. Besides the ecological point of view, there is an economic argument as well. Currently, a “biomass” fuel is still more expensive than traditional fossil fuels. However, the increasing rise of fuel prices may

soon make this statement untrue. Therefore, the energy value of biomass fuel is at least worth consideration.

The average energy value of dry wood or straw is approximately 4.2 kWh for one kilogram of raw material (Palz, Coombs & Hall, 2006). For comparison, about 2 kilograms of biomass is needed for the equivalent 1 litre of crude oil to be combusted with the same efficiency. Additionally, there is about 20% more energy in 1 kilogram of wood or straw than that obtained from one kilogram of brown coal. However, biomass has lower specific weight; therefore, maintaining the same amount of energy requires a significantly larger volume, which results in higher costs for storage and transportation. Another disadvantage is its decreasing energy output with increased water content.

Table 1.1: Comparison of the heating potential of some fossil fuels and biomass (Bédi, 1998; Jandačka, & Malcho, 2007).

Fuel	Unit	Energetic value (MJ)	Energetic value (kWh)
Light fuel oil	Kg	42	11,7
Biodiesel (MERO)	Kg	37	10,3
Natural gas	m ³	30	8,3
Biogas	m ³	24	6,7
Hard coal	Kg	28	7,8
Dry wood [$w_t = 10\%$]	Kg	16,5	4,6
Fresh wood [$w_t = 50\%$]	Kg	10	2,77
Brown coal	Kg	12	3,3
Straw [$w_t = 10\%$]	Kg	14	3,9

Advantages of using biomass in a biofuel form:

- Biomass is a stable domestic source of fuel, contrary to crude oil, gas, coal and nuclear energy. Its volume and price can be predicted with sufficient precision into the future.
- Lowering CO₂ emissions and other exhalants helps to fulfill international obligations.
- Burning this biomass is neutral and has no effect in increasing greenhouse gas concentration in the atmosphere.
- Sulphur concentration in biomass is more than 100 times lower compared to that in brown coal.
- The use of biomass for energy provides more ecological advantages – increase in forest quality and the prevention of soil erosion.

- Decentralized energy production reduces losses caused by logistics.
- Biomass can be produced and recovered without significant investment.

Biomass is all biodegradable fractions of products, agricultural waste, and residues (both vegetable and animal substances) from forestry and related branches, as well as biodegradable fractions of industrial and municipal waste (EU Directive, 2001).

Biomass sources can be found all around us. Agriculture and forestry are the main producers of biomass. Agricultural biomass consists of plant and grain phytomass, fruit tree and vine dendromass, high-energy plants, biofuel biomass, extrusions and silage from biofuel production, and farm animals' excrements. Biomass as a result of forestry is mainly composed of forest management dendromass, especially designated forest and fast-growing wood.

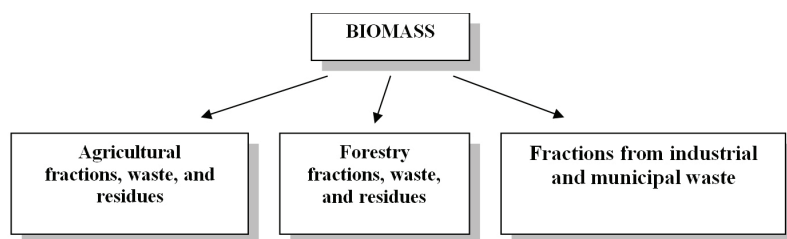


Figure 1.2: Classification of biomass (EU Directive, 2001).

However, biomass from other productions should also be included among potential sources. This biomass consists of municipal green vegetation, wind-belts, bank vegetation, and biomass produced by the woodworking industry, as well as biodegradable fractions of industrial and municipal waste (leftovers, paper, etc.).

Forests cover up to 42% of Slovakian terrain. These forested areas are a great potential source of accumulated energy. It is important to note that healthy trees, which are of higher-grade timber than firewood, cannot be considered as a source of potential energy. The potential is in waste timber, which is produced proportionally to the forest area.

Since ancient times, wood has remained one of the most universal and extensively used materials. Wood production for forest cover is the largest domestic source of material and energy. Logging and wood processing industries represent an important source of waste biomass suitable for energy production. Low demands are placed on soil quality for forests, and wood plants can grow on land that is unsuitable for agricultural crops.

Some of the physical properties of wood include: density and mass, moisture, thermal properties, electric and magnetic properties, and acoustic properties. Thanks

to its unique qualities, different woods are among one of the most extensively used materials. Wood has a relatively low weight in addition to good strength, which is important for construction materials. The mechanical properties of wood play an important role in its technological processing (cutting, pressing, bending, drying, etc.). Therefore, from this perspective, the behaviour of wood under stress should be known so that it can be optimally processed. Mechanical properties are divided into two groups: basic and derived properties. The basic properties include, for example, elasticity, strength, and plasticity. Included among the derived properties are: hardness, creep resistance, resistance to permanent load, and resistance to fatigue. The diversity of mechanical properties in wood and their dependence on direction is called anisotropy of mechanical properties (Chovanec & et al. 1985). Another group that can be considered are also the technological properties of wood, which include, for example the pliability, connectivity, workability, ability to be dried, and so on.

Wood, like any other material, is formed of atoms and molecules that are in a random or orderly arrangement in space. The degree of homogeneity of the atomic and molecular arrangement in wood and their orientation in particular covalent and hydrogen bonds determines the size and orientation of the mechanical properties on the micro-structural and macro-structural level (Chovanec & et al. 1985).

Table 1.2: Young's modulus and Poisson's ratio of some domestic wood materials along the main directions of elastic symmetry (L, R, T); ($w_a = 11 - 12\%$).

Woody plant	Along fibres		Radial direction			Tangential direction			
	Poisson's ratio		Young's modulus	Poisson's ratio		Young's modulus	Poisson's ratio		Young's
			E_L			E_R			E_T
	μ_{RL}	μ_{TL}	(MPa)	μ_{TR}	μ_{LR}	(MPa)	μ_{RT}	μ_{LT}	(MPa)
Spruce	0,489	0,557	13650	0,99	0,023	789	0,687	0,014	289
Ach	0,508	0,566	15789	0,727	0,056	1875	0,467	0,044	1269
Oak	0,365	0,452	11778	0,601	0,014	2046	0,328	0,038	1029
Poplar	0,507	0,59	13938	1,19	0,037	884	0,356	0,014	349

Many experiments have demonstrated that the elastic deformation of a low volume of wood proves the difference in the three mutually perpendicular directions, particularly in the longitudinal, radial and tangential directions (Chovanec & et al. 1985). The difference in mechanical properties in the longitudinal and perpendicular direction of the fibres results from binding energies of the wood chemical components. Covalent bonds are more active when a stress is applied in the longitudinal direction, whereas hydrogen bonds are effected when the stress is applied in the perpendicular direction.

A material characterized by such mechanical properties is known as orthogonal-anisotropic, i.e. orthotropic.

Wood is the oldest construction material known to man. Its properties depend on the species, habitat conditions (altitude, soil, light conditions, wind conditions, etc.), age, and spot in the trunk. Wood is flexible, does not lose carrying capacity due to heat, and resists creep. Wood is a good insulator of heat and vibration. Wooden interiors are generally a pleasant environment (e.g. in iron and glass, man can work, but not live) and it is also aesthetically pleasing (Chovanec & et al. 1985).

Wood represents a flexible, strong, and yet lightweight material that has excellent thermal insulation properties. It is able to carry heavy loads and dampen vibrations. It is easy to manipulate with cutting tools and can be connected and solidified with metal and other fillings. It has aesthetically impressive decorative features and can have excellent resonant properties. These natural characteristics of wood allow its use in the manufacture of building structures and furniture, musical instruments and packaging, sports equipment, and other products for general use. Many forms and uses of wood grow progressively with time.

Table 1.3: Properties of select wood species (Kremer, 1995).

Wood species	Wood density (Kg/m ³)	Strength (N/mm ²)			Shear strength (axial)	Young's modulus (N/mm ²)	Durability classes DIN EN 350-2
		Tensile strength (axial)	Compressive strength (axial)	Flexural strength			
Spruce	470	80	40	68	7,5	10.000	4
Pine	520	100	45	80	10	11.000	3 – 4
Larch	590	105	48	93	9	12.000	3 – 4
Birch	650	137	60	120	12	14.000	5
Beech	690	135	60	120	10	14.000	5
Oak	670	110	52	95	11,5	13.000	2
Ash	690	130	50	105	13	13.000	5
Locust	730	148	60	130	16	13.500	1 – 2

Wood became the subject of comprehensive research in chemistry, plastics technology and electronics, and also in atomic physics. Nowadays, the use of wood is used in shipbuilding; for the manufacture of wagons; in aeronautics, electronics and food industries; in the manufacture of composite materials, paper, and pulp; as well as other sectors (Chovanec & et al., 1985). The use of wood results in significant sources of chemical substances and energy as well as a source of sustenance for mankind. New ways of adjustments have been developed by which wood acquires completely new properties.

Wood of various species dried to a consistent weight has practically the same elemental chemical composition and thus the same density of wood substance. It contains, on average, 49.5 % carbon, 44.2 % oxygen, and 6.3% hydrogen. The leaves contain 46-47 % carbon, 7 to 7.3% hydrogen, 40 to 41 % oxygen, and up to 2% nitrogen, which is explained by the presence of larger quantities of proteins, chlorophyll, and other compounds (Požgaj & et al., 1997).

The bark contains more hydrogen but less carbon, less oxygen but a much higher mineral content. In addition to organic substances that are an essential part of wood, wood also contains minerals (that become ash when burned). Differences in the amount of ash produced depends on the conditions of tree growth, soil quality, season, age of the tree, part the tree, and so on. The proportion of ash after combustion of the wood varies mostly between 0.2 and 1.5%. The amount of ash after the combustion represents 6-7 % from the bark, 3.5 to 4 % from leaves, and 4-5 % from the roots (Chovanec & et al., 1985; Požgaj & et al., 1997).

Table 1.4: Chemical composition of some sources of energy (Chovanec & et al., 1985; Jandačka & Malcho, 2007; Požgaj & et al., 1997; Trenčiansky, Lieskovský & Oravec, 2007; Zacharda & et al., 2008).

Parameter	Unit	Fuel							
		Fossil fuel		Dendromass			Phytomass		
		Hard coal	Brown coal	Hardwood	Softwood	Bark	Willow	Hay	Straw
C content	(%)	73 – 84	65 – 75	50	51	46 – 48	49	49	49
H ₂ content	(%)	3,5 – 5,0	4,5 – 5,5	6,1	5,2	5,5 – 6,0	6,2	6,3	6,3
O ₂ content	(%)	2,8 – 11,3	20 – 30	43,3	42,2	36 – 40	44	43	43
S content	(%)	0,5 – 4	1 – 3	-	-	-	0,05	0,2	0,1
N ₂ content	(%)	< 1,5	1 – 2	0,6	0,6	0,3 – 1,2	0,5	1,4	0,5
Ash content	(%)	6 – 15	6 – 12	0,4 – 0,7	0,2 – 0,5	3 – 6	2	7	5
Heating value of dry matter	(MJ.Kg ⁻¹)	27 - 31	21 – 23	17,5 - 19	18 – 20,8	18 - 19	18,8	19,4	18,5

Even if the chemical composition of biomass differs among plant species, plants contain on average about 25% lignin and 75% hydrocarbons or sugars. The hydrocarbon component consists of sugar molecules linked together in long chains of polymers. Two major hydrocarbon components are cellulose and hemicellulose. Nature uses long cellulose polymers to build fibres that give plants necessary strength. Lignin acts as the glue that holds cellulose fibres together. Chemical components in wood can be divided according to Figure 1.3 (Požgaj & et al., 1997).

Table 1.5: Substance quantities in hardwoods and softwoods (Chovanec & et al., 1985).

Organic substances in wood	Hardwoods (%)	Softwoods (%)
Cellulose	40-50	40-50
Hemicellulose	25-35	25-30
Lignin	20-25	25-35
Pectin	1-2	1-2

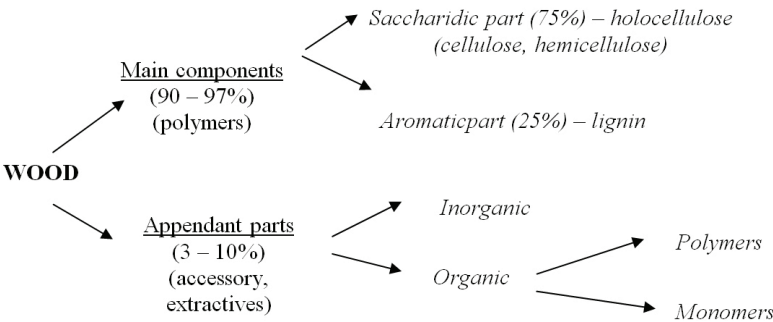


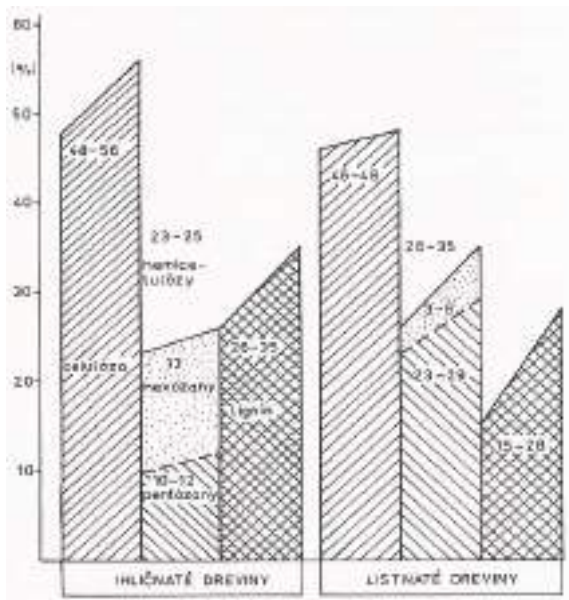
Figure 1.3: Chemical components of wood (Požgaj & et al., 1997).

Adhesion of the cell walls in wood is provided by the important component lignin. Lignin content is higher in softwoods than hardwoods. Experiments have confirmed that if the lignin is removed from the wood structure, the strength of wet wood significantly decreases. It can be concluded that lignin has a hydrophobic function in cell walls. Lignin reduces the effects of moisture; in fact, it protects cellulose – cellulose bonds. The main function of lignin, as a phenolic component, is to connect fibres (intercellular layer), but also to act as a strengthening factor for cellulose molecules within cell walls. Lignin adds strength to wood, especially for the resistance of compression. It envelops polysaccharides, with which is partly linked by a chemical bond. Distribution of the three main components of wood (cellulose, hemicellulose, and lignin) across the cross-section of a cell wall is not uniform (Figure 1.5) Middle lamella is composed almost solely of lignin. Lignin quantity decreases towards the lumen and cellulose, whereas in the hemicellulose it increases (Chovanec & et al., 1985).

Regardless of the main components of wood biomass (polysaccharides and lignin), each woody plant contains many other substances (called extractives) that are produced during growth by various complex biochemical reactions. These extractives are, for instance: fats, waxes, proteins, alkoids, simple and complex phenols, simple

Table 1.6: Representation of the major components in various woody plants.

Woody plants	Content (Mass percentage)			
	Hemicellulose	Cellulose	Lignin	Extractives
Scotch pine	28,5	40	27,7	3,5
Eastern white pine	22,4	42,3	25,4	8,3
Grey spruce	30,6	39,5	27,5	2,1
White spruce	25,8	42,1	27,8	2,3
Silver birch	32,4	41	22	3,0
Beech	35,3	39,2	20,9	4,1
Red maple	23,1	44,5	22,8	2,5
Liriodendron	30,3	33,5	25,3	3,6
Aspen	25,5	50,2	18,2	2,4

**Figure 1.4:** Incidence of major organic matter in coniferous (left) and deciduous (right) trees (Požgaj & et al., 1997)

sugars, pectin, starches, terpenes, saponin, glycosides, and essential oils. These substances do not belong to the structural part of the cells, but in most cases they have protective functions. Their distribution in wood is uneven.

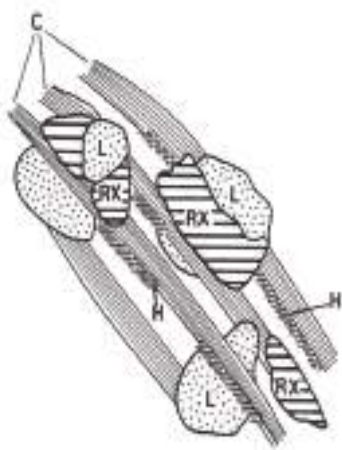


Figure 1.5: Biomass fibres - cellulose fibres (C), lignin (L), hemicelluloses (H) and wood fibres (RX). (Požgaj & et al., 1997).

Biomass waste, from forestry for example, comes in many forms, including: wood chips, shavings, sawdust, splinters, remains of large-diameter wood after logging, waste from handling, tree stumps, and roots from comprehensive system of soil preparation. All these wastes provide possibilities for harvesting and the transformation into energy. In most cases, energy provided by wood is thermal energy, which can be used for water heating and heating facilities.

In addition to the production of heat, there also exists the possibility of producing electricity. The most popular form of wood biomass energy recovery is direct combustion technology due to low cost, easy handling, and high reliability. Of course, burning biomass is not the only method of thermo-chemical conversion. Gasification and pyrolysis are included as well.

However, proper modification of these kinds of wood biomass is very important for effective recovery of their energy potential. A refinement process leads to homogenization of material composition and size, the adjustment of moisture content, the modification of its composition, and the alteration of other physical and mechanical properties. Solid biofuel can be processed for optimization of the shape for combustion; speed, length, and evenness of burning; and optimal shape for automated transport during the combustion process. The density of energy limits comfort during transport and minimizes costs of transportation and storage. Chemical properties of fuel can be changed by refining the raw material. Such modified biofuels have unlimited stability without biodegradation. At the same time, it is essential to ensure the harmonious relation of impacts of energy recovery of waste on environmental quality in particular through the implementation of best available technologies (BAT). These technologies

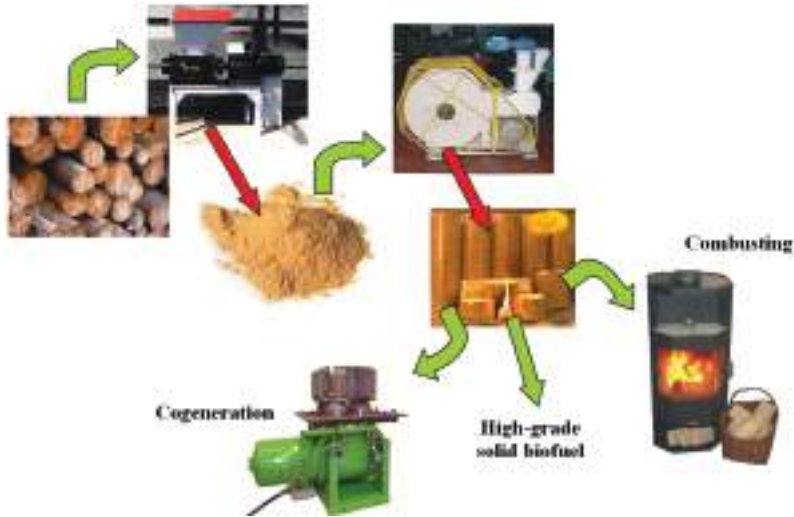


Figure 1.7: Schematic representation of the densification process.

will recover otherwise unutilized waste effectively and without further impacts on the environment. Technologies of densification are one possible solution in transforming suitable amounts of organic waste material into a modern biofuel by pressing it into a compact unit in the form of pellets or briquettes. However, before biomass waste can be compacted into briquettes and pellets, it is necessary to modify its physical and chemical properties. It is necessary to mechanically adjust biomass waste prior to densification in almost all cases. The basis of any modification of waste is sorting. Waste is generally heterogeneous; it is a mixture of different materials. The aim of sorting is to separate utility constituents from unnecessary components based on their different characteristics. Disintegration is used to soften or reduce the size of the waste material fraction. Drying biomass is, in addition to a thermo-technical process, a process which particularly improves the properties of biomass.

Production of high-grade solid biofuels is a good way to efficiently recover biomass and other waste energies. A 21st century fuel must satisfy the criterion of high comfort and security in addition to fulfilling energy, environmental, and economic criteria during its recovery. Modern energy carriers will be made of harmless raw materials – waste -- and should be suitably-shaped and of uniform size fraction, density, and moisture content.

2 Analysis of the Densification Process

Solving problems associated with the densification process requires complete knowledge about the pressing process of wood biomass itself. This means that we must understand the distribution of pressing forces; parameters affecting the pressing process and their related interactions; ideal material for pressing and their structure and properties; dependencies between material properties and densification parameters; as well as existing pressing technologies. We know that wood, in terms of recovering energy through densification, must be prepared in a way that is advantageous for pressing. Wood biomass intended for pressing must be disintegrated and, in some cases, sorted to obtain a homogeneous fraction size. It is also necessary to obtain ideal water content either through drying (commonly used) or humidification (spraying, steaming). Transforming wood biomass into a usable biofuel commonly utilizes wood chips or saw dust as a raw input material. With these, it is necessary to understand their mechanical and chemical properties, particle geometry, force interactions between individual particles, and the effect of these “material” properties on the pressing process. Thus, these parameters must be considered to predict and ensure the final quality of the briquette product. Standards exist which determine the quality of the briquette. Understanding the quid lines within these standards allows for the solution to many problems that originate from the design of the pressing machine.

2.1 Particulate Matter from Wood Biomass

Particulate matter from wood biomass are composed of contacting solid particles (solid phase) and liquid/gas phase. The liquid/gas portion fills the pores between the solid particles, known as the external pores. Internal pores exist within the particles themselves. If the solid particles are sufficiently porous, the surface (open) pores and internal (closed) pores are known. The quantity of liquid/gas within the external pores can vary. Sometimes it is in the form of absorbed water on the surface pores of the solid particles, and it can penetrate into the internal pores of the particles themselves, which substantially changes their geometric and physical properties. Surface pores increase the surface area of particles while internal pores effect the density or strength of the solid particles.

The relationship between the size of the surface and properties of the solid particles is essentially that, when enlarging the size of the surface, the amount of atoms and thus the overall size of the interface between phases also increases. Efforts in increasing the number of binding imbalances between atoms commonly manifests in a large spectrum of varying surface interactions, which are mainly in absorption and catalytic effects that result in significant change to the colloidal and hydraulic properties.



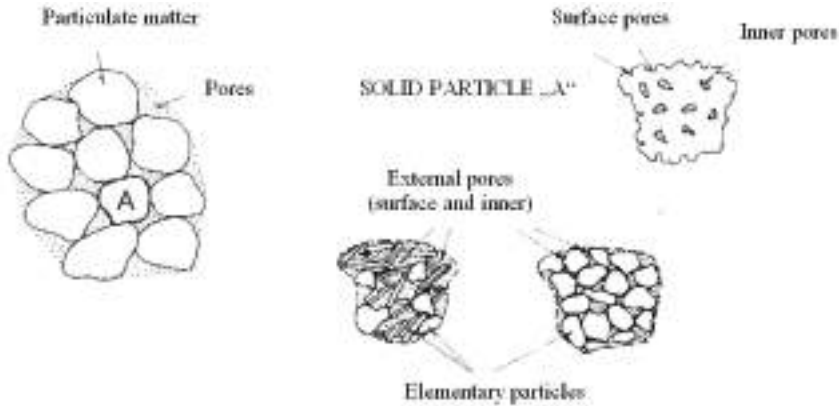


Figure 2.1: Solid particles “A” in particulate matter (Jindra, 1984).

Increasing the surface of the solid substance has the natural effect of increasing its reactivity – as is true in homogeneous and heterogeneous reactions. Chemical reactivity is essentially proportional to the surface size. Exceptions to this are situations when the surface pores are coated with an inert or non-reactive substance. However, in cases where the particle size is reduced by means of mechanical-chemical methods (shredding, for example) the exposed internal surfaces also increase the reactivity. There are many examples which document the mutual relationship between surface size and properties of solid particulate matter. However, it is necessary to remark that the change in physical-chemical properties of solid particles is related to only significant changes in surface size. Nevertheless, it is obvious that even small changes in particle size fundamentally affect the spectrum of application for these substances. Therefore, the size of the surface affects most of the substance properties (Fedá, 1977). The relationship between surface size and physical-chemical properties of the solid particles resides in the fact that increasing the size of the surface increases the number of atoms that compose the interface between phases. For individual particles, the amount of atoms in the interface between phases increases relatively, which increases the total number of atoms in the particles system.

Solid particles in particulate matter contact each other. This contact restricts free movement between individual solid particles and thus affects the particulate matter strength and stiffness. The particle degrees of freedom in wood chips change with respect to the magnitude of stress throughout the deformation process. Increasing stress reduces the degrees of freedom until the magnitude of stress reaches the yield stress of the particle (wood chip), resulting in the disintegration and subsequent increase in movement freedom.

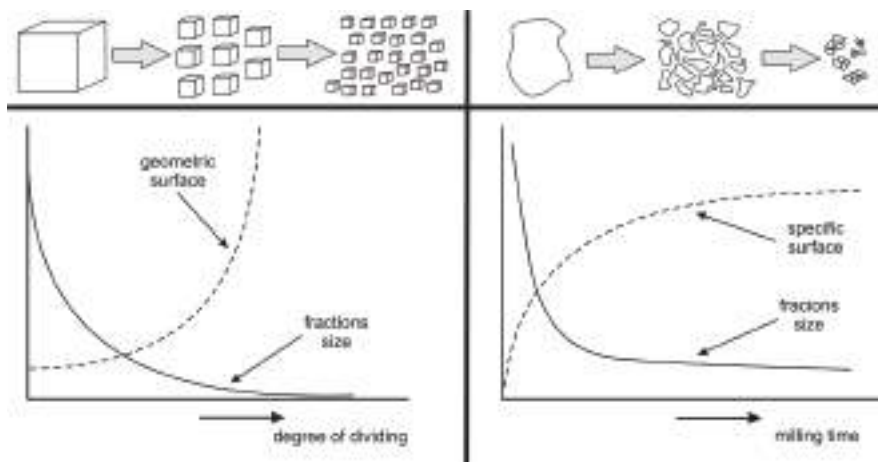


Figure 2.2: Difference between changing surface size of abstract and real solid bodies (Jesenák, 2008).

This property is exploited in the pressing process of solid particles, in which the action of pressure and heat densify solid particles into a compact whole. However, when pressing, stress must not exceed the yield strength of the solid particles. The goal is to minimize the pores occupying space between solid particles, contribute to stronger interactions between individual particles, and thus obtain a higher particle density briquette.

2.2 Structure of Wood Biomass Particulate Matter

The mechanical properties of particulate matter depend also upon their arrangements. If we are considering the arrangement of particles, we are talking about the structure or texture of the particulate matter. The structure (internal) of particulate matter defines the character and geometric arrangement of the solid particles and the behaviour of interactions between them. This behaviour depends on the number of structural bonds, which is a result of the size, shape, roughness, and strength of the solid particle. All of these factors influence the conceptual structure of the particulate matter and its heterogeneity (alternating surfaces of fine and coarse particles; particles of varying composition, shape, orientation), known as the texture or sometimes known as the macrostructure. It is important to differentiate between the structure of substances composed from particles of varying size (substances with varying degrees of polydispersion) and substances with equal polydispersion but with varying absolute size of particle. Fig. 2.3 describes examples of particulate matter composition (structure). In general, the most complex structure occurs when varying particulate matters are mixed together.

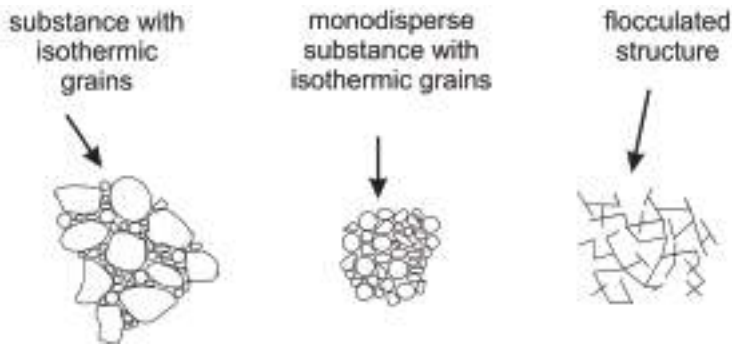


Figure 2.3: Examples of particulate matter structure (Jesenák, 2008).

In terms of pressing, it is very important to know the composition of the particulate matter, its structure, and the size of particles. The size and structure significantly affect the bonding mechanisms and the strength of these interactions upon pressing. These properties affect the continuity of the densification process and quality of the briquette product.

Factors that restrict the structure of the particulate matter can be divided into three groups: character of the solid particles, geometric arrangement of the particles, and the behaviour between them. In the first group belongs, foremost, the individual and group characteristics of the solid particles: the strength, composition, and structure of the solid particle; and the size, shape, and roughness. The most widely used characteristic of this group is the granularity curve – diagram of the particle phase composition (Fig. 2.4). It is possible to change the phase composition before the pressing process. The amount of water can be reduced, to a degree, by drying. The smaller the particle, the higher the effectiveness when drying.

However, this statement doesn't apply over the complete range of particle sizes due to compliance with safety factors when drying so as to prevent combustion of dust particles. The goal is obviously not to obtain completely dry particulate matter (complete removal of water). If completely dry particulate matter were pressed, we would obtain sintered particles, which are unacceptable.

Unlike the described properties, parameters such as structure and state variables form the second and third group of factors. Examining these factors requires following the substance state, which necessitates using an undisturbed sample. The second group of factors defines the geometric arrangement of the solid phase. The simplest of them is the average volumetric mass from which we can derive porosity. If solid particles create a cluster of elementary particles, then it is important to describe the geometric arrangement of the clusters.

The third group of structural characteristics includes the bonds between solid particles. For particulate substances, it is possible to differentiate two types of bonds:

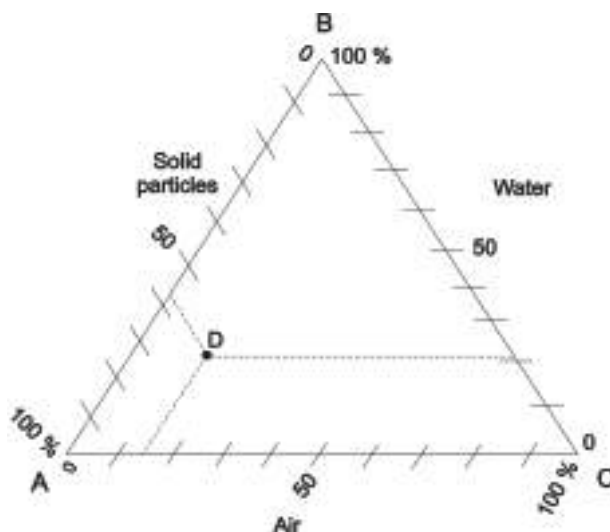


Figure 2.4: Triangle diagram representing the phase compositions (Fedá, 1977).

bonds effected by external loading (including weight), which are referred to as frictional bonds, and bonds created from internal stress, referred to as cohesive bonds. These occur as a result of varying bonding mechanisms that affect the mechanical properties as well as the pressing process itself. In Section 2.4, we describe them in more detail. The theoretically calculated tensile strength of particles σ_t in relation to the size of the particle x from the initial substance for varying bonding mechanisms can be seen in Fig. 2.5. The dashed line parallel to the x-axis divides the diagram into areas I and II. Area I represents a briquette subject to high pressure. In this area, the particles of the briquette are in surface contact (Jindra, 1984). Area II represents the pressing process where bridges are applied around the contacts between particles (Jindra, 1984).

The diagonal lines divide the diagram into regions, which define the bonding mechanisms. From this diagram, it is also clear that disintegrating wood biomass into very small particles is good practice before pressing so that we obtain the best mutual bonding between material particles.

2.3 Predominant Mechanisms of Internal (force) Bonds

Related contact phases in the particulate substance create force bonds between particles based on various mechanisms. These bonds affect all mechanical properties of the particulate matter. Even though detailed research about the effects of individual bonding mechanisms exist, only a few of these are suitable for the mathematical

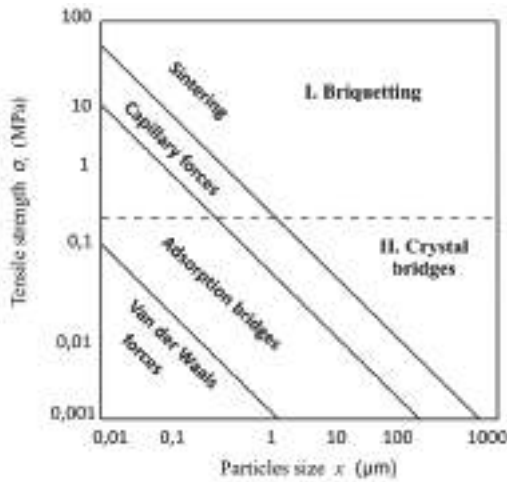


Figure 2.5: Theoretical strength of the bonded substance particles in relation to the size of particle before pressing (Jindra, 1984; Medek, 1991).

description of the particulate system. The particle contact strength is a collection of elementary bonds on the surface of the actual contact. In real particulate substances, all types of bonding mechanisms practically act together. The known theoretical approach in calculating the adhesion forces on a part of a system apply to roughness. The Jenkins test, a shear test, is used to experimentally prove the calculations. The tensile strength of adhesion can be specified from the characteristic values obtained for bulk material, i.e. many points of contacting particles characterised by the coordinate K and roughness of the contact surface. Emerging bonds between particles influence several factors, mostly the saturation of liquid (amount of liquid component on the surface of the particle and between particles).

Under current definitions, we can distinguish between:

- dry particulate substance – a substance that does not contain any liquid phase; only a solid consisting of particles with air/gas filling the spaces between them. Saturation $S = 0$ (Fig. 2.6a).
- damp particulate substance – a substance which consists of a liquid phase together with the solid phase, where some liquid occupies the spaces between particles. Saturation $0 < S < 1$ (Fig. 2.6b, c, d).
- paste – a particulate substance which consists of only a liquid phase comprised of particles whose pores are completely filled with liquid. Saturation $S = 1$ (Fig. 2.6e).

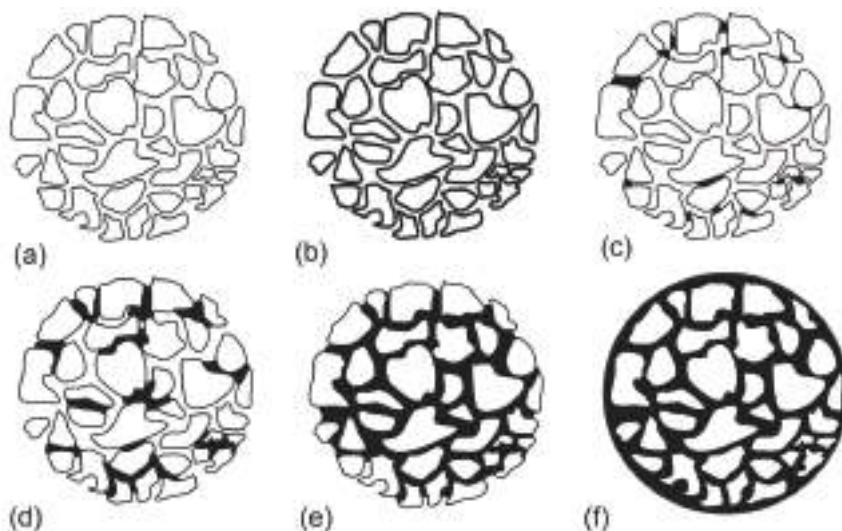


Figure 2.6: Schematic representation of varying degrees of saturation between particles (Pietsch, 1991). (a) no saturation – dry; (b) absorption layers; (c) liquid bridges (pendular state); (d) transition state (funicular state); (e) completely saturated state (liquid state); (f) droplet.

According to Pietsch(1991), we know five main groups of bonding mechanisms and several subgroups:

1. Bridges of solid character– bonds that occur between the contacts of the particles created by bridges in the solid phase. Their occurrence results in:
 - sintered particles (diffusion of atoms and molecules at higher temperatures);
 - partial melting of the particle surface (melting adhesion – cold welding);
 - chemical reaction between particles (usually activated by moisture);
 - hardening of the binding between particles (usually activated by moisture);
 - recrystallization of the solid phase from the liquid component;
 - drying (recrystallization of soluble components and the elimination of non-liquid particles).
2. Adhesion-cohesion forces – bonds created by adhesion in the interfacing surfaces and action of internal cohesive forces caused by:
 - use of high-viscosity adhesives (particle bonding with a high-viscosity adhesive),
 - formation of absorption layers $< 3 \text{ nm}$ / (small particles attracting surrounding atoms and molecules creating an absorbing layer.
3. Surface stress and capillary pressure– interactions with the most common bonding mechanism between particles of moist materials being:
 - liquid bridges (created in materials with a higher liquid content in the contact area (water, capillary condensation),

- capillary pressure (if the pore volume between particles in the disperse system is filled with water, capillary pressure creates a bond between the pores and surface of the particles).
- 4. Attractive forces between particles—even in cases where bridges of some substance do not occur between particles, a force bond caused by molecular interactions create fields in the particles. The particles themselves can emit these fields which we categorize as:
 - molecular forces (Van der Waals force, valence forces – free chemical bonds – hydrogen bonds),
 - electrostatic forces,
 - magnetic forces.
- 5. Shape bonds—particles in the shape of fibres, flakes, petals, or particles with irregular surfaces that attach/tangle with each other when pressed.

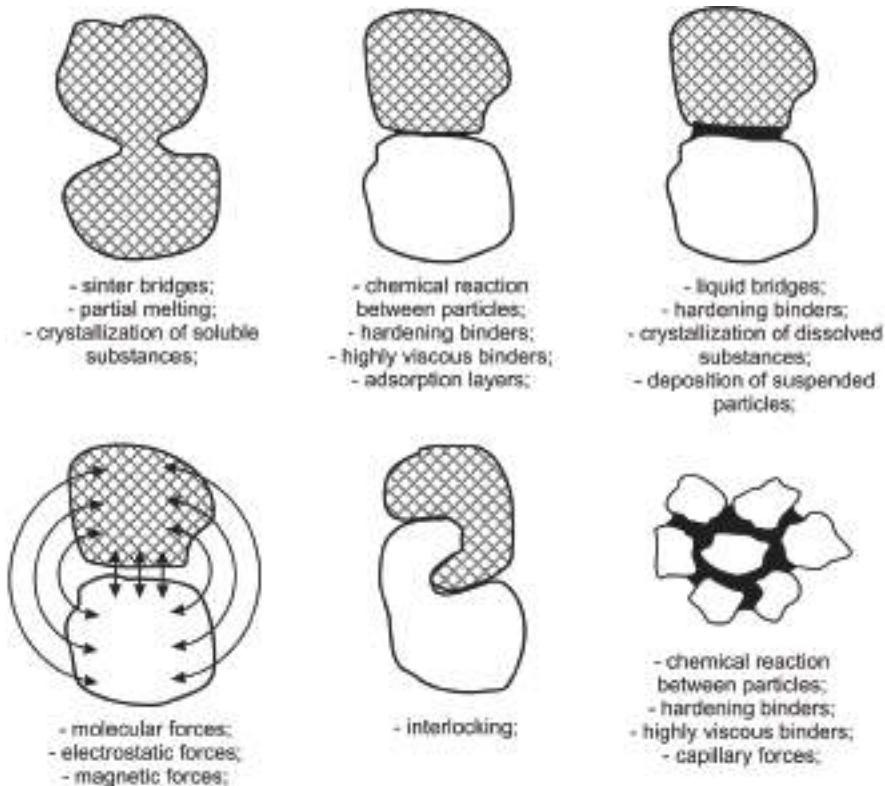


Figure 2.7: Schematic (illustration) representing bonding mechanisms (Pietsch, 1991).

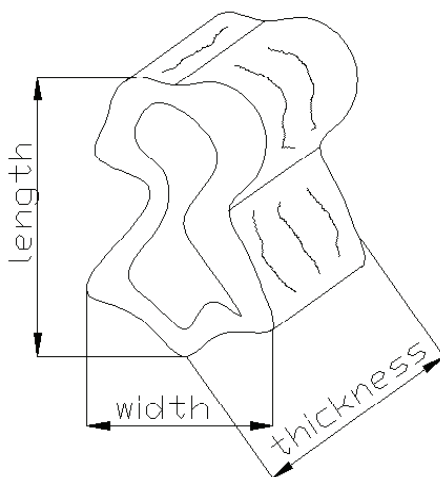
Bonding forces caused by the mechanisms discussed above can be considered internal cohesive forces acting between particles. These forces ensure a bond between particles even after we release external forces acting on the particles. Normal compression and friction forces occur in the areas loaded by external forces.

2.4 Properties of Disintegrated Wood Biomass

When cutting, disintegrating, or processing wood, chips and saw dust are a resulting by-product. Their shape, size, and quantity are dependent on the physical-mechanical properties of wood as well as the shape, size, and sharpness of the cutting tool or technology used to process the wood. We know that the process of wood disintegration is important with respect to the size of input fractions when pressing. Therefore, it is necessary to know the properties of disintegrated wood.

Chips – wood particles caused by cutting, disintegration, or machining of wood. Depending on the method of processing, these particles are also known as splinters, shavings, dust, or fibres and can be unified under a common term – disintegrated wood mass (DWM). DWM characteristically does not occupy the whole space of its bulk mass; air spaces exist between the solid particles. This reality puts DWM into the category of dispersion – a disproportionate system composed of two phases. Another typical property of DWM is that when each individual wood particle shifts, the overall external shape of the bulk changes as well. These properties specify DWM as a loose bulk material.

Amongst the basic descriptors characterising the physical and physical-chemical properties of bulk DWM are:



- size of particles,
- particle shape,
- granularity,
- moisture content,
- bulk density,
- bulk angle,
- slip angle,
- falling speed,
- compressibility of wood matter,
- electrical properties of dust particles,
- volatility of dust particles,
- coagulation of dust particles,
- abrasiveness,
- biological effects of dust particles on the human organism.

Figure 2.8: General dimensions of a DWM particle (Hejma& et al., 1981).

All of the above-mentioned properties describe and characterize DWM; however, not all of them are important in terms of pressing. The main parameters (properties) that characterize DWM are particle size and shape, granularity (granular composition), moisture, bulk density, and particle compressibility. In the next part of this monograph, we will dedicate more attention to these properties, since they directly affect the pressing process of wood materials.

2.4.1 Particle Size of DWM

The size of DWM particles is one of the most basic properties. It affects the physical properties of the bulk DWM such as bulk density, granular composition, slip angle, and pour angle. It also affects the movement properties of the particle in various transport systems, vacuum systems, and drying and separation conditions.

Particle dimensions are defined as the dimensions of the right angle parallelogram described by a given particle, where:

- length is the largest dimension of the particle,
- thickness is the smallest dimension of the particle,
- width is the third dimension of the particle.

Particle size defines the largest dimension of the right angle parallelogram around a particle (length of particle).

DWM particles resulting in wood cutting, disintegration, or wood-working differ considerably in size. A homogeneous fraction size can be achieved only in disintegrating machines or through sieving/screening the raw material. The maximum size of the particle is dependent on the type of disintegrating machine, size of the disintegrating wedges, or screen size following the disintegrating chamber. The maximum and minimum particle dimension from wood-working can be seen in Table 2.1. In the disintegration process, the maximum and minimum particle dimensions depend on the size of the screen.

2.4.2 Particle Shape of DWM

As was mentioned before, the geometric shape of wood particles depends on the wood-working process, and each result in very diverse dimensions. Nevertheless, we can attempt to divide each type of bulk material into groups:

- *isometric particles* - particles which have approximately the same dimension in all three directions (fine chip fractions, fine dust fractions)
- *flat particles (laminar)* – length and width are significantly larger than the thickness (splinters, shavings, flakes).
- *fibre particles* – particles with significant elongation in one direction (fibre, medium thickness dust particle fractions).

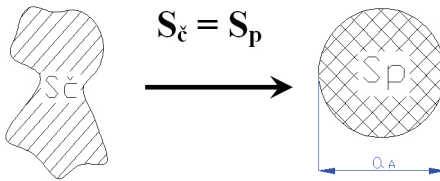
Table 2.1: Size of DWM particles (Dzurenda, 2002).

DWM particle method	Particle Size	
	minimum (mm)	maximum (mm)
Chips (wood chipper)	2.5	40
Splinter (wood disintegrating machine)	0.16	6.5
Wet sawdust (sawdust from wood ban saw)	0.064	4.8
Dry sawdust (longitudinal circular saw)	0.027	3.5
Dry wood fibres	0.5	4
Wood dust (belt sanding)	0.001	0.5

Depending on the technological properties of DWM and on other properties, an effort is made to characterize bulk particles by a single dimension. Depending on the processing method of DWM, such a dimension can be defined as follows:

- *equivalent particle diameter* a_A – diameter of the circle a_A , whose area S_p is the same size as the projected real particle areain the direction of its thickness $S_{\check{c}}$ (Fig. 2.9).

$$a_A = \sqrt{\frac{4 * S_p}{\pi}} \quad (\text{mm}) \quad (2.1)$$

**Figure 2.9:** Equivalent particle size according to the projection of a_A (Dzurenda, 2002).

- *equivalent particle diameter* a_v – is a diameter of the sphere whose volume is V_g is equivalent to the real particle $V_{\check{c}}$.

$$a_v = \sqrt[3]{\frac{6 * V_g}{\pi}} \quad (\text{mm}) \quad (2.2)$$

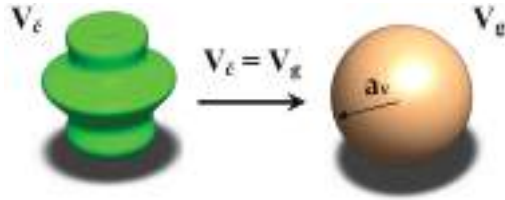


Figure 2.10: Equivalent particle size with respect to volume a_v (Dzurenda, 2002).

2.4.3 Granularity of DWM

Granularity (granular composition) characterizes the quantitative representation of individual particles, or group of particles of specific size, of the bulk material. Bulk material is a system (a set of particles with varying size) that can be sorted into classes depending on the size of its fractions. Table 2.2 displays classes according to STN 260070 and their corresponding fraction size.

Table 2.2: Classes of bulk material granularity according to STN 260070.

Class	Granularity (mm)	Definition
Very fine	0.07 – 0.50	A
Fine	0.50 – 3.50	B
Fine grain	3.50 – 13.0	C
Medium grain	13.0 – 75.0	D ₁
Coarse grain	over 75.0	D ₂
Irregular	fibrous, diverse, irregular size	E

In Hejma& et al.(1981), a different granular classification is given. According to this source, loose bulk wood materials are categorized as thick, medium thick, medium, fine, and very fine. Granularity of fractions can be seen in Table 2.3.

STN 260070 also states that dust is a fine fraction of bulk material including wood fractions from 0.1 μm to 1000 μm . According to this standard, dust is classified by:

- very fine dust with particles from 0.1 μm to 10 μm ,
- fine dust with particles from 10 μm to 100 μm ,
- thick dust with particles from 100 μm to 1000 μm .

Table 2.3: Granularity of bulk wood materials (Hejma& et al., 1981).

Granularity labeling	Fractions dimension (mm)
Coarse	$a > 1$
Medium coarse	0.5 – 1
Medium	0.3 – 0.5
Fine	0.03 – 0.3
Very fine	0.001 – 0.03

The properties of wood dust with a particle size of 10 μm to 100 μm differs from other dust categories in that it has very slow sedimentation, remains airborne for a long time, is explosive and conductive, and tends to form clusters in humid conditions.

The most common method for defining the granular composition of bulk materials is to run them through screen “sieving,” or a system of successive screens graduating with finer mesh sizes. There exist analytical methods for determining granular composition, including, for example, blowing or microscopic analysis. However, these methods tend to be slower and more expensive; therefore, the sieving method according to standards in Holm & et al. (2008) is the favoured method.



Figure 2.11: Sieving devices, different mesh types, and configurations (square or circular mesh) (Fischer; Retsch).

2.4.4 Moisture Content of DWM

Moisture of wood material is determined through the weight of water m_v within its volume. It is usually expressed in the form of absolute humidity or relative humidity of the wood material (STN EN 844-4).

When we discuss absolute humidity, we are defining the weight of water as a ratio to the weight of absolutely dry wood. When we define the weight of water as a ratio to the weight of wet wood, we are referring to the relative humidity. Absolute and relative wood humidity is expressed as a percentage % and is calculated through the following relations (STN 490103):

$$w_a = \frac{m_v}{m_0} \cdot 100 = \frac{m_w - m_0}{m_0} \cdot 100 \text{ (%)}$$
 (2.3)

$$w_r = \frac{m_v}{m_w} \cdot 100 = \frac{m_w - m_0}{m_w} \cdot 100 \text{ (%)}$$
 (2.4)

$$m_w = m_0 + m_v \text{ (kg)}$$
 (2.5)

where:

w_a – absolute humidity

w_r – relative humidity

m_0 – weight of absolutely dry wood (weight of wood after drying at $103 \pm 2^\circ\text{C}$)

m_w – weight of wood at a humidity of “w”

m_v – weight of water in the wood

The conversion of relative wood humidity to absolute, and vice versa, is possible using these equations (STN 490103):

$$w_a = \frac{100 \cdot w_r}{100 - w_r} \text{ (%)}$$
 (2.6)

$$w_r = \frac{100 \cdot w_a}{100 + w_a} \text{ (%)}$$
 (2.7)

Relationship between absolute and relative humidity in wood is illustrated in Fig. 2.12.

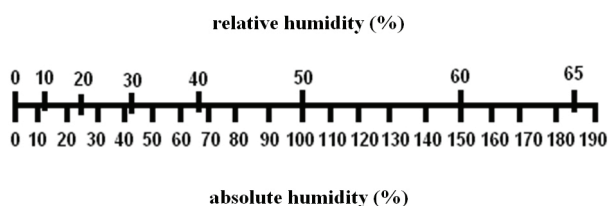


Figure 2.12: Nomogram for the mutual conversion between absolute and relative humidity in wood (Požgaj & et al., 1997).

The mechanisms holding moisture within wood are explained in STN 490103 and Požgaj & et al.(1997), which discuss the chemical-bonded water, free water, and bound water.

Chemically-bonded water is a result of chemical compounds. They cannot be eliminated by drying, only by combustion. For this reason, it is included in the absolute dry state of the wood. Its content is determined through chemical analysis of wood, and its overall content represents about 1-2 % of the dry wood matter. It has no effect on the physical/ mechanical characteristics.

Free water, known also as capillary water, mostly fills the lumen of the wood or sometimes the free space between cells in which it is drawn by capillary behaviour. It is only present in the wood when the cell walls are filled with bonded water. Its quantity ranges from the point of fibre saturation (PFS) to completely saturated wood with water. Its maximum quantity can exceed 100 % w_a . Its content depends mainly on the volume of lumen cells and, to a smaller degree, the intercellular spaces in the wood matter. Although it has some effect on the physical/mechanical characteristics of the wood, it is not nearly as influential as bound water.

Bound water, known also as hygroscopic water, is located in the cell walls and is bonded by hydrogen in the OH hydroxide group of the amorphous part of the cell and hemicellulose. The water bound within wood has moisture content from 0 % to PFS, whose average humidity value for the wood used in this work was 30% w_r (Požgaj& et al., 1997). PFS is defined as a wood state consisting of bound water with the cell walls saturated such that the free spaces in the cell are filled with water. The moisture content in the wood used in this paper was 22 to 35 % w_r at the PFS (Požgaj & et al., 1997). It depends mainly on the species of wood, methods of extraction, and other effects. Bound water has the greatest, and therefore most important, effect on the physical/mechanical characteristics of the wood matter. The boundary between bound water and free moisture is determined through the limit of cell wall saturation (LCWS) or hygroscopic limit (HL). Such a state is defined when the cell walls are fully saturated while the lumen does not consist of any free water. The difference between LCWS and HL is defined by the environment that the wood is in. LCWS refers to water in a liquid state, whereas the water in the HL is in a gaseous state. At 15 – 20°C,

both variables have approximately the same moisture content (about 30 % w_r), but unlike LCWS, HL is dependent on the surrounding temperature and with growing temperature decreases (Požgaj & et al., 1997).

In practice, the degree of moisture in the wood is distinguished by (Požgaj & et al., 1997):

1. Wet, long-term exposure to water (over 100% w_r);
2. Freshly-cut (50 to 100 % w_r), in some cases up to 180 % w_r depending on wood species
3. Air dry (15 to 20% w_r)
4. Room dry (8 to 10 % w_r)
5. Absolutely dry, dried at 103 ± 2 °C

Wood saturation is dependent on the moisture level of the wood. It is the ability for the wood, thanks to its porous structure, to draw in liquid water. The amount of free water is dependent mainly on the volume of the pores within the wood. The saturation rate of the core is slower than for the bark (Fig. 1.1). With increasing density, saturation rate of wood decreases. Higher temperatures accelerate the saturation rate of the wood. Moisture, as a contributing factor to the densification process, will be given further attention when we discuss parameters affecting the quality of briquette.

2.4.5 Bulk Density of DWM

In discussing the particulate matter of wood, it is necessary to recognize one property of its bulk, which is its angle of repose. The angle of repose is a characteristic element that defines the properties of the particulate matter. Its range of values can be, theoretically, from zero to ninety degrees. The closer its value is to zero, the less cohesive the particulate matter is and vice versa.

However, in our case (pressing of DWM) the bulk density is more important. The bulk density is defined as the ratio of particle mass m to its volume V_s (Dzurenda, 2002).

$$\rho_s = \frac{m}{V_s} \text{ (kg.m}^{-3}\text{)} \quad (2.8)$$

For the measurement of bulk density, as well as the angle of repose for that matter, we can use Gary's apparatus (Fig. 2.13). The test is very simple. A 1000ml chute with open bottom fills a sample container 350mm below the chute by means of free falling particles and gravity. After the sample is completely filled, the top is levelled and the specimen is then weighed. With known volume and weight of the specimen, we can use Equation 2.8 to determine the bulk density of the wood matter.



Figure 2.13: Gary's apparatus for measuring bulk density of particulate matter (Dzurenda, 2002).

Testing for the angle of repose utilizes the same apparatus, except the sample container is removed and the particles are allowed to drop freely onto the flat surface below, creating a cone-like shape. After pouring, the height and diameter of the poured bulk are taken. Cohesive substances have a higher angle of repose, while non-cohesive substances have a lower angle. The lower the angle, the easier the material will pour allowing for reliable dosages of material. However, a low angle of repose represents materials that are less cohesive, requiring more area needed for their storage, which can be problematic.

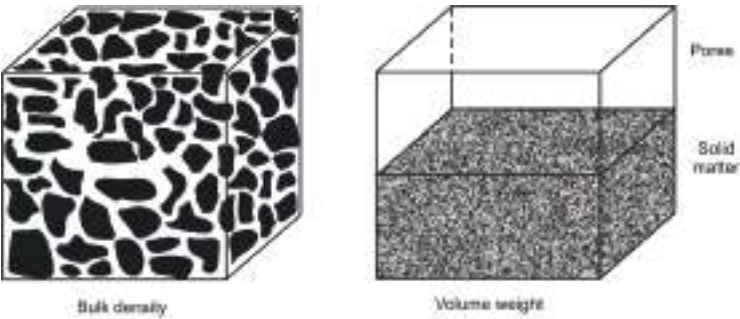


Figure 2.14: Illustrative representation between bulk density and volume weight (Simanov, 1995).

Bulk density is dependent on the properties of the bulk material (wood species, granularity, moisture content). The approximate dependence between wood density and bulk density of DWM can be expressed by:

$$\rho_s = (0,3 \div 0,5) \cdot \rho \text{ (kg.m}^{-3}\text{)} \quad (2.9)$$

Table 2.4: Bulk density of some wood materials with a moisture content of $w_r=10\%$ (Dzurenda, 2002).

DWM	Bulk Density (kg.m ³)
Spruce shavings	80 – 124
Spruce sawdust	113 – 166
Beech sawdust	195 – 218
Wood chips	238 – 259
Dust from sanding	256 – 279

2.4.6 Spacing and Porosity of DWM

Gaps between wood particulate matter in contact with each other in bulk can be represented as pores. Since solid particles consist of surface pores and internal pores, porosity of the bulk material represents the volume of these pores or volume around the particles.

These parameters are not only important for particulate matter (represented by a volume that can be filled with a liquid phase) but also for storage, transportation, burning processes, and automated incineration. Spatial porosity impacts effective storage, packaging, transport, and manipulation of particles with varying sizes.

Spacing effects (Bejda & Miklušová, 2002):

- Shape of the particle – for example, a cubic configuration has a spacing of $\varepsilon = 0$, whereas a spherical particle will never obtain zero spacing.
- Fraction composition – compacting biofuels is monodisperse (same size and identical shape. Spacing larger than polydisperse substances, which fill their spaces with dust.
- Particle organization – spherical particles of identical diameter have six configurations and six different values of ε . The spacing depends also on the number of contacts between particles. At the walls, spacing is greatest.

Calculating spacing can be done using Equation 2.10, where V_e [mm³] is the volume of the spaces, and V_t [mm³] is the overall volume:

$$\varepsilon = \frac{V_e}{V_e + V_t} \quad (2.10)$$

Spacing is almost impossible to determine for bulk materials in, for example, a sac or container. It is very difficult and work-intensive to determine the orientation of each individual briquette in space. Problems are thus necessary to simplify and place objects into bricks whose dimensions represent the maximum dimensions in space. Significant inconsistencies in particle shapes can also greatly affect filling of the spaces.

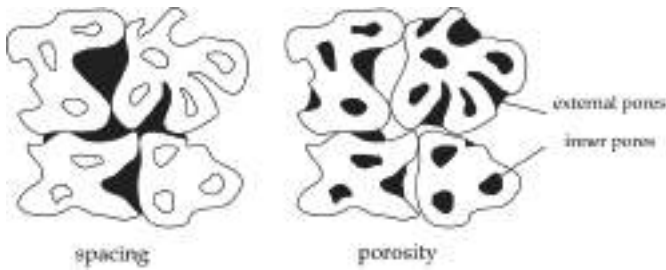


Figure 2.15: Illustrative representation of spacing and porosity (Pietsch, 1991).

2.4.7 Compressibility of DWM

The compressibility of DWM is defined as the ability for the bulk material to change its volume with respect to an externally applied force. It is characterized by the coefficient of compressibility β_s , which gives the relative change in volume for the unit change in applied pressure.

$$\beta_s = \frac{1}{V_o} * \frac{dV}{dp} (\text{m}^2 \cdot \text{N}^{-1}) \quad (2.11)$$

Compressibility is one property that, in pressing, affects the process. Compressing DWM densities decreases the size and porosity of the bulk material while the volumetric weight increases through the application of external force. The simplest way to evaluate the compressibility of a particulate matter is by uniaxial compression (pressing). A specimen is prepared with a so-called oedometer. The sample is pressed by a force and the corresponding compressibility curve, which describes the dependence of compression for of the pressed sample, is obtained.

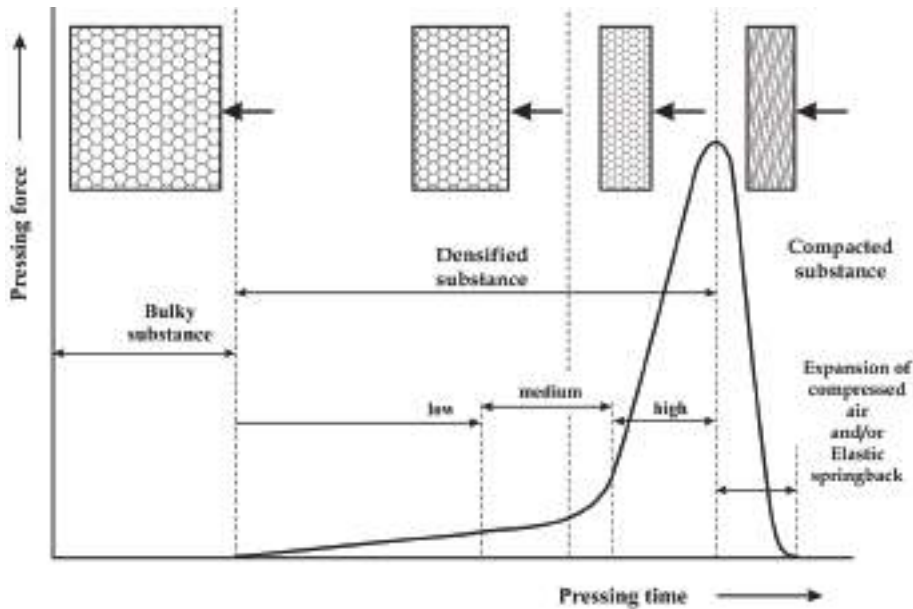


Figure 2.16: Change in wood mass depending on force applied (Pietsch, 2002).

Under the applied pressing pressure, solid particles of the material are pushed closer to each other until they create a strong adhesive bond. In the upper part of the previous figure, four sketches representing the change in structure of a bulk material during compaction are shown. The accompanying effects of applying high pressure to the particles are a change in the shape and size of the particles and therefore a change in the overall volume. In the bottom part of the previous figure, the growth of pressing force with respect to time assumes that the movement of the piston is constant until the maximum value. After obtaining the maximum value, the movement of the piston is in the opposite direction and also constant. In the first step of densification, the particles within the material regroup and, due to lower forces, do not change shape and size of the particles. In the second step, where the pressing force begins to rise, fragile particles break and elastic particles deform (sketch 3 and 4). It is necessary to consider two interesting properties which affect and limit the densification process as well as the performance of the pressing machine. Pressing excess air within the pores and elastic deformation affect the densification and compressibility of the particles. They cause specific cracks and weaknesses in the briquette which can lead to breakage of the briquette. The gradual increase in pressing pressure changes the size of the particles until deformation, reducing porousness. The degree to which the particles deform is given by the compressibility of individual types of bulk wood materials. Compressibility is one of the properties which, when pressing, affects the whole process. The maximum pressing force is defined by the structural and

kinematic properties of the pressing machine, and therefore it is necessary to design a densification process for wood with respect to the machine's capabilities.

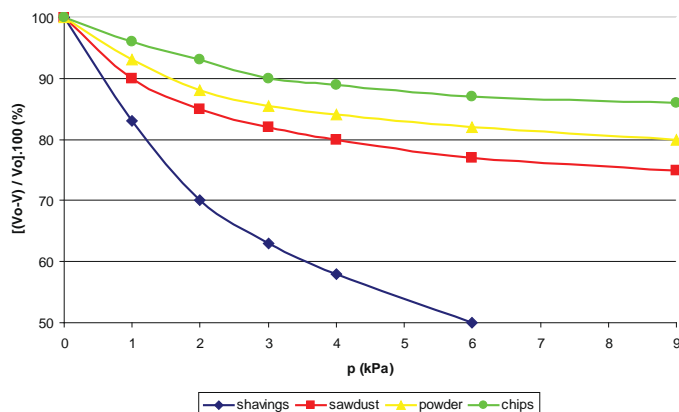


Figure 2.17: Compressibility of some types of wood materials (shavings, dry saw dust, wood powder, chips) (Dzurenda, 2002).

On the previous figure, we can see the differences in compressibility for various types of bulk wood materials. The compressibility coefficient of wood matter consisting of isometric particles have values of $\beta_s=0.75-0.85$ and $\beta_s=0.50$ for wood shavings. The compressibility of chips, wood dust, dry sawdust, and shavings with respect to pressure can be seen in Fig. 2.17.

2.5 Evaluating the Quality of the Briquettes with Respect to Standards

As for any area of research, densification of wood biomass into a biofuel requires us to define the quality of production. Solid biofuels are characterised by a wide range of quality, defining their physical and chemical composition, such as the size of the particles, moisture content, heating potential, or the sulfur, chlorine, nitrogen, and potassium content. It is therefore very important to define the properties and standardize parameters of solid biofuels (ECS, 2013a; 2013b). Standardization of solid biofuels is a very important and necessary prerequisite to expand the market for solid biofuels. Stating some standards for solid biofuels can contribute to simplifying the offering of biofuel, its conversion into energy, establish biofuel in the market, and reach an economical understanding for price of fuel. Furthermore, standardization can guarantee that each fuel fulfills the demands for combustion and therefor guarantees

the environmental cycle minimizes emissions of dangerous substances into the atmosphere and environment. It also defines the connection between production and operational machines for energy conversion (furnaces, etc.). Standardization helps reduce potential expenses in the system and thus minimizes the overall investment into such systems.

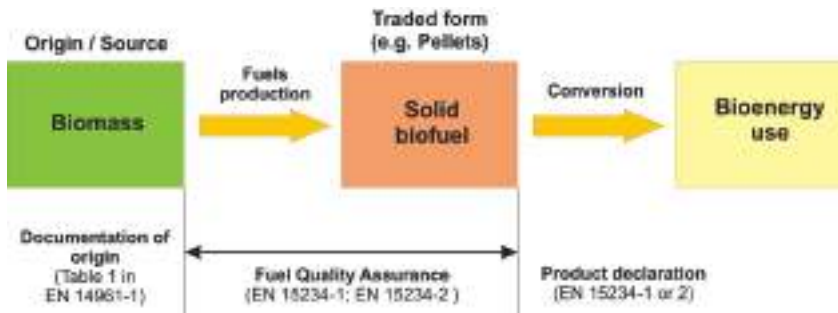


Figure 2.18: Effects of Technical Standards in the production of solid biofuels (STN EN 14588).

Currently, many Standards exist for solid fuels. Legislation, manuals, and technical standards define the requirements for the production, energy uses, and markets for solid biofuels. They are necessary for sellers as they objectively clarify the rules and simplify the quality requirements in the market. They also help create markets by making all relevant information readily available. Control and interpretation is based on quality requirements, which also increase the competition of this fuel and categorizes the quality of the fuel, storage, and supply (Šooš & et al., 2010).

It is important to comment that the European Union Standards are defined for solid biofuels under CEN 14588 and CEN 14961, for example. These standards define biofuels, raw materials for production, indicators of quality, procedures for determining properties of the biofuel, and the rating system for the final product quality. In more developed countries (countries with a higher production of biomass), other standards existed even before those put in place by the EU. These national standards were the basis for creating the newly-formed EU Standards. In Slovakia, the market for biofuels is gradually increasing. It is therefore necessary to be up to date on the current European Union Standards. Each valid EU Standard must be implemented into Slovakian Standards. Presently, biofuel production in Slovakia, as well as abroad, adhere to current legislation; however, some initial technical standards of some EU states are still used. Therefore, current as well as past EU Standards will be discussed.

2.5.1 Dividing of Briquettes According to Standards of Some EU Countries

Before the development and the acceptance of current EU legislation (up to 2005), most standards were defined at a national level for countries such as Austria, Germany, and Sweden. Adapting these standards was easy for other EU countries. A briquette/pellet as a final product from the densification process, further referred to as a biofuel, must satisfy some specific conditions stated in the Technical Standards. In Europe, Germany, Austria, Sweden, and Italy, Standards exist for the production of special solid biofuels. More Standards exist to supplement these basic Standards.

In **Austria**, a national Standard exists defining the quality of the production, storage, and transport of wood pellets and briquettes, more specifically known in Ö-Norm M 7135:

- ÖNORM M 7135 – briquettes from natural wood, pellets, and briquettes; requirements and legislation for testing;
- ÖNORM M 7136 – briquettes from natural wood and wood pellets; ensuring quality in the transport and logistics to the end user;
- ÖNORM M 7137 – Briquettes from natural wood, wood pellets; conditions for the storage of pellets at the end users facilities;

Certificates for solid high-grade biofuels in **Germany** were established on the DIN 51731 and DIN Plus Standards (DIN 51731; DIN Plus). The certification is based on initial evaluation and conformity, such as routine monitoring. Monitoring is restricted to the yearly repetition of tested products in a laboratory certified by DIN CERTCO (German Institute for Standards).

- DIN 51731 – Testing of solid fuels; briquettes from natural wood; requirements and testing;
- DIN Plus – certificate scheme; wood pellets for use in small furnaces;
- DIN 51731 defined the term “*briquette*” as a fuel artificially created by pressing bulk materials without any added substances and suitable for burning.

The basic dimensions of a briquette or pellet according to DIN 51731 as well as Ö-Norm M 7135, are given in Tab. 2.5. The limit density of pellets and briquettes is $1 - 1.4 \text{ kg.dm}^{-3}$, with a moisture content below 12%, ash content below 1.5%, and heating energy from 17.5 to 19.5 MJ.kg⁻¹. Further addressed in the Standards are the bulk density, abrasiveness, chemical limits, trace elements, and emissions. At the present, they are produced exclusively for energy (Fig. 2.19 and 2.20). Standards state that extrusions up to 25mm in diameter are considered pellets, and anything larger is considered a briquette. Briquettes are formed into cylinders, blocks, and parallelograms. They are typically used in larger automatic furnace systems or home furnaces and fireplaces without any atomization.

Table 2.5: Dimensions of wood pellets/briquettes (HP) and from cortex wood (RP) produced in line with DIN 51731 and Ö-Norm M 7135.

Standard	Size group	Diameter D_N , height or thickness (mm)	Length (mm)
DIN 51731	HP 1	> 100	> 300
	HP 2	60 to 100	150 to 300
	HP 3	30 to 70	100 to 160
	HP 4	10 to 40	< 100
	HP 5	4 to 10	< 50
Ö-Norm M 7135	HP 1 (pellets)	$4 \leq D_N < 10$	$\leq 5 \times D_N$
	HP 2	$10 \leq D_N < 40$	$\leq 4 \times D_N$
	HP 3 (briquettes)	$40 \leq D_N < 120$	≤ 400
	RP 1 (pellets)	$4 \leq D_N < 10$	$\leq 5 \times D_N$
	RP 2	$10 \leq D_N < 40$	$\leq 4 \times D_N$
	RP 3 (briquettes)	$40 \leq D_N < 120$	≤ 400



Figure 2.19: Examples of varying shapes of briquettes from wood.



Figure 2.20: Examples of pellets from wood.

The research in this work will focus on the production of briquettes up to 20 mm in diameter. The measurement stand is described in detail throughout this monograph. Briquettes can be created not only from wood but other types of materials such as phytomass (Fig. 2.21).



Figure 2.21: Briquettes from various types of materials (Melcer & Melcerová, 1985).

In **Sweden**, a wide variety of Standards that define the qualitative parameters of briquettes and pellets, evaluate quality, and specify testing methods to determine various parameters have been adopted. Standards for briquettes describe three classes, which differ mainly in the size and ash content. Some basic technical standards from Sweden are:

- SS 187106 - Solid biofuels and peat – Terminology;
- SS 187120 - Solid biofuels and peat – Pellets – Classifications;
- SS 187123 - Solid biofuels and peat – Briquettes - Classification;

In **Switzerland**, the qualitative properties of solid biofuels were controlled according to Swiss National Standards SN 166 000 (Testing of solid fuels – compressed untreated wood, requirements, and testing), which was identical to the German DIN 51731 standard.

In **Great Britain**, briquettes were introduced through a project organized by the Department of Trade and Industry (DTI). One of the results of this project was the categorization of possible uses of briquettes and furnaces utilizing them for homes (up to 15kW). This category was developed to the extent of measurement and evaluation of biofuel parameters at the national level.

Similarly, **Italian** Standards include CTI-R04/5 (Caratterizzazione del pellet a fini energetici: quality parameters of briquettes for energy); CTI-R03/1 (Biocombustibili: specifiche e classificazione: biofuel – specification and classification); UNI/TS 11263 (Technical specification for the characterisation of briquettes for energy). **France** also had its own standards, including NF label (NF Granules Biocombustibles: French standard for granular biofuels). In **Norway**, there is the NS 3165 (Biofuel – Cylindrical pellets of pure wood – Classification and requirements. In **Russia**, there is the GOST 23246 – 78 (Crushed wood: Terms and definitions). Additionally, **Finland** has SFS 5875 (Solid recovered fuel: Quality control system).

Even though **Denmark** is a large user of pellets, there has never been any national Standard implemented for their use. Only the “Legislation for biomass” (BEK no.638 from 03.07.1997), in which it is only stated that briquettes must be made purely of wood with no additional substances, existed.

Other European countries (Estonia, Latvia, Greece, Cyprus, Poland, Portugal, Spain, Malta, Holland, Luxemburg, Belgium, Ireland, Hungary, Slovakia, Czech Republic, Slovenia, and Bulgaria) have never implemented any national level Standards for biofuels mainly because:

- the biomass market (mainly in briquettes) in these countries was very young;
- these countries waited for legislative implementations from the EU.

2.5.2 European Technical Standards for Briquettes Quality Evaluating

STN CEN 15357 applicable also in Slovakia, defines the following basic terms:

- “*biofuel*” – a fuel made, directly or indirectly, from biomass.
- “*briquette*” – a block or cylinder from solid alternative fuel made by the agglomeration of bulk material.
- “*pellet*” – a piece of solid alternative fuel made from agglomerated bulk material in a pressing form (disk or drum) with a diameter equivalent to < 25 mm.

The now valid standard in Slovakia defined by STN CEN 14588 defines these basic terms:

- “*solid biofuel*” – a solid fuel made, directly or indirectly, from biomass.
- “*biofuel briquette*” – compressed biofuel made with or without additives in the pressing process in the form of cubes or cylinders, obtained through pressing biomass powder. Raw materials for briquettes can be wood biomass, herbal biomass, fruit biomass, and other types of biomass.
- “*biomass pellets*” – compressed biofuel made from powder biomass with or without pressing additives most commonly in cylindrical form of random length typically from 5 to 30 mm with broken ends.

Technical standardisation of solid biofuels is administered by the European committee for standardisation CEN/TC 335 (ECS, 2013a; 2013b). This committee was formed in 2001 and since then has gradually prepared new standards for the area of solid biofuels. The European committee for CEN standards has prepared 38 published Standards for solid biofuels (CEN-TC 335). These Technical Standards are proceeded by all existing national Standards of European countries and member states of the EU, which must be implemented into each state's national Standards.

In Figure 2.22 and 2.23, valid EN Standards for solid biofuels regarding the size of briquettes and pellets are shown. In general, an extrusion with a diameter of $D < 25$ mm is known as a pellet while briquettes are defines as anything larger. Further size and shapes of the briquette are defined within the six-part set of Standards STN EN 14961: Solid biofuel, specifications, and class.

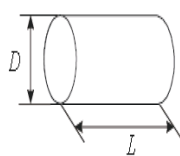
Origin: According to 6.1 and Table 1		Woody biomass (1); Herbaceous biomass (2); Fruit biomass (3); Blends and mixtures (4).
Traded Form (see Table 2):		Pellets Diameter (D) and Length (L)
Dimensions (mm)		 <p>The diagram shows a 3D perspective of a cylinder representing a pellet. A vertical double-headed arrow on the left side indicates the diameter, labeled 'D'. A horizontal double-headed arrow along the bottom edge indicates the length, labeled 'L'.</p>
D 06	6 mm ± 1.0 mm, and 3.15 ≤ L ≤ 40 mm	
D 08	8 mm ± 1.0 mm, and 3.15 ≤ L ≤ 40 mm	
D 10	10 mm ± 1.0 mm, and 3.15 ≤ L ≤ 40 mm	
D 12	12 mm ± 1.0 mm, and 3.15 ≤ L ≤ 50 mm	
D 25	25 mm ± 1.0 mm, and 10 ≤ L ≤ 50 mm	Figure 3: Dimensions (mm).

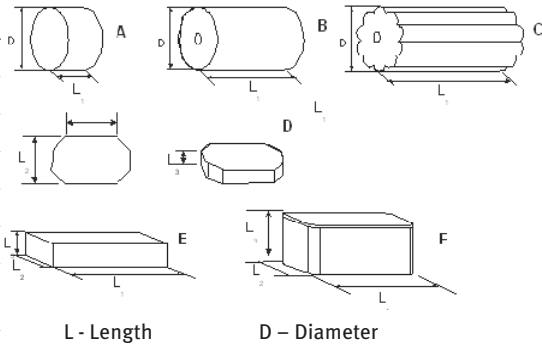
Figure 2.22: Specifications for pellet properties – excerpt from the STN EN 14961-2 Standard.

These Slovak Standards are based on existing European Technical Standards. At present, most European Standards for solid biofuels have been adapted. In the next table, a comparison of briquette parameters, which are published in some national and EU Standards, is shown.

2.5.3 Indicators of Quality and Evaluation of Briquettes with Respect to Standards

Solid, high-grade biofuels are considered high quality if they fulfill the requirements defined within Technical Standards. Technical Standards define multiple parameters (indicators), which are used to evaluate and monitor the quality solid, high-grade

Origin: According to 6.1 and Table 1	Woody biomass (1); Herbaceous biomass (2); Fruit biomass (3); Blends and mixtures (4).
Traded Form (see Table 2):	Briquette
Dimensions (mm)	
Diameter (D) or equivalent (diagonal or cross-sectional), (mm)	
D 40	$25 \leq D \leq 40$
D 50	≤ 50
D 60	≤ 60
D 80	≤ 80
D 100	≤ 100
D 125	≤ 125
D 125+	> 125 (should be given max. value)
Length (L), (mm)	
L 50	≤ 50
L 100	≤ 100
L 200	≤ 200
L 300	≤ 300
L 400	≤ 400
L 400+	> 400 (should be given maximal value)



L - Length

D - Diameter

Figure 2: Examples of briquettes.

Figure 2: Examples of briquettes.

Figure 2.23: Specifications for briquette properties – excerpt from the STN EN 14961-3 Standard.

biofuel (SHGB). Each monitored parameter/indicator of quality is precisely described, with a procedure for its identification, and for each parameter the precise limit for its value is defined. If a briquette falls within these limits, it can be considered a quality product. However, this limit for quality is valid only in cases where the solid biofuel satisfies all other monitored quality indicators (mechanical and chemical/thermal).

Quality indicators of solid biofuels can be divided into two basic groups (Lehtikangas, 2001; Rönnbäck, Sager & Alakangas, 2012):

- physical-mechanical indicators
- chemical-thermal indicators.

Table 2.6: Comparison of some briquette parameters specified in national and European Standards.








Standard	O-NORM M7135	SS 187120	DIN 51731 / DIN Plus	EN 14961-2	CTI	- R	04/5	British	BioGen / UK
Parameters of pellets									
Fuel specification	Pellets	Pellets	Pellets	Nonchemical treated wood without bark	Group and A®	according to use A®	to the of B C	source additives	Premium Alternate pellets
Diameter (mm)	from wood 4 to 10	from wood group 1 up to 4	from wood group 2 up to 6	D06 to 6	6, 8	6, 8	6, 8	10-	< 4 – 20 > 10 - > 20
Length (mm)	up to 100	-	-	D08 to 8	-	-	-	25	-
Density (kg/dm³)	≥ 1,0	-	< 50 / < 5 x D _N from 1,0 to 1.4 / > 1.12	D06 ≤ 5 x d D08 ≤ 4 x d	-	-	-	-	> 40 lbs/ft² (0.527 kg/dm³)
Moisture content (%)	≤ 12	≤ 10	≤ 12	≤ 10	≤ 10	≤ 10	≤ 10	≤ 15	≤ 10
Bulk density (kg/m³)	-	≥ 600	≥ 500	recommended to find volume	620-720	620-720	620-720	≥ 550	> 600 > 500
Heating potential (MJ/kg)	≥ 18.0	≥ 16.9	17.5 – 19.5 / 18.0	≥ 16.9	≥ 16.9	≥ 16.9	≥ 162	yet to be defined	> 4.7 > 4.2 kWh/kg
Abrasion (%)	< 2.3	-	- / < 2.3	-	-	-	-	-	-
Ash content (%)	≤ 0.5	≤ 6.0	≤ 1.5	≤ 0.7	≤ 0.7	≤ 0.7	≤ 1.5	yet to be defined	< 1, < 3 or 6

Table 2.6: Comparison of some briquette parameters specified in national and European Standards.

Continued















Standard	O-NORM M7135	SS 187120	DIN 51731 / DIN Plus	EN 14961-2	CTI	- R	04/5	British	BioGen / UK
Parameters of pellets									
Mechanical durability (%)	-	-	- / -	≥ 97.5	≥ 97.7	≥ 97.7	≥ 95.0		
Additives (%)	up to 2	yet to be defined	yet to be defined	up to 2 **	Any	yet to be defined	yet to be defined		
Dust content (%)	-	≤ 0.8	≤ 1.5	≤ 1.5	-	-	-	< 0.5	< 0.5
Sulphur content (%)	≤ 0.04	≤ 0.08	≤ 0.08 / ≤ 0.04	≤ 0.05	≤ 0.05	≤ 0.05	≤ 0.05	< 300 ppm	< 300 ppm
Nitrogen content (%)	≤ 0.3	-	≤ 0.3 / ≤ 0.3	N0.3 ≤ 0.3 N0.5 ≤ 0.5 N1.0 ≤ 1.0 N3.0 ≤ 3.0 N3.0+ > 3.0 CL0.03 ≤ 0.03	≤ 0.3	≤ 0.3	≤ 0.3	defined	defined
Chlorine content (%)	≤ 0.02	≤ 0.03	≤ 0.03 / ≤ 0.02	CL0.07 ≤ 0.07 CL0.10 ≤ 0.10	≤ 0.03	≤ 0.03	yet to be defined	< 800 ppm	< 800 ppm
Arsenic content (mg/kg)	-	-	< 0.8 / 0.8	-	-	-	-	-	-

Table 2.6: Comparison of some briquette parameters specified in national and European Standards.

Continued

Standard	O-NORM M7135	SS 187120	DIN 51731 / DIN Plus	EN 14961-2	CTI	- R	04/5	British	BioGen / UK
Parameters of pellets									
Lead (mg/kg)	-	-	< 10 / < 10	-	-	-	-		
Cadmium (mg/kg)	-	-	< 0.5 / < 0.5	-	-	-	-		
Chromium (mg/kg)	-	-	< 8 / < 8	-	-	-	-		
Copper (mg/kg)	-	-	< 5 / < 5	-	-	-	-		
Silver (mg/kg)	-	-	< 0.05 / < 0.05	-	-	-	-		
Zink (mg/kg)	-	-	< 100 / < 100	-	-	-	-		
Halogens (mg/kg)	-	-	< 3 / < 3	-	≤ 1 %	≤ 1 %	≤ 1 %	yet to be defined	

* Chemically unmodified product from agricultural and forestry (for example: whole wheat corn flour, corn starch, and rye flour) can be mixed with the base material in the production of briquettes to increase energy and improving production and abrasion resistance.

** Biomass products only from agricultural and forestry, which is not chemically treated. Types and additives yet to be defined.

◉ Without additives

◉◉ With additives

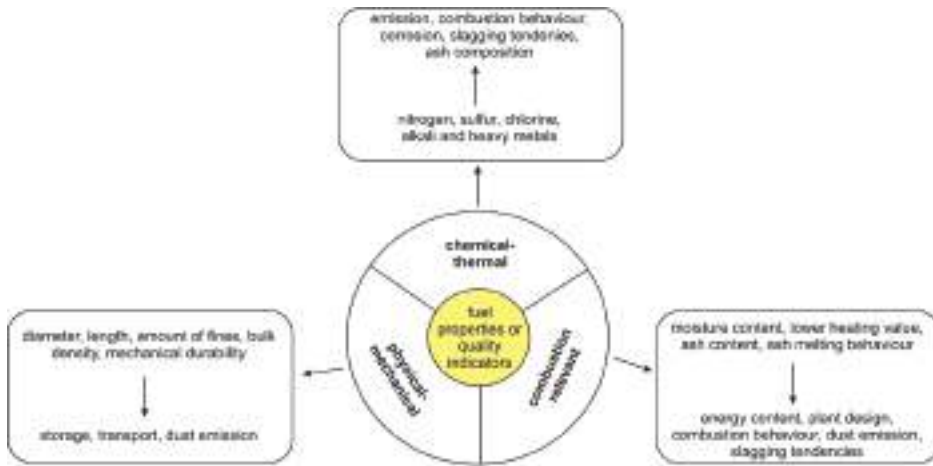


Figure 2.24: Property effects of fuel in the combustion process (EN Plus Handbook, 2013).

The growth phase and origin of the biomass affect mainly the chemical-thermal properties, whereas the material processing phase most importantly affects the mechanical properties of the final product.

Before the implementation of European Standards (EN 14961) for the evaluation of SHGB, every producer and seller within the EU focused on achieving the previously mentioned Austrian (Ö-NORM M 7135) and German (DIN 51731 a DIN Plus) Standards. These Standards defined, in more detail, requirements for production and indicators of briquette quality.

ÖNORM M 7135:2000 Compressed wood or compressed bark in natural state--pellets and briquettes-- requirements and test specifications. Vienna, Austria: Österreichisches Normungsinstitut

Description: compressed wood in its natural state – pellets and briquettes – requirements and specification of tests. The implementation of this Standard prompted the wood-working industry. It is a unified standard for pellets and briquettes. This Standard restricts use and defines basic terms and requirements for extrusions and described testing methods.

DIN 51731:1996 Testing of solid fuels – compressed untreated wood, requirements, and testing. Berlin, Germany: Deutsches Institut für Normung

Description: solid fuel testing – pressing wood, requirements, and testing. This Standard contains the definition for wood extrusions, their labelling, dimensions, allowable emissions, and testing procedures. This Standard divides briquettes from wood waste into five size groups.

DIN Plus:2002 Certification Scheme – Wood pellets for use in small furnaces.
Berlin, Germany: DIN CERTCO-Gesellschaft für KonformitätsbewertungmbH

Description: certification scheme – wood briquettes/pellets for use in small heating systems. This Standard presents a new certification scheme and combines the requirements according to the German DIN 51731 and Austrian ÖNORM M 7135 Standards. The requirements in relation to maximum property values of some products change compared to the certification of wood briquettes/pellets according to the DIN testing scheme.

The sixth chapter in ÖNORM M 7135, seventh chapter in DIN 51731 and part 3.11 of DIN Plus are dedicated to pellets. Therefore, we will further discuss these chapters. The detailed description for quality assurance in the areas of logistics, transport, and storage is written in ÖNORM M 7136. The storage requirements at the user facility is described in ÖNORM M 7137.

As discussed above, we can divide the quality indicators of the briquettes into two categories. The first concerns the chemical-thermal indicators of quality. Select Technical Standards of some European countries include the ash content, heating value, moisture content, and chemical and trace elements. The second category consists of so-called mechanical indicators of quality including parameters such as briquette density and abrasion resistance. The first category is essentially dependent on the type of compressed material (its properties and chemical composition). The second category depends on several parameters, which also include the experimental research described in this work. These parameters are mentioned in further chapters. Mechanical indicators are very important and decisive to this research, and therefore their descriptions will be discussed.

Currently, the most important Standard for solid biofuel is the EN 14961 (in Slovakia, STN EN 14961 since 2010): Solid biofuels, specifications and categories of fuel, consisting of the following parts:

- Part 1: General requirements (EN 14961-1)
- Part 2: Wood pellets for industrial use (EN 14961-2)
- Part 3: Wood briquettes for non-industrial use (EN 14961-3)
- Part 4: Wood chips for non-industrial use (EN 14961-4)
- Part 5: Wood for burning in non-industrial uses (EN 14961-5)
- Part 6: Non-wood pellets for non-industrial use (EN 14961-6)

The goal of these European Standards is to provide definite and clear principles for the classification of solid biofuels, and these standards currently serve as a tool allowing the effective trade of biofuel because the conditions for quality are clear between the seller and buyer. This European Standard is prepared for all groups of users. The subject of this Technical Standard is the specification of categories for quality. The fundamental goal is to describe solid biofuels according to (STN EN 14961-1):

- origin and source
- main forms of trade

Solid biofuels are traded in various shapes and sizes. The size and shape affect the manipulation of the fuel as well as the combustion process.

STN EN 14961 includes the physical-mechanical indicators of quality as:

1. Bulk density (STN EN 15103)
2. Particle size distribution (STN EN 15149)
3. Particle density (STN EN 15150)
4. Mechanical durability (STN EN 15210)
5. Length and diameter of the briquette (STN EN 16127)

The same standard defines the chemical-thermal indicators of quality as:

1. Moisture content (STN EN 14774)
2. Ash content (STN EN 14775)
3. Net caloric value (STN EN 14918)
4. Carbon, hydrogen, and nitrogen content (STN EN 15104)
5. Chloride, sodium, and potassium content dissolved in water (STN EN 15105)
6. Sulfur and chlorine content (STN EN 15289)
7. Volatility dust matter (STN EN 15148)
8. Content of primary elements (STN EN 15290)
9. Content of secondary elements (STN EN 15297)

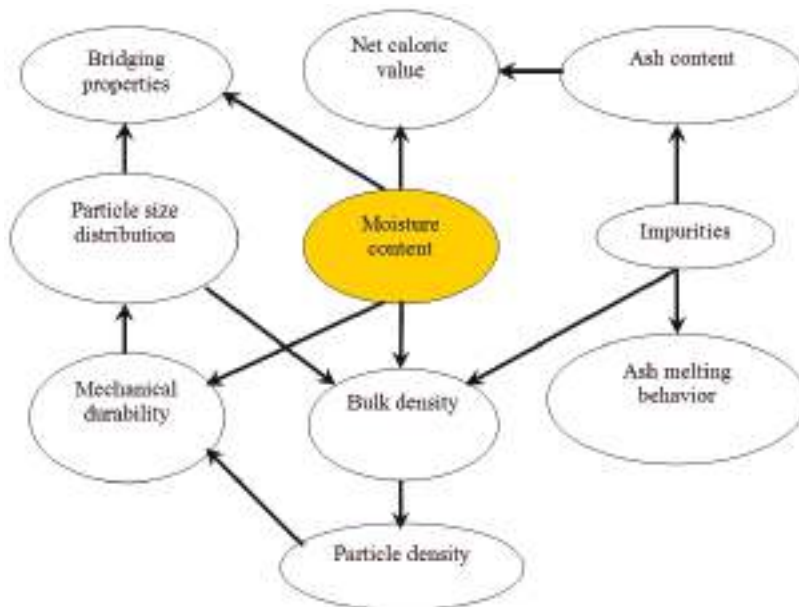


Figure 2.25: Dependence between the mechanical and chemical-thermal properties (Rönnbäck & et al., 2012).

In the following text, only a few monitored parameters, mainly mechanical ones, will be discussed. In terms of the proposed experimental research, it is not necessary or practical to describe the chemical-thermal indicators of quality. From experience, quality of the briquette is primarily evaluated according to its density. Below, some procedures are given for determining parameters.

Fundamentally, we know of several mechanical indicators. In practice, various procedures for determining some mechanical indicators of quality are distinguished. Amongst the most basic and mandatory indicators are those given by the set of Standards “CEN-TC 335/Solid biofuels”, which describe bulk density, size of particles, density of particles, mechanical durability, and the length and diameter of the briquette.

Then, the mechanical indicators and procedures, which were used before EU Standards (Ö-NORM M 7135 a DIN 51731) for briquette density (DIN 51731), bulk density (SS 187120), or mechanical durability (SS 187120) are known. Some producers that own testing equipment to determine the afore mentioned standards, still test the basic parameters according to the “old” standards.

In practice, the procedures for simple determination of briquette quality are performed by submersion tests, visual evaluation, and the informative observation of hardness and strength (simple compression splitting).

According to valid European Technical Standards, quality is determined by the parameters in the following Tab. 2.7.

Table 2.7: Limit values for the indication of quality for pellets according to STN EN 14961-2.

Indicator	Unit	Label	ENplus-A1	ENplus-A2	EN-B
Diameter ¹⁾	mm	D06		6 ± 1	
		D08		8 ± 1	
Length ²⁾	mm	L		3.15 ≤ L ≤ 40	
Mechanical resistance	w-% ³⁾	DU	≥ 97.5	≥ 97.5	≥ 95.5
Bulk weight	kg.m ⁻³	BD		≥ 600	
Particle size (< 3.15 mm)	w-% ³⁾	F		< 1	

¹⁾ (m/m) – weight ratio.

²⁾ actual diameter of pellet; ²⁾ quantity of pellets longer than 40 mm cannot exceed 1% (m/m); ³⁾ in the delivered state, % (mm) – weight ratio in percentage.

Table 2.8: Limit values for indication of quality for briquettes according to STN EN 14961-3.

Indicator	Unit	Label	A1	A2	B
Diameter ¹⁾ or	mm	D	to be est.	diameter, width	and length
Length ¹⁾	mm	L ₁			
Width ¹⁾	mm	L ₂	to be est.	diameter, width	and length
Height ¹⁾	mm	L ₃	to be est.	diameter, width	and length
Mechanical resistance ²⁾	w-% ³⁾	DU	≥ 95	≥ 90	≥ 90
Particle density	g.cm ⁻³	DE	≥ 1.0	≥ 1.0	≥ 0.9

¹⁾ (m/m) – weight ratio.

¹⁾actual diameter of briquette; ²⁾informative test only if they are traded in bulk;³⁾in the delivered state,

% (mm) – weight ratio in percentage.

In determining the briquette quality according to STN EN 14961-3, the briquette density is investigated instead of the bulk density. Furthermore, the length, diameter, and mechanical durability of the briquette are monitored. The European STN EN 14961-3 Standard defines briquettes made from wood in three quality categories: A1, A2, and B. This division depends on the properties and source of the raw material as well as the characteristics and parameters of the product.

Density of the briquette and bulk density

Briquette density is the most important indicator of quality. The density for briquettes made from wood (HP1) given by ÖNORM M 7135 must be greater than 1.0 kg.dm⁻³. Briquettes from core wood (RP1) must have a density of at least 1.0 kg.dm⁻³. The German DIN 51731 Standard states that briquettes must have densities between 1.0 – 1.4 kg.dm⁻³, whereas the certification scheme of DIN Plus requires a minimum value of 1.12 kg.dm⁻³. There is an obvious and direct dependence between the density of the briquette and bulk density. The Swedish SS 187120 Standard (for pellets made from wood) does not define a required density for the briquette but rather the minimum bulk density in three categories. The Austrian ÖNORM M 7135 and German DIN 51731 a DIN Plus do not define requirements for the bulk density of the briquette/pellets. Density of the product is important for its manipulation. Briquettes must be cohesive so as not to crack and break into small pieces. Higher briquette densities from wood matter typically increase their strength. Briquettes with higher density also have a longer burn, which is a very important factor considering their use as fuel. Higher density also positively affects the long-term stability of the briquette volume and shape and decreases its ability to absorb moisture.

DIN 52182 (supplement to Standard DIN 51731) describes the procedure for evaluating briquette density. After densification, the briquette is placed in a stable climate. Gradually, the diameter, length, and weight are measured; however, these initial values are not possible to interpret due to dilation, a common undesirable

effect. It is necessary however to consider dilation when determining the density. Dilation changes the dimensions of the briquette due to relaxing effects of the pressing pressure and depending on the mutual interactions of the pressing temperature, moisture of the input material, and input fraction size. Based on these values, dilation character can be positive or negative (the size of briquette can increase or decrease). The mentioned Standards also take into consideration the stabilization duration, which is the time required for the briquette to stabilize and dilate. During this time, the briquette is continually measured. If, within 24 hours, the weight changes by 0.1% or less, the briquette is considered stable. The final weight is then defined as the weight of the briquette m_n (kg; g). At this point, the geometric dimensions are measured, the volume is calculated V_n (m³; cm³), and the specific density is determined from Equation 2.12. The density must be specified within 0.01 g.cm⁻³.

$$\rho_n = \frac{m_n}{V_n} \text{ (g.cm}^{-3}\text{)} \quad (2.12)$$

Bulk density is defined as the ratio of weight m_v and volume V_s .

$$\rho_s = \frac{m_v}{V_s} \text{ (g.cm}^{-3}\text{)} \quad (2.13)$$

Briquette/pellets abrasion and mechanical durability

Many methods exist today to specify the mechanical and abrasion resistance of the pellets. The method used depends on the Standard used. The abrasion and mechanical durability of the pellets is understood as the resistance to crumbling or chaffing as a result of manipulation, storage, and transport. Some Standards describe procedures to determine the amount of chaffing, whereas others determine the mechanical durability. Testing is done by shaking and rubbing the pellets. However, a pellet is never tested for both abrasion and mechanical durability. It can be said that the abrasion value after testing is the “opposite” of the value for mechanical durability. For example, from 100% of the tested volume, if 2.5 % is rubbed from the pellets after abrasion testing, then 2.5% is wasted due to abrasion. In terms of mechanical durability, after testing, 97.5% would be the recorded testing value, meaning that 97.5% of the tested pellets remain intact.

Abrasion is commonly defined for pellets only (HP4 and HP5 for DIN 51731 and DIN Plus Standards; and HP1, HP2, RP1, and RP2 for the ÖNORM M 7135 Standards). Abrasion is an important parameter for the practical assessment of quality. These Standards prescribe its required value and the procedure to determine it. Abrasion is related to requirements that restrict the formation of dust particles in the automated transportation of solid biofuels and thus is an indicator of the reduced risk of explosion during burning.

Measuring abrasion for large-diameter briquettes is typically not done; however, STN 44 1309 Standard specifies the “Determination of briquette abrasion”.

Abrasion testing is simple and quick. The test is performed using the Lignotester machine (LT – II or NHP 100), which is shown in Fig. 2.26 on the left. Pellets are tested without any dust particles; thus, before the test, a fine sieve is used to remove any dust from the surface of the pellet (3.15mm mesh in regards to ISO 3310-1). After the preliminary dust removal, $100g \pm 0.5g$ of pellets are placed in the testing apparatus (Ligno tester) and subjected to 70mbar of airflow over a 60-second interval. The artificial airflow simulates transport, collision, and friction between individual pellets and the walls of the apparatus. Abrasion is then specified as the weight of left-over material in a percentage % according to Equation 2.14. This test is performed five times, and the recorded abrasion value is specified as the arithmetic average of the values obtained from each test.



Figure 2.26: Apparatus used for testing the mechanical resistance depending on standards used.

Abrasion calculation:

$$AR = \frac{m_E - m_A}{m_E} \cdot 100 \quad (2.14)$$

where AR – abrasion (%),
 m_E – weight of pellets before test(kg),
 m_A – weight of pellets after test (kg).

Acceptable deviations for the arithmetic average is within 0.2 % of the maximum allowable abrasion with respect to ÖNORM M 7135 (DIN Plus AR_{max} = 2.3 %).

The mechanical durability, or mechanical strength, is the ability for each compressed biofuel particle (briquette/pellet) to remain in an undisturbed state, for example, during manipulation and transport. Mechanical durability is an important parameter for the practical assessment of quality. This parameter is related to the requirement to reduce dust in the automated transportation process to prevent explosion when burning.

To evaluate the mechanical durability, a test-appropriate dosage of pellets is shaken in a testing chamber. The testing apparatus can contain up to three of these chambers. Mechanical durability is calculated from the weight of left-over samples and the weight of separated material after testing. The minimum sample size is 2.5 kg. Samples should be divided into 4 equal dosages, which are sieved according to ISO 3310-2. Dosages are tested after an initial separation of dust particles. Determining the mechanical durability requires at least two tests of sieved pellets with a dosage weight of 500 ± 10 g. The sample must be weighed before testing to within 0.1 g of accuracy. In the testing apparatus, the pellets are chaffed by means of rotational movement in the chamber. After 500 rotations at $50 \pm 2 \text{ min}^{-1}$, the pellets are removed, sieved by hand, and weighed. The mechanical resistance is then calculated using Equation 2.15. The results must be calculated to the nearest hundredth of a gram (g).

$$D_U = \frac{m_A}{m_E} \cdot 100 \quad (2.15)$$

where:

D_U – mechanical resistance (%),

m_A – weight before sieving and testing (kg, g),

m_E – weight after sieving and testing (kg, g).

Strength in compression

Strength in compression is another important indicator of quality with respect to resistance when manipulating, transporting, and storing briquettes. Standards so far do not specify any required value for this indicator/parameter. Therefore, stating the strength value in compression is based on the STN 44 1309 Standard for the “Collection and modification of mechanical testing of carbon based briquettes.”

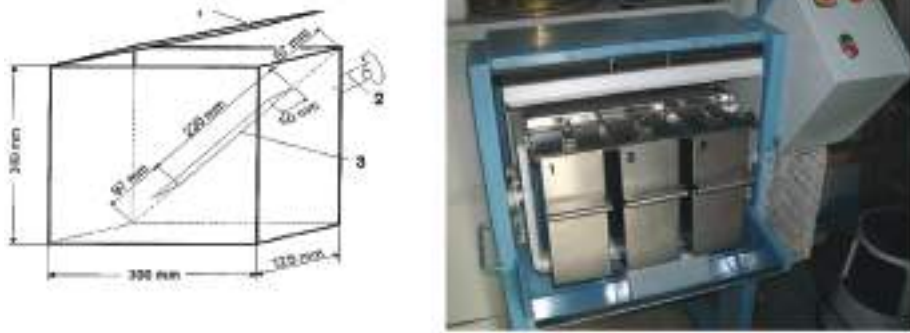


Figure 2.27: Apparatus for testing the mechanical resistance of pellets/briquettes STN EN 15210-1. (1-filling cavity, 2-driven shaft, 3-contact plate).

The term “briquette strength” refers to the maximum pressure on the punch or piston during compression testing under the following conditions according to the following expression:

$$\sigma_{Bt} = \max p \text{ (MPa)} \quad (2.16)$$

The briquette is placed for testing between the circular pistons of the press, where it is uniformly loaded with increasing pressure until complete disintegration of the briquette. The test utilizes undisturbed and complete briquettes. Briquettes are placed in the center between two circular pistons of 30 mm diameter. The pressing piston uniformly increases load on the briquette throughout the test, and the maximum pressure obtained during the test before disintegration gives the strength of the briquette in compression. Seven randomly-selected briquettes are used for testing, and the recorded value from the tests is determined from the arithmetic average of five test values (the highest and lowest test values are excluded). The resulting strength of the briquette is then:

$$\sigma_{Bt} = \frac{1}{5} \cdot \sum_{i=1}^5 p_i \text{ (MPa)} \quad (2.17)$$

where: σ_{Bt} – strength of briquette in compression (MPa),
 p_i – value of the maximum pressure at the i -th measurement (MPa).

This standard is valid for carbon-based briquettes; however, it can also be applied to briquettes made from biomass.

According to STN 441309, two pressure-related strength tests for cylindrical briquettes exist. They are a test for simple compression and a splitting test. For both tests, the force is measured until the specimen breaks apart (failure). The indicator

value for the splitting test is proportional to the force and inversely proportional to the length of briquette.

The strength test by splitting is conducted according to STN 441309. Strength test of a cylindrical briquette is calculated by:

$$\sigma_{Br} = \frac{F_{max}}{L_N} \quad (\text{N.mm}^{-1}) \quad (2.18)$$

where: σ_{Br} – strength of briquette when splitting (N.mm^{-1}),
 F_{max} – maximum force acting on the briquette (N),
 L_N – length of briquette (mm).

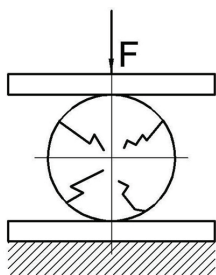


Figure 2.28: Splitting test.

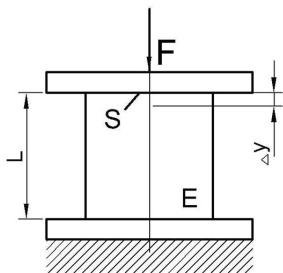


Figure 2.29: Strength test in compression.

Strength test of briquette in simple compression is performed according to STN 441309.

Strength of a cylindrical briquette is calculated as:

$$\sigma_{Bt} = E \cdot \varepsilon \quad (\text{MPa}) \quad (2.19)$$

$$\varepsilon = \frac{\Delta y}{l_n} \quad (-) \quad (2.20)$$

where:

σ_{Bt} – is the strength of the cylindrical briquette (MPa),

E – module of elasticity (MPa),

ε – ratio of deformation (-),

Δy – pressing the briquette after achieving maximum loading force (mm),

l_n – length of briquette (mm).

Determining quality of pellet – practical procedures

Apart from the above description of the basic Standards defining the indicators of quality, it is possible to use some practical procedures to obtain some initial information about the briquette. However, these procedures are not supported by any standards. Biofuel producers and sellers only use them for simple and quick evaluation of briquette quality.

Pellets/Briquette colour

Colour is usually the first property of pellets that a buyer first notices. Colour usually indicates the type of material used (wood, straw, pulp, etc.). However, colour can differ between pellets of the same material, indicating the method of compaction, finishing, quality of raw material, and cleanliness. In general, “light” colour pellets are considered high-quality. They typically have low ash content and are usually made from a clean and high-quality raw material. “Dark” pellets usually contain wood bark, which has a porous structure; as a result, chemical substances and dust particles are easily caught in these pores. Dark pellets also indicate a high degree of sawdust degradation or the use of raw material from old wood chips (Mikuláš&Vícha, 2006; 2007). In some countries, the colour is a very important quality for buyers, and therefore dark product is undesirable in a supplier’s point of view. Based on the above, and proven by experience, darker briquettes typically contain an undesirable composition compared to light briquettes.



Figure 2.30: Various colours of pellets depending on the raw material used.

The effectiveness of certain wood types in the burning process is generally known. Pellets made from spruce or pine typically do not cause any problems in the burning process itself. However, pines emit higher CO emission for its production (when storing) and has an undesirable smell. These raw materials have high sap content, and, when burning, there is a tendency for debris and carbon to form in the chimneys of the furnaces. Spruce and other hard woods have very good burning characteristics in furnaces with low emissions, light aroma, and low sap content.

It is necessary to comment that darker pellets are not only caused by wood bark content and older material, but also by the production process. Since the production process of pellets inherently uses high temperatures and pressures, low moisture content of input fractions, and slower pressing process, pellets without any bark wood can also turn out darker. A drawback to pressing can be a loss in volatile elements, which degrades the product heat potential. Pressing at higher pressures causes the outer surface of the pellet to sinter. This effect is actually a benefit as cracks do not form on the surface of the pellets. The pellets are also more compact and coherent. If the pellets as a whole are darker but without any dark spots, it can be assumed that there is no bark wood in the pellet material.



Figure 2.31: Pellets made from core wood.

Surface of the briquettes/pellets

The surface of the briquette is another significant indicator of quality. A shiny, compact surface indicates high quality. If the surface is spotty and non-uniform, it can indicate inhomogeneity in the production process and thus a non-uniform burning characteristic in the furnace (Mikuláš & Vícha, 2006; 2007).



Figure 2.32: Shiny, uniform surface of the pellet.

Briquettes/ pellets hardness

The hardness of the briquette typically relates to quality. In principle, harder briquettes have higher quality and thus higher concentration of energy (greater heat potential in the same volume). Hardness can be determined by the so-called submersion test in water (Mikuláš & Vícha, 2006; 2007). Briquettes with a high density ($> 1 \text{ kg.dm}^{-3}$) must be submerged. If the briquette crumbles within 5 minutes, the product is of very low quality. If the briquette crumbles within 15 minutes, the product is of average quality. If the briquette lasts up to 20 minutes without crumbling, the product is of high quality.

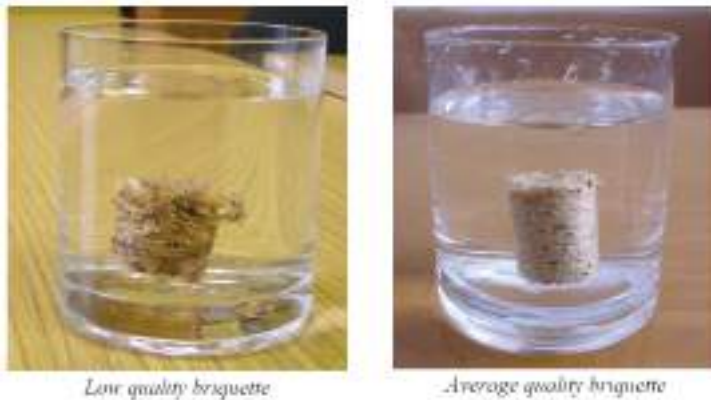


Figure 2.33: Hardness testing by submersion into water.

Another way to test the product hardness is to use a specialized apparatus (KAHL). A briquette is placed between a die and punch. The apparatus applies force on the briquette until it breaks.



Figure 2.34: KAHL apparatus for testing pellet hardness.

2.6 Parameters Affecting the Resulting Quality of the Briquette

The goal of experimental research described within this monograph is to define the effects of some parameters on the resulting quality of briquette and to determine the relations between these parameters. After a detailed analysis of the known affecting parameters, the effects on the densification process can be observed. The analyses of

these parameters were divided into three groups since they are not of similar character. The first group of parameters relates to the type of pressed material; the second group relates to the pressing technology; the third group envelopes all structural parameters:

1. pressed material:
 - type,
 - fraction moisture,
 - moisture content,
 - temperature,
2. pressing parameters:
 - pressing method,
 - temperature in the pressing chamber,
 - pressure in the pressing chamber,
 - pressing speed,
 - holding time,
3. structural parameters:
 - diameter, length, and shape (conical) of the pressing chamber,
 - type, diameter, and shape of the pressing tool,
 - material, finishing (surface roughness), and surface treatment of the pressing chamber and tool,
 - back-pressure acting on the compressed plug,
 - length of the cooling channel,

Obviously, it is not possible to perform experiments for all of the above parameters, and therefore it was necessary for the present study to choose the most appropriate parameters. Parameters which showed the greatest effect on the resulting quality of the briquette and were also easily measured were chosen. Based on these criteria, one material was chosen from the first group of parameters with definite moisture content and fraction size for analysis. From the pressing parameters, based on detailed analysis, the pressing pressure and temperature in the pressing chamber were chosen.

2.6.1 Type of Material Being Pressed

The type of material chosen for pressing is a very important factor that can significantly affect the final quality of the briquette. If wood waste is pressed, the parameters (pressure, temperature, etc.) are different than those of other types of raw materials. At the same time, pressing soft wood is different than pressing hard wood. For different types of materials and input conditions, different briquette properties (heating potential, moisture content, ash content, etc.) are obtained. Each type of material has a specific density. The density significantly impacts the mechanical-physical properties of wood. A higher specific density of the input material generally

results in better densification. Therefore, we can say that the density of wood affects its mechanical and chemical processing, influencing the quantity of wood mass required. For example, hard wood is stronger, harder and more resistant to wear and mechanical processing than soft wood.

Table 2.9: Density of the most common industrial wood materials (Bozděch & Černák, 1987; Kremer, 1995).

Wood species	Density of absolutely dry wood ρ_0 (kg.m ⁻³)
Spruce, fir, poplar	350 – 400
Basswood, aspen	400 – 450
Pine	500
Larch, Douglas fir, willow, alder, birch	500 – 600
Ash, maple, chestnut	630
Brest, oak	640 – 650
Beech	680
Acacia, hornbeam	730 – 800

Another factor affecting material densification is the presence of various types of dirt, dust, and cork, which negatively effects the briquette strength. Heating potential of the material is also very important since the densified product will concentrate this energy uniformly.

Porosity relates to density in wood. Porosity is the volume of the pores in a unit volume of wood. Pores are cavities created by the lumen, the intercellular space with capillary radius greater than 100 μm , and cavities in the cell walls of each individual wood element up to 100 μm .

The density of wood and its weight are mainly affected by the moisture content, thickness of annual growth rings and ratio of summer wood, position in the trunk, and tree life. Wood density and weight increases with water content such that the weight and volume of the wood do not change proportionally. Whereas the weight of wood can increase with rising moisture content until its complete saturation, the volume of the wood can change until the PCS. Further increase in moisture within the wood does not change its volume.

From data shown in the following figure, it is obvious that the type of material being pressed significantly affects the pressing process. To be consistent, it is necessary to work with one type of material. Due to time limitations, the experiments in the first phase were performed for one type of wood material. According to the procedures presented in this work, it should be possible to repeat the experiments with different types of materials.

Based on available sources of wood sawdust, pine sawdust was utilized in the first phase of the experiments. Pine (*pinus sylvestris* L.) is a coniferous tree with a triangular (conical) shape and originates from central and northern Europe, Asia, and Siberia. Pine wood is yellow or pinkish with a light brown structure when freshly split and turns reddish brown when exposed to the air. Pine is less resistant to biological disease, but is easily dried and processed. It has a wide application in construction materials (interior, exterior, and even marine), in furniture, and in chemical industries (pulp, wood fibre, turpentine) (Kremer, 1995). Some mechanical and thermal properties of pine are given in the following table.

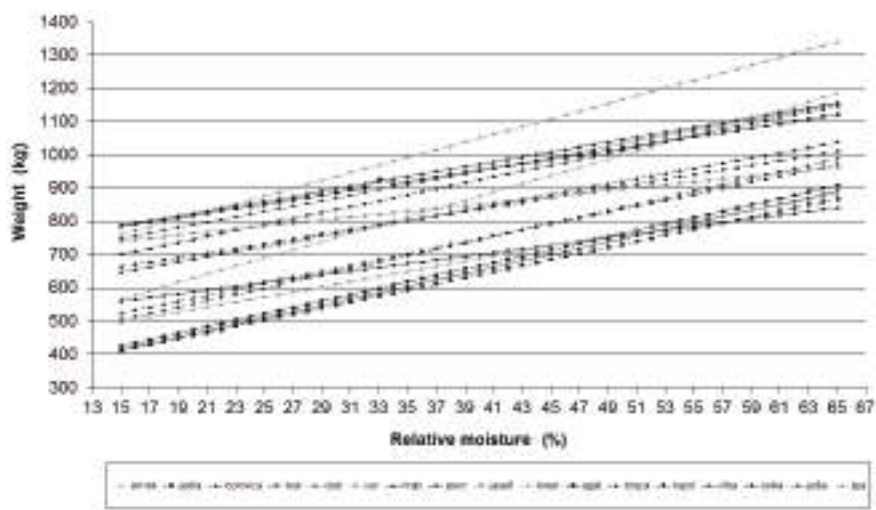


Figure 2.35: Dependence of wood weight on the moisture of different types of wood (Bozděch & Černák, 1987; Kremer, 1995).

2.6.2 Pressure in the Pressing Chamber

The pressing pressure is one of the most important factors affecting the resulting briquette strength. With increasing pressure, the briquette tends to strengthen until the yield strength of the material is reached. This also reduces the tendency of the briquette to absorb moisture when stored (Horníková, 2009). In the densification of organic waste, 55 to 355 MPa of pressure is typically used (Kegel, 1948). It is important to find an ideal combination of force and force vector direction to obtain a highly compact briquette. The pressing method has an effect on the composition of the briquette layers and thus on its overall strength.

Table 2.10: Some selected properties of pine – *pinus silvestris* (Holm & et al., 2006; Chovanec & et al., 2006; Požgaj & et al., 2006).

Density	Volumetric weight	Unit weight when dry	Volumetric shrinkage	Heating potential at
at $w_r = 0\%$	at $w_r = 25\%$			$w_r = 25\%$
500 kg.dm ⁻³	680 kg.pm	510 kg.m ⁻³	11,2 - 12,4 %	13,6 MJ.kg
Strength in compression	Bending strength	Module of elasticity in the direction of fibres E_L	Module of elasticity in the radial direction of fibres E_R	
55 N.mm ⁻²	80 N.mm ⁻²			
	Bending strength	6634 MPa	320 MPa	
Hardness	at $w_r = 12\%$	Poissons number ν_{LR}	Poissons number ν_{RL}	
28,5 MPa	1900 N	0,337	0,016	

The analysis of pressure is very interesting and complicated. To obtain a high quality briquette, it is necessary to ensure a maximum pressure within the pressing chamber. This value represents the pressure needed for compaction and feed of the briquette through the chamber. Other parameters affect the pressing pressure. Biomass under pressure undergoes two types of deformation: elastic and plastic (Chuchrjanskij & Lehký, 1953). Elastic deformation returns to its original position once the pressure is released; however, plastic deformation permanently deforms the material. The pressing temperature, pressure, and moisture content of the material have a direct effect on the elastic and plastic deformation observed. In Figure 2.36, obvious changes can be seen in pressure when pressing a pine briquette with consistent fraction size of input material ($L = 2$ mm), moisture content ($w_r = 10\%$), and temperature (55°C). However, the pressing pressure was different in each test. We can see that lower pressures resulted in crumbling of the briquette. At higher pressures, the briquettes are more consistent and compact. Fig 2.37 confirms the reasoning that higher pressures increase briquette density.

**Figure 2.36:** Briquettes with different acting pressures (Križan, Šooš & Vukelić, 2008).

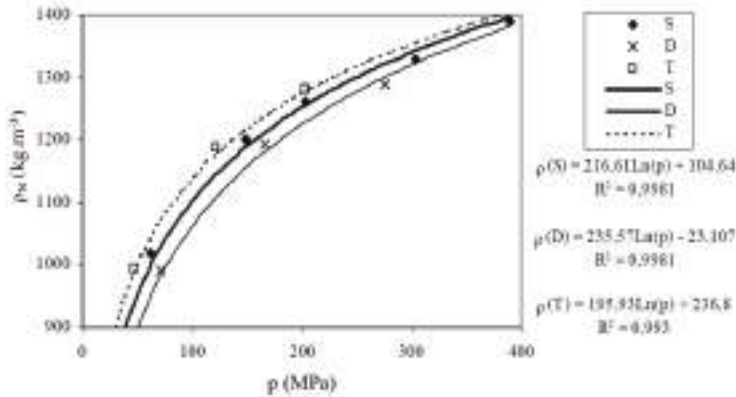


Figure 2.37: Dependence of density on the pressure for pellets made from pine (S), oak (D), and reed (T). ($T = 20^{\circ}\text{C}$, $w_f \approx 7\%$) (Kegel, 1948).

2.6.3 Pressing Temperature

The pressing temperature has a significant effect on the quality and strength of the briquette. This parameter influences the excretion of lignin from the cellular structures. Lignin is a very important substance in the densification process because it confers adhesive effects in the pressed material. It connects fibres and acts as a reinforcing factor in the cellular molecules. The more lignin in the input material, and the more that can be extracted in the pressing process, the more uniformity and thus quality can be achieved from the resulting briquette. Lignin strengthens the briquette primarily in compression, but lignin is excreted only at certain pressing temperatures, which must be attained for the densification process to be optimized. Lignin plasticizes at approximately 120°C , but this temperature, of course, depends on the exact material being pressed. From the known dependencies between strength and temperature, we know that it is not necessary to excessively increase temperature (Fig. 2.38). Higher temperatures not only evaporate water but also volatilize substances. Therefore, it is necessary to evaluate exactly how much the temperature should rise without losing heating potential of the briquette. Volatile substances are, by the way, very important components of a biofuel in terms of their heating potential.

Pressing at room temperature (20°C) results in an unstable briquette; the product has low strength, which causes faster crumbling when burning, resulting in a faster burn. With constant pressure but increased temperature, the strength of the briquette increases until a certain point. Increasing the temperature from 20°C to 100°C yields insignificant losses from the evaporation of water (Holota & et al., 1984). Heating to 150°C initiates the evaporation of volatile substances, which reduces the heating potential of the briquette (Holota & et al., 1984). Of course, temperature also affects the moisture content of the product.

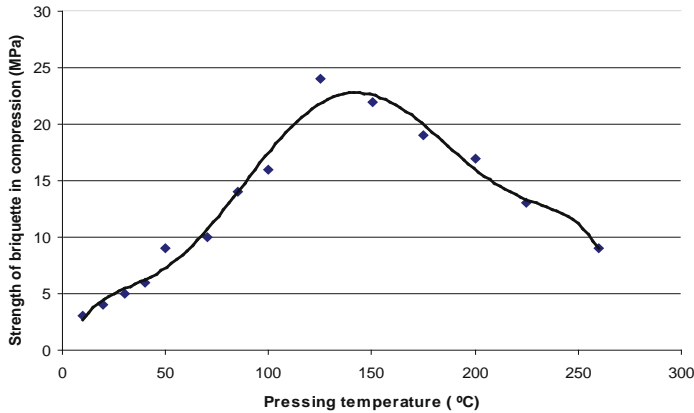


Figure 2.38: Dependence of briquette strength on the pressing temperature ($p = 100$ MPa, $w_r = 20\%$, $L = 1$ mm) (Sitzmann & Buschhart, 2009).

When heating wood biomass, its physical and chemical properties change (Chuchrjanskij & Lehký, 1953). One sign of this change is colour. High temperatures cause the vessels within the wood to decompose into their base elements. It is known that wood consists of cellulose, lignin, and hemicelluloses. Cellulose creates the primary structure, whereas the lignin and hemicelluloses influence this structure. Temperature and moisture content affects substances filling the cellulose structure by causing them to soften and plasticize. Heat alone can cause this to occur; however, this phenomenon is not nearly observed to the same extent when moisture is also an influence (Solár, 1997).

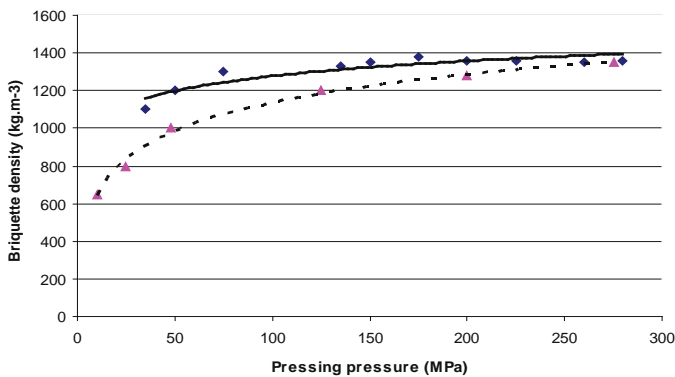


Figure 2.39: Dependence of density on pressure when pressing preheated samples at $T = 200$ °C and room temperature $T = 20$ °C (Kádarová, 2005).

Pressure and temperature go “hand in hand” in the densification process. If we increase the pressure, we can reduce the required temperature, and vice versa. This is nicely shown in Figure 2.39. Pressing at higher temperatures obtains the desired density without increasing the pressure. It is also generally known that it is economically advantageous to increase temperature rather than pressure.

Under the effects of heat and moisture, the physical-mechanical properties of wood biomass temporarily or permanently change. Temporary changes cause the material to return to its original state once cooled. These changes occur mainly when the pressing conditions are not optimal with respect to the pressed material. Temporary changes include the sharp increase in elastic deformation when heated and the resultant change in strength at different temperatures. Permanent changes in the properties of wood biomass, caused by the temporary increase in temperature, do not allow the material to return to its original state after cooling. Permanent changes occur under optimal pressing conditions in terms of the material properties of the pressed material. The effect of heat on the permanent physical-mechanical and chemical properties of wood biomass depends (to an extent) on the temperature, method used, and heating duration. Increasing temperature and prolonging the heating process increases these effects (Chuchrjanskij & Lehký, 1953).

2.6.4 Moisture of the Input Fraction

The maximum moisture content of the material is an essential condition for the optimal densification of biomass. The effects of moisture content on the material must be known to produce quality briquettes as defined by Standards. It is known from specific results from research discussed in the literature that density and strength depend on the moisture content of the pressed material, for example, in the case of pressing wood sawdust (Kegel, 1948). It is also known that other materials, when pressed, those characteristics have similar dependencies. We can therefore say that the briquette quality depends on the moisture in the pressed material and that a large difference is not expected in the optimal level of moisture between different materials. The optimal value, however, is not known. Based on our knowledge and experience, an acceptable level of moisture is from 8-18% (Šooš, 2007). If the moisture content of the pressed material is very low, or very high for that matter, the particles become inconsistently arranged, and the resulting briquette becomes unstable. Research and experiments have shown that moisture has an effect on the plasticization of lignin. The temperature at which lignin softens depends also on the type of wood and method of isolation. The temperature at which lignin changes phase from a solid to a plasticized state is proportional to its molecular mass and non-proportional to the level of moisture (Hejma& et al., 1981). The effects of water content (material moisture) on the transition temperature of lignin can be seen in the following figure.

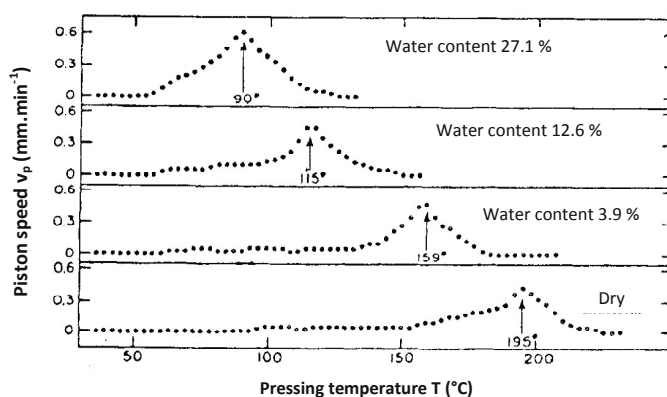


Figure 2.40: Effects of moisture on the transition temperature of spruce lignin at different piston speeds (Hejma & et al., 1981).

The mutual interaction between the material moisture, pressing pressure, and pressing temperature is very interesting. If we set the process to the optimal temperature and pressure but allow moisture content to be out of the optimal range, the resultant briquette will be unstable. Proper temperature ensures that the lignin plasticizes in the pressed material, and the optimal pressure provides densification of this material. However, if the pressed material has high moisture content, escaping water in the form of steam cracks the briquette. If the moisture content is very low (below 6 %), the briquette must be pressed with a higher temperature and pressure to obtain a high-quality product, which becomes economically unprofitable. With reduced moisture content, the heating potential must be increased. A certain moisture level causes a higher temperature in the densification process and thus a higher quality briquette. Optimal moisture level improves the compaction of the material by causing a degree of sintering which improves the unity of the briquettes particles. Optimal moisture levels are one of the focuses of current experimental research.

2.6.5 Size of the Input Fraction

The size of the input fraction also has an important effect on the densification process because larger input fractions increase the energy needed for densification. However, briquettes formed from large input fractions have lower homogeneity and strength. On the other hand, a large portion of fine particles allows for better material densification. The resulting briquette is uniform, of high quality, and reaches higher volumetric density. With increasing fraction size, the bond strength between particles decreases, causing them to crumble. The quality of a briquette, as a result, decreases and the necessary pressing pressure increases. When pressing, especially

without additives, the surfaces of the grains must contact on the greatest possible area. The size of this contacting area increases as the fraction size becomes finer and higher pressing pressures are applied. Existing relationships prove that the size of the input fractions should be as small as possible in order to obtain the desired briquette strength (Kádarová, 2005).

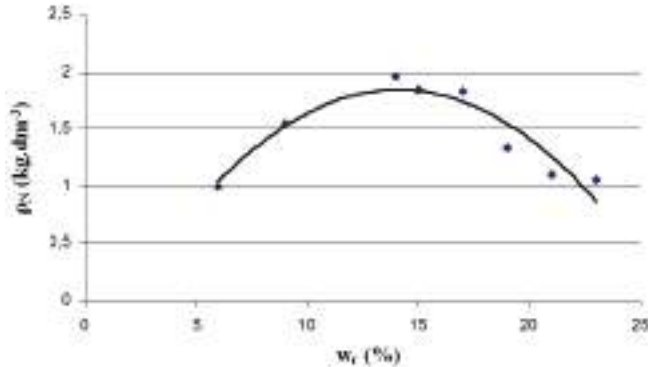


Figure 2.41: Dependence of briquette density on the moisture level of the material (wood sawdust) (Kádarová, 2005).



Figure 2.42: Comparison of briquettes made from Pine sawdust with varying fraction sizes. ($p=159$ MPa, $T=25^{\circ}\text{C}$, $w_r=10\%$) (Križan, 2009).

Particles exist in the material that, due to external forces, deform and load mainly the contact regions. In terms of densification, it is very important that the described forces are created between particles. The strength of the resultant bonds increases with decreasing fraction size. However, the optimum fraction size changes with respect to the densification technology employed and the raw material used. Therefore, it is important to investigate the effects of fraction size on the resulting density and strength of the briquette. Fraction size is thus one of the parameters that I attempted to identify and verify through the following experiments.

2.7 Mathematical Model of the Pressing Process of Wood Biomass

This chapter summarizes the knowledge obtained from various models which best describe uni-axial pressing technology. One of the goals of our experimental research is to identify relationships between pressing wood biomass and the basic theory for pressing. Because one of the stated goals was to create a mathematical model for the pressing process, it was also necessary to perform an analysis of existing mathematical models and compare and evaluate them. It is necessary to understand the pressing process itself – densification, pressure distribution within the chamber, effects of chamber geometry on the pressure distribution, and so on. Based on this knowledge, the effects occurring within the pressing chamber during compaction and the resulting behaviour of the material can be effectively described. The mechanical properties of all wood materials can be improved by the densification of their components (increasing the unit volume of wood). How the force is applied in the pressing process determines whether the process is a uniaxial (single-axis) or multi-axial (hydrostatic) pressing process.

2.7.1 Theory for Uniaxial Pressing of Particulate Matter

The boundary conditions for uniaxial pressing are kinematic ($\varepsilon_r = 0$, $\varepsilon_a = \varepsilon_v$), and the stress tensor is anisotropic (which is why the process is also known as anisotropic consolidation). An undesirable effect in uniaxial pressing is shell friction, which partially decreases the theoretical value of anisotropic consolidation. Anisotropic stresses in uniaxial pressing creates a compressive structure, and therefore, compressive anisotropy. The nonzero stress deviator evokes dilatational behaviour (Fedá, 1977).

According to Fedá (1977), uniaxial pressing of a linear, elastic substance under a constant coefficient of lateral pressure K_0 creates a condition that has been experimentally verified to behave according to Hooke's law:

$$\varepsilon_a = \frac{1-2}{E_v} (1+2K_0) \sigma_a \quad (-) \quad (2.21)$$

where E_v is the module of elasticity in the vertical direction. For elastic deformation, the structure does not change, and therefore $E_v = \text{konšt}$. Deformation of the particle matter occurs primarily due to irreversible shifting of the particles. Because the structure of the particle matter changes, $E_v \neq \text{konšt}$ and the stress function σ_a , which characterizes the intensity of the structural alteration, also changes. Equation 2.19 can then be written:

$$d\left(\frac{dh}{h_0}\right) = \frac{d\sigma}{E_0}(-) \quad (2.22)$$

where E_0 is the volumetric module of elasticity (MPa), $\sigma = (\sigma_s)$ is the average stress (MPa), and h_0 is the initial height of the sample (mm).

In general, Equation (2.22) is used for uniaxial pressing, where $\sigma = \sigma_a$.

After mathematical revisions and the substitution of the boundary conditions, according to Fedá (1977), the final relationship for two basic types of compression curves can be achieved. In cases of linear changes in the module of deformation E'_0 along with the stress σ for $n' = 1$ (exponent describing the intensity of structural changes), the following equation is valid:

$$\Delta e = C(1 + e_0) \ln \frac{\sigma}{\sigma_0} (-) \quad (2.23)$$

Where C is the compressibility coefficient, $n' = 0$, representing an unchanged structure. However, particulate matter has $n' > 0$. In cases where $n' \neq 1$, we can substitute $C(1 - n') = C_0$ and $n' - 1 = n$ to obtain:

$$\Delta e = C_0(1 + e_0) \left[1 - \left(\frac{\sigma_0}{\sigma} \right)^n \right] (-) \quad (2.24)$$

where $\Delta e = e_0 - e$ is the change in porosity and e_0 is the initial value of porosity for $\sigma = \sigma_0$ (Fedá, 1977).

In the literature, many analytical expressions are given for the compressibility curve, but these expressions often contradict each other regarding the value of porosity. For n' approaching +1, the curves are rather similar. According to Fedá (1977), Equation 2.23 applies for pressing brittle materials with low plasticity, and Equation 2.24 applies for pressing plastic substances. Furthermore, according to Fedá (1977), it is obvious that Equation 2.23 for $n' = 1$ is more realistic for predicting the slower growth in resistance of the particulate matter against compression, where the sliding contact becomes

predominant and the loading character significantly changes the structure of the particulate matter. Its range of application is therefore limited to all brittle powders, with a “sliding” factor to describe the contacts grinding and regenerating, including metallic powders with an oxidized surface, mainly at low pressures, and other substances with anisotropic particles, such as textile materials, wood, saw dust, and shavings, etc.. For all of these raw materials, the contacting bonds do not significantly grow under pressure, and a highly compressive structure results. Equation 2.24 for $n < 0$ characterizes a higher growth in structural resistance to compression caused by a gradual increase in contacting pairs. Its validity is therefore considered mainly for powdery soft metals; metals subject to higher pressures, where the surface oxide is breached and the coefficient of intergranular friction sharply increases; and for particulate matter with isometric grains.

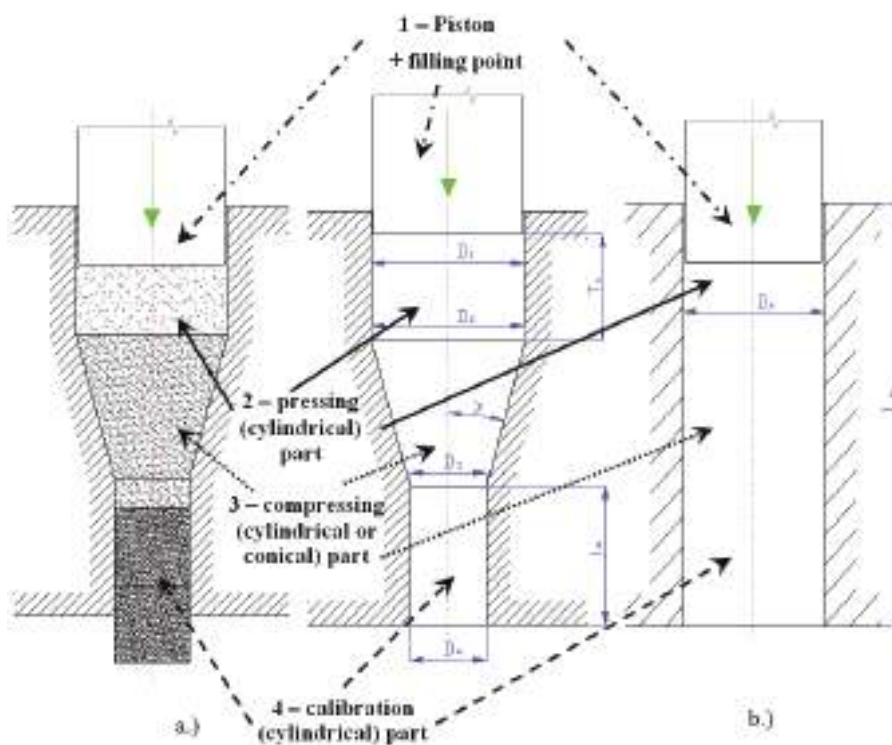


Figure 2.43: Basic parts and main dimensions of the pressing chamber a.)conical; b.) cylindrical (Storožev& Popov, 1978).

2.7.2 Theory of Forward Pressing

The description of uniaxial pressing requires the knowledge and description of forward pressing as one of the basic technologies for cold, volumetric forming. When forming by pressing, material pours from a closed cavity through an opening. The shape of this opening defines the lateral cross-section of the briquette. This process can be related to the densification of biomass. All existing principles, even densification technologies, utilize these concepts. The theory of forward pressing and the description of pressure ratios within the pressing chamber are given for the simplest case, where the pressing chamber is constructed of four parts (Fig. 2.43). This type of pressing chamber is most commonly used in the construction of pressing machines. Pressing chambers with cylindrical cross-section are also used (Fig. 2.43) over the entire length of the chamber or a conical chamber over the entire length. Cylindrical or ply matrices, the opening of the briquette machine, use a diverse variation of cylindrical and conical shapes (Fig. 2.44). The described type within this work mimics typically-used pressing chambers.

In the schematic below, the pressing part (2) acts as the input container. Material is transported to this part, and under external forces from the pressing piston (1), the densification process may begin. The (conical) compacting part (3), known also as the redactor, is part of the pressing chamber where the basic deformation of the input material is performed. It is here that the densification process is performed due to pressing forces and the pressure against the chamber walls. In the calibration part (4), the briquette is kept under pressure for a specific period of time to reduce the effects of dilation (Blaščík & Polák, 1987; Storožev & Popov, 1978). The calibration part gives the briquette its final shape and holds it for a defined period under pressure and heat to reduce the tendency of the briquette to crumble after leaving the pressing chamber. A briquette pushed through a cylindrical calibration part usually changes very little, and the changes that do occur are a result of dilation and elastic limits of the briquette. The magnitude of their effects depends on pressing parameters (pressing temperature and pressure) and the properties of the pressed material (moisture content and fraction size). Due to dilation, a typical undesirable side effect, a stabilization phase must be ensured throughout which dilation occurs. After this time, the briquette is considered to be stable and its density can be determined. Throughout dilation, and more so if excess moisture is present, the briquette changes dimensions and weight. Within the briquette, cracks occur, which reduce the density and quality of the briquette. Cracking also affects the resistance to crumbling and the moisture saturation from the air. The briquette can crumble when manipulated. The stabilization time (dilation time) can be shortened by using a longer calibration part in the chamber and employing the optimum levels of moisture and fraction size in the input material.

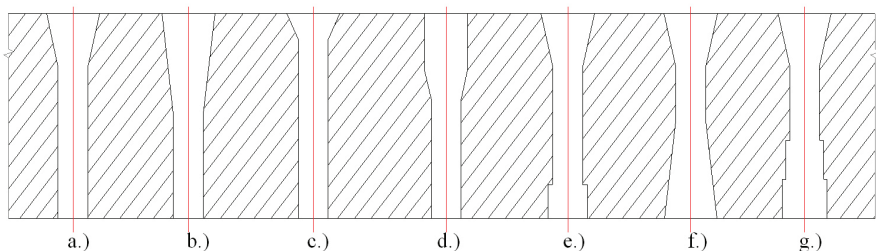


Figure 2.44: Examples of different chamber cross sections - a.) classic; b.) deep; c.) shallow; d.) well; e.) cylindrical; f.) conical; g.) step (Perez, 2009).

The pressure ratios are calculated separately for parts 2, 3, and 4 of the pressing chamber (Storožev & Popov, 1978). The resulting relationship for calculating the forming (pressing) pressure within the chamber is given as a sum of the pressing pressures acting within each part of the chamber. The following formulas are valid for pressing chambers which have a conical pressing part. Describing the pressure ratios within the pressing chamber based on the fundamental theory for forward pressing can be explained for part 4 of the chamber. The required pressure required in a cylindrical cross section of the calibration part is given by (Storožev & Popov, 1978):

$$p_4 = \sigma_k \frac{4\mu l}{D_4} \text{ (MPa)} \quad (2.25)$$

where: μ – friction coefficient (-); σ_k – yield strength of the material (MPa); D_4 – diameter of the calibration part of the pressing chamber (mm); l_4 – length of the calibration part (mm).

Calculating the pressure distribution in the pressing part, conical part 3, is described by equation 2.26 (Storožev & Popov, 1978). This equation, however, is valid for pressing chambers with a small angle γ .

$$p_3 = \sigma_k \cdot \left(1 + \frac{\mu + 0.5}{2\gamma} \right) \cdot \ln \frac{S_2}{S_4} + p_4 \text{ (MPa)} \quad (2.26)$$

where: γ – conicity of the walls in part 3 (°)

S_2 – cross section area of the output of the pressing part (mm²)

S_4 – cross section area of the input into the calibration part (mm²)

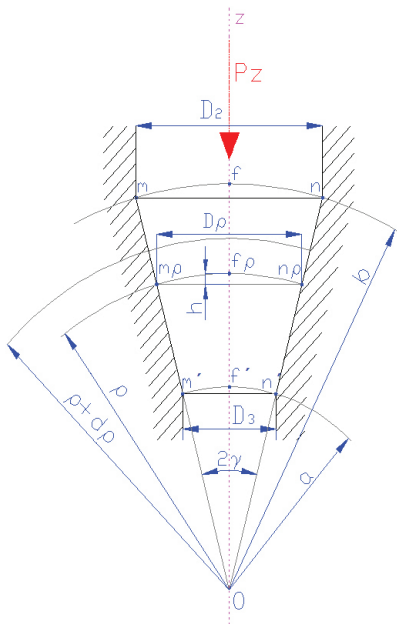


Figure 2.45: Spherical coordinates describing the upper and lower interfaces of plastic deformation concentration in a conical pressing chamber (Storožev & Popov, 1978).

When investigating the conical pressing part (3), spherical coordinates are used (Storožev & Popov, 1978). The interface between the concentrated plastic deformation approximates the area mfn of the spherical shelter with the radius b and limit angle 2γ (Fig. 2.45); in this case, f represents concentration and not the friction coefficient. The lower interface is represented by the area $m'f'n'$ of the spherical shelter with a radius a and the same limit angle 2γ . In this case the diameter D_3 is equal to D_4 . The authors of a well-established study (Storožev & Popov, 1978) assume that the particle shift at the concentration of deformation occurs on the radius ρ . Determining dV , they take an elementary volume, bordered by the spherical surfaces with a radius satisfying the values of ρ and $\rho + d\rho$ and conical chamber surface.

The plastic deformation concentration of our material features a large inconsistency between the grain size and the occurrence of residual stresses. In practice, the predominant first type of deformation concentration in the pressing part (2) originates during pressing (2). Because the plastic deformation in the pressing part (2) in this case practically doesn't exist, we can assume that the material is shifted as a whole and has equally distributed elastic pressure. Storožev & Popov (1978) show that, in favourable friction conditions, the pressures on the walls of the feeding chamber are much lower than the axial pressure. Friction forces decrease linearly with shorter stroke lengths of the piston T_k . These problems are solved by means of the balanced work method (Storožev & Popov, 1978). The fundamental law of this method is, for

plastic deformation, that the work of external forces acting on specific paths is equal to the work of the internal forces ($A_{out.} = A_{int.}$). For this case, work A_3 was set to be equivalent to the sum of the work of the resulting force F_3 of the active pressure on the upper interface where the plastic deformation is concentrated plus the total deformation work to change the shape (A_p) plus the work of the contact friction forces on the conical surface (A_f) and the work needed to overcome the resistance in the calibration part (4).

The pressure on the pressing piston acting in part 2 of the chamber is calculated by the following equation (Storožev & Popov, 1978):

$$p_2 = p_3 + \sigma_k \cdot \frac{2T_k}{D_2} \text{ (MPa)} \quad (2.27)$$

where: T_k – distance between the piston and lower end of the pressing part of the chamber (mm); D_2 – is the diameter of the pressing part (mm)

The resulting relationship for the calculation for (pressing) pressure in the pressing chamber is determined by the following relation, where we substitute into Equation 2.27 the value of p_3 from Equation 2.26 and p_4 from Equation 2.25 (Storožev & Popov, 1978):

$$p = \sigma_k \cdot \left(\ln \frac{S_2}{S_4} + \ln \frac{S_2}{S_4} \cdot \frac{\mu + 0,5}{2\gamma} + \frac{2T_k}{D_2} + \frac{4\mu I_4}{D_4} \right) \text{ (MPa)} \quad (2.28)$$

This simple procedure allows for the design of an appropriate, optimal shape for the pressing chamber. It also allows for the calculation of acting pressures in the pressing chamber.

2.7.3 Mathematical Model – “Force Relations in the Channel”

The mathematical model in this section describes the pressure ratios within an enclosed pressing chamber of a uniaxial vertical press. The use of a backpressure plug prevents material loss when filling the chamber (Fig. 2.46). The pressure ratio within the pressing chamber between the piston and plug are explained by Horrihs (1985) for an element of pressed material dx with neglected briquette weight throughout the pressing process.

From the pressure ratios within the chamber, it is possible to express the equation for force equilibrium as:

$$\left[p_m - (p_m + dp_m) \right] \frac{\pi D_k^2}{4} - \mu p_r \pi D_k dx = 0 \quad (2.29)$$

When pressing bulk materials, we typically deal with anisotropic pressure (higher pressures are perpendicular). The ratio between the primary stresses (radial σ_r / axial σ_m) is defined as the residual pressure coefficient, or horizontal pressing ratio λ . For a disperse material, λ reaches values from 0 to 1.

$$\lambda = \frac{\sigma_r}{\sigma_m} = \frac{p_r}{p_m} \Rightarrow p_r = \lambda p_m \text{ (MPa)} \quad (2.30)$$

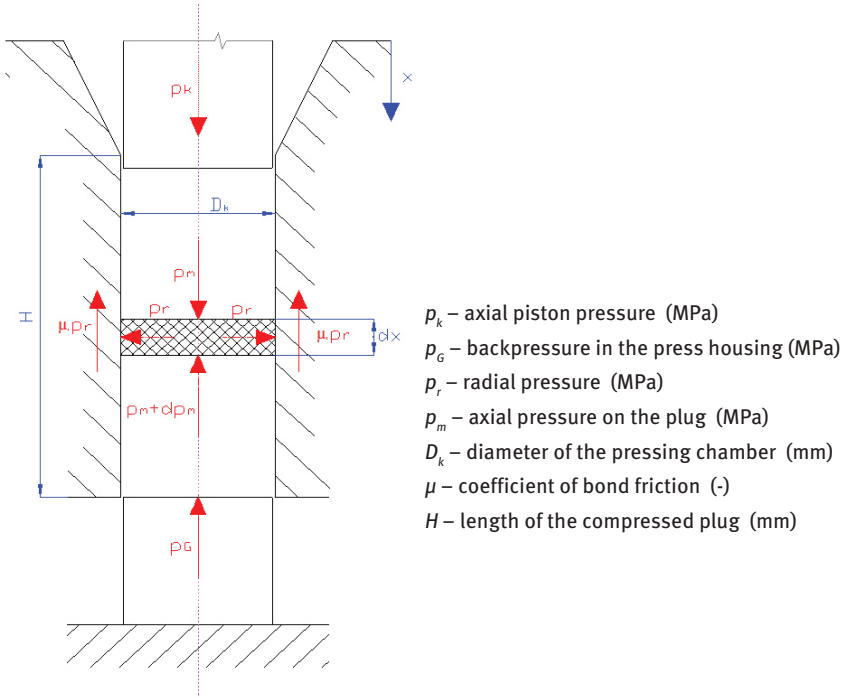


Figure 2.46: Pressure distribution in the pressing chamber in a uniaxial vertical press (Horrichs, 1985).

After substituting Equation 2.30 into Equation 2.29, and applying the following boundary conditions: $x = 0$, $p_m = p_k$, pre $x = H$, $p_m = p_G$
 The following relation is obtained (Horrichs, 1985):

$$p_k = p_G \cdot e^{\frac{4\lambda \cdot \mu \cdot H}{D_k}} \text{ (MPa)} \quad (2.31)$$

Equation 2.31 gives the relationship between the piston pressure p_k and the backpressure acting on the pressed material p_G . This relationship can be used for pressing in an open chamber where the back pressure is generated by static friction between the material and chamber walls. From this relation, we can further determine the magnitude of necessary backpressure in the operation and determine the static friction in the press column (Fedá, 1977). Modifying Equation 2.31 can yield values for p_G for any desired value of p_k .

$$p_G = p_k \cdot e^{\frac{4 \cdot \lambda \cdot \mu \cdot H}{D_k}} \text{ (MPa)} \quad (2.32)$$

Experience has shown that, after pressing, it is still necessary to determine the pressure of the pressed plug. Horrichs (1985) developed a formulation of relationships for the pressed plug because, after freeing the load on it, residual radial pressure dominates within the plug ($p_{r,r}$). This residual pressure causes briquettes to fracture. Therefore, it is very important to determine the relationship between the pressing pressure and length of the chamber (calibration part). Throughout the pressing process, the axial stress is dominant. After pushing out the plug, the dominant stress becomes radial. This means that the change in direction of stress must occur under the unloaded state – when pushing out the plug. Therefore, a point exists when the axial and radial stress are equal (hydrostatic stress state). After this point, the radial stresses become larger than the axial stresses until pressure is completely removed and residues of axial stress remain. The briquette is necessary to push out of the pressing chamber only when its strength f_c is greater than the residual radial stresses $\sigma_{r,r}$, or $p_{r,r}$. If this condition is satisfied, it is ok to push the briquette out of the calibration part of the pressing chamber.

The behaviour of the axial and radial pressure is formulated with respect to the initial compaction in the pressing phase with relief and over-loading states in the pressing phase according to the following relations (Horrichs, 1985):

a) for pressing the plug (plastic range), the following is true:

$$p_r = \lambda \cdot p_m \text{ (MPa)} \quad (2.33)$$

b) for the movement of the already compacted plug (elastic range), the following is true:

$$p_r = A \cdot p_m + p_{r,r} \text{ (MPa)} \quad (2.34)$$

Where the constant A represents the material constant, which gives the degree of elastic properties in the pressed material.

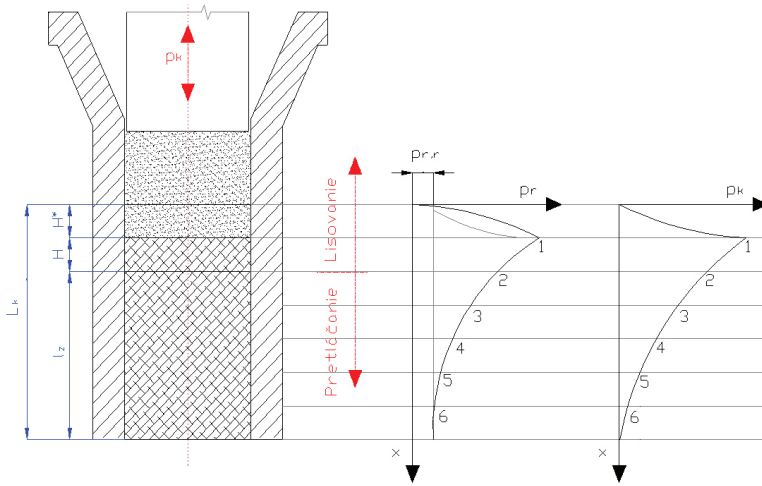


Figure 2.47: Behaviour of the radial and axial pressure when pressing in the pressing chamber (Horrihgs, 1985).

Based on pressure conditions in the pushing phase, force equilibrium at any random point (of the already pressed material) of an element (Fig. 2.46) that is part of the pressing column (Fig. 2.47) can be evaluated. With the applied boundary conditions for $x=l_z$, and considering Equation 2.34 Horrihgs, 1985), we can formulate a relationship (2.35) between the pressure on the evenly compressed plug and the length of column l_z :

$$p_m(x) = \frac{p_{r,r}}{A} \cdot (e^{\frac{4\mu \cdot A}{D_k} \cdot (1-x)} - 1) \text{ (MPa)} \quad (2.35)$$

After substituting Equation 2.32 into Equation 2.35, we obtain the following relation:

$$l_z = \frac{D_k}{4 \cdot \mu \cdot A} \cdot \ln \left(A \cdot \frac{p_G}{p_{r,r}} + 1 \right) \text{ (mm)} \quad (2.36)$$

The overall length of the pressing channel L_k is given by the sum of column length l_z and plug length H ; L_k can be calculated directly from the following Equation 2.37, since $l_z \gg H$:

$$L_k = \frac{D_k}{4 \cdot \mu \cdot A} \cdot \ln \left(A \cdot \frac{p_G}{p_{r,r}} + 1 \right) \text{ (mm)} \quad (2.37)$$

Calculating the length of the pressing channel (length of the pressed column) is possible only with known length of the pressed plug H (using the pressing ratio, dimensions of the pressing chamber, and the material properties) for known pressure p_k and known material constants μ , λ , A , a , and $p_{r,r}$.

2.7.4 Mathematical Model – “Pure Elastic Case”

The next mathematical model, stated according to Holm & et al.(2006), considers the pressing of the elastic material in the pressing chamber. The derived relations give the expressions for changing plug pressure.

The chosen element has the form of a plane disk with thickness dx and radius r . The center of this disk is identical with the axis of the press opening. The differential change in pressure dp_m through dx is given by the relation:

$$dp_m = \frac{dF_m}{\pi \cdot r^2} \Rightarrow dF_m = dp_m \cdot \pi \cdot r^2 \quad (2.38)$$

Friction force dF_μ acting on the differential area $2\pi \cdot r \cdot dx$ is given by relation:

$$dF_\mu = \mu \cdot dF_N = \mu \cdot p_N \cdot 2\pi \cdot r \cdot dx \quad (2.39)$$

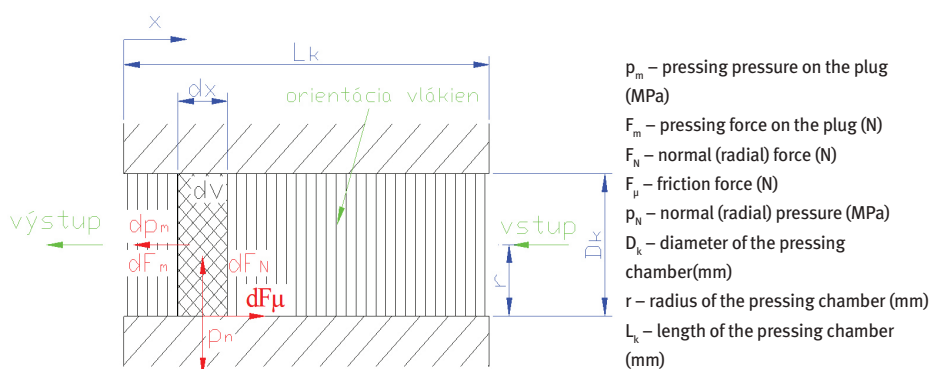


Figure 2.48: Schematic sketch of the forces and pressures acting on an element volume dV within the pressing channel (Holm & et al., 2006).

The following relations are based on the assumption that wood is an orthotropic material (independent mechanical properties in the parallel and perpendicular directions of the grain fibres). Holm & et al.(2006) assume that the fibre bundles in pellets are oriented perpendicular to the larger dimension of the pressing chamber. Furthermore, there is a consideration that the material has elastic properties (dependence between stress and strain) that are described by Hooke's law. For materials fulfilling Hooke's law, the ratio between stress and strain is constant.

Stress causing expansion is considered positive, whereas stress causing shrinkage is considered negative. If expansion or shrinkage of an element occurs due to some applied force and is lost due to the loss of this force, the material is known as elastic. The relationship between the parallel and perpendicular fibre bundles and radial deformation describes the individual coefficient between materials (Poisson's coefficient). Poisson's coefficient is defined as the ratio of elongation for a specific element ν_{RL} . Therefore the coefficient ν_{RL} can be written as:

$$\nu_{RL} = \frac{\text{longitudinal}_{-}\text{extension}}{\text{radial}_{-}\text{extension}} = \frac{\frac{dv}{dr}}{\frac{du}{dx}} \quad (2.40)$$

The above expression assumes that the material is homogeneous throughout its cross section (Holm & et al., 2006). In the case of spatial restrictions of pellets in the pressing channel, radial expansion is not possible, and therefore elongation manifests itself as a stress on the walls of the pressing channel. Hooke's law for the lateral and radial directions of fibre bundles can be written as follows:

$$F_L = E_L \cdot S_r \cdot \frac{\Delta r}{r} \quad (\text{N}) \quad (2.41)$$

$$F_R = E_R \cdot S_k \cdot \frac{du}{dx} \quad (\text{N}) \quad (2.42)$$

where E_L and E_R are modules of elasticity in the lateral and radial directions of the fibre bundles, respectively. $S_r = 2 \cdot \pi \cdot r \cdot dx$ is the area of the element side shell, and $S_k = \pi \cdot r^2$ is the cross-sectional area of the element. Furthermore, if the acting pressure in the lateral and radial (p_r/p_k) directions are compared and the following conditions are applied (Holm & et al., 2006): p_r is equal to p_N and p_k is equal to p_m . Then:

$$p_N = G \cdot \nu_{RL} \cdot p_m \quad (\text{MPa}) \quad (2.43)$$

where G is the ratio between the module of elasticity in the lateral and radial directions of the fibre bundles (E_L/E_R).

If the following equations (2.38 and 2.39) are made equal to each other, we obtain the differential equation in p_m :

$$dF_m = dF_\mu \rightarrow dp_m \pi \cdot r^2 = \mu \cdot p_N \cdot 2\pi \cdot r \cdot dx \quad (2.44)$$

After rewriting these equations using Equation 2.43 and its solution for $x - x_0 = L_k$ according to [11], we obtain the resulting relation, which gives the relation for the change in pressing pressure on the plug in the pressing channel of the chamber, where p_{m0} is the initial pressing pressure:

$$p_m = p_{m0} \cdot e^{\frac{2 \cdot \mu \cdot G \cdot V_{RL} \cdot L_k}{r}} \text{ (MPa)} \quad (2.45)$$

2.7.5 Mathematical-physical Model – “Material Composition”

The following mathematical-physical model is a model which best reflects the target model of experimental research. It was designed by Kádarová (2005). It is shown that the densification process itself is very complicated and involves many parameters affecting its process. This mathematical model is valid only within a certain range of pressure changes and temperature intervals of the material. The mathematical-physical model designed and based on experimental results has the following form (Kádarová, 2005):

$$\rho = A_K \cdot (p + B) \cdot e^{kp \cdot (p_{max} - p)} \quad (2.46)$$

where: ρ – is the resulting density ($\text{kg} \cdot \text{dm}^{-3}$); A_K – coefficient calculated based on experimental results characterising shape and curvature of the curve; B – material constant; kp – coefficient related to the pressure, calculated from experimental results; p_{max} – coefficient chosen based on monitoring of pressure (maximum pressure) (MPa); p – pressing pressure (MPa)

This equation characterises the relationship between briquette density and pressing pressure. For every temperature level, it was necessary to calculate from experimental values. Coefficient kp was calculated based on experimental results. Increasing the densification pressure allows us to reach high values of volumetric density. Based on monitored pressures (Kádarová, 2005), the maximum effective pressing pressure was determined to be $p_{max} = 400$ MPa. It is irrational and, in most cases, ineffective to exceed this pressure value. Then, Equation 2.46 has the following form:

$$\rho = A_K \cdot (p + B) \cdot e^{kp \cdot (400 - p)} \quad (2.47)$$

Experiments were performed with the following materials – spruce and oak sawdust, straw, and stems (Kádarová, 2005). In this work, the following material constants (B) were experimentally obtained:

- spruce sawdust, $B = 96$
- oak sawdust, $B=110$
- straw, $B=106$
- stems, $B=221$

Constant B depends on the content of volatile substances up to 76%, where it sharply decreases (Kádarová, 2005). A similar behaviour can be related to cellulose content up to 48%. Increasing these variables has little effect on the constants. Based on this work, the author concludes that, within the range of these changes and for the content of volatile substances and cellulose, constant B results as the material constant.

Similarly, the experimental findings and values for coefficient A_k for each mentioned material are reported (Kádarová, 2005). However, this coefficient changes with respect to temperature, meaning that it is a function of the material temperature. Constant k_p is a coefficient related to the pressure and is the same as coefficient A_k for the functional temperature of the material

2.7.6 Summary

From the above mathematical descriptions, one can understand that not many mathematical models describing the densification process exist. The ones that do exist are based on pressing theory and describe the pressure ratio throughout the pressing process. These models can be used in the design of the pressing chamber or the formulation of a mathematical model that describes the effects of structural parameters on the resulting briquette quality. However, models describing the effects of changing technological parameters on the pressing process and effects on the pressed material do not exist. It is very important to find a relationship between the density of the briquette and pressing pressure, temperature, moisture content, and input fraction size. Knowledge of these relationships could provide an excellent insight for designing pressing machines (chambers). The experiments within this monograph hence strive to design and justify the use of a mathematical model describing the densification process for application to real practices.

3 Pressing Stand and Methods of Processing Measured Data

This chapter of the monograph is dedicated to experimental research of densification process in laboratory conditions. This chapter will focus on information summarised in the second chapter, including the process of compaction, the parameters affecting the process, material properties, and so on. Included are the design of an experimental pressing stand, machines, tools, and equipment. The design of a multifactor experiment for verifying the effect of analyzed parameters on the quality of the briquette is also discussed. Data from experiments are evaluated with the aid of several statistical procedures and methods, as well as the design of a mathematical model for compaction, which describes the effect of analyzed parameters and their interactions. In the final part of this chapter, the importance of these experiments is discussed. Additionally, the optimization process for the pressing chamber and the design of software for the optimisation of structural parameters for the pressing chamber are explained. Finally, prediction of the quality of the mould from setup parameters is discussed. This software can provide valuable information for biofuel producers and pressing machine designers.

3.1 Pressing Stand Design

The experiment requires the availability of appropriate equipment. Because the necessary experimental equipment was not directly available, I designed it. The design was kept simple, but it was ensured that the finished equipment could meet experimental requirements. First, it was necessary to solve the question of the administration of pressing force. The WPM hydraulic press (Fig. 3.1), which is available in the Institute of Applied Mechanics and Mechatronics, was employed for this design. On the basis of the workplace for this hydraulic press, we measured the maximum limits for the proposed pressing stand construction. Because the hydraulic press WPM is mounted vertically, the stand was adjusted. The hydraulic drive of the press enables the pressing force magnitude and pressing speed to be regulated. This press also provides the operator a simple configuration and readings for force. The maximum possible force applied by the press is 100 kN.

The pressing stand construction, which enables the measurement of several parameters, was also considered. Because the material's water content and fraction size are so-called material properties, they were not considered in the first phase of the experiment. However, it was thus necessary to design a construction that would enable the pressing temperature and other design parameters to be easily changed.



This was not the subject of our research, but for future purposes, it was considered for the institute to investigate these parameters. Fig 3.2 shows a 3D model of the pressing stand, and the constructed stand itself can be seen in Fig 3.3. The designed pressing stand allows for the study of the length of the pressing chamber, the effect of the conicity of the chamber walls, and the change of friction factor between the briquette material and the chamber to be investigated. After minor adjustments, it is also possible to study the effect of diameter change of the chamber.



Figure 3.1: Hydraulic press used in the experiment.

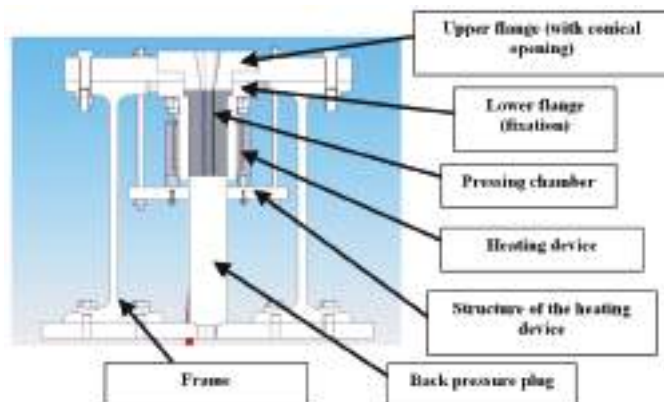


Figure 3.2: 3D section of the press stand and its basic components (Križan, 2009).

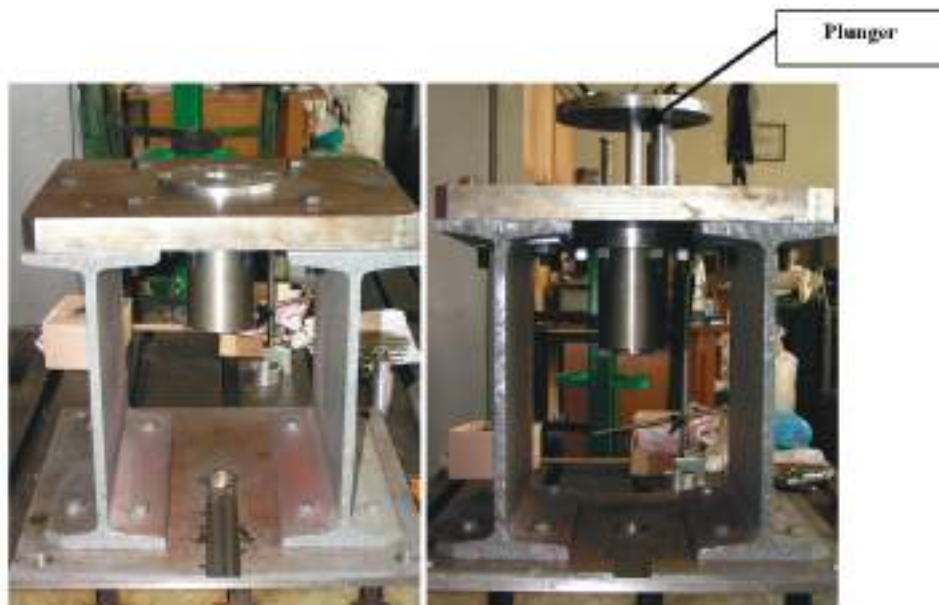


Figure 3.3: Constructed pressing stand before installing into the hydraulic press (Križan, 2009).

The next step was the design of a heating device for the pressing stand. With a continuous mode of compaction, temperature fluctuations arise as a result of the machine kinematics and friction between the pressing tool and material. But, in our case, this is excluded. The heating device provides heat to the chamber and thus heats the workspace while running the process. An additional requirement from the heating device was the knowledge and control of the temperature inside the chamber and a flexible response when changing this internal temperature. From the previous analysis, we know that the temperature inside the chamber during the pressing process affects the briquette. After finding an appropriate heating device manufacturer, three temperature sensors were installed into the chamber evenly along the length of the pressing opening, as can be seen in Fig. 3.4. Obviously, the preliminary calculation of heat transfer was also investigated. A heating coil, which is generally used in chemical laboratories while heating solvents in flasks, was employed as the heating element. The coil's power is 600W, and the coil was fixed to the bottom flange of the stand (Fig. 3.5). The heating coil is controlled by another heat sensor, which was fixed into the opening diagonally across the chamber, 2mm under the edge of the pressing opening (Fig. 3.4b and 3.5a). This setup provided the heating object used for the experiments.

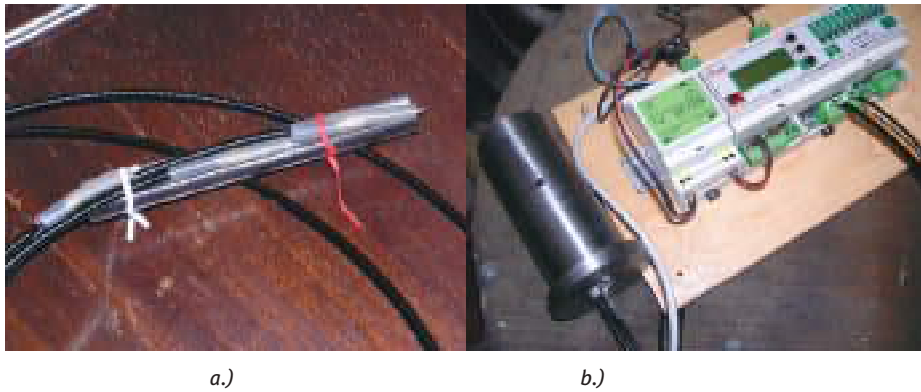


Figure 3.4: Test and verification of heat transfer– a.) sensor placement; b.) pressing chamber with mounted sensors (Križan, 2009).



Figure 3.5: Fixed heating coil– a.) mounted heat sensor; b.) connection of heating coil and sensors to the control unit (Križan, 2009).

Based on the results from a preliminary heat transfer test, a heating device with an intelligent heating coil was designed. It allowed for the smooth balance of heat fluctuations with high sensitivity to maintain the temperature around the nominal pressing temperature. This heating device supplies the chamber with heat according to a configured value within a few minutes. After constructing and mounting the heating system, the pressing stand was ready to meet the requirements of the experiment.

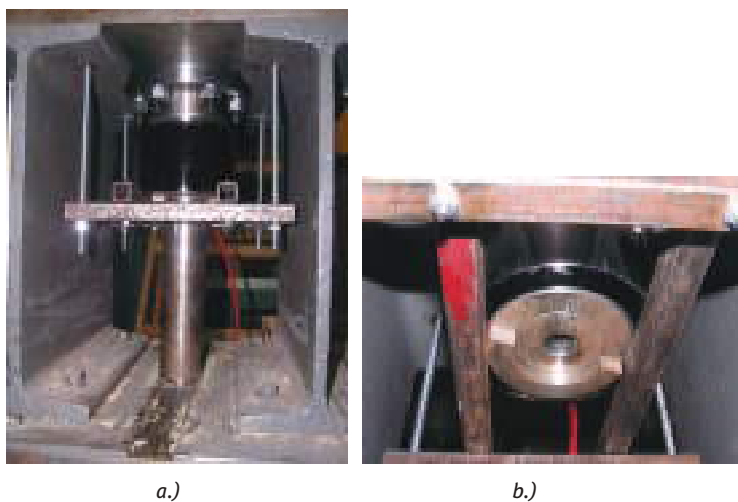


Figure 3.6: Designed heating device – a.) mounted heating device; b.) bottom view (Križan, 2009).



Figure 3.7: Experimental pressing stand prepared for the experiment (Križan, 2009).

3.2 Experiment Design and Description

It is necessary to design a set of experiments to verify the effects of analyzed parameters so that a mathematical model for compaction can be constructed. The model was aimed to incorporate four basic parameters and their mutual interactions affecting the resulting briquette. On the basis of the previous analysis, we recognize that it is necessary to design a multifactor experiment to study the effect of pressing pressure, temperature, size of entering fraction, and moisture content of the material. In this part of the chapter, we will describe the design and procedure for the experiments.

3.2.1 Experiment Design

If we consider a system with four parameters, it is possible to assume the input value as a function with four variables $\rho = f(p, T, w_r, L)$. Based on this concept and advice and recommendations given by experts in this area, the experimental procedure according to a centered composite design was designed.

Table 3.1: Centered composite design for the experiment requirements (Chudý & et al., 1999).

i	k1	k2	k3	k4
1	-1	-1	-1	-1
2	1	-1	-1	-1
3	-1	1	-1	1
4	1	1	-1	1
5	-1	-1	1	-1
6	1	-1	1	-1
7	-1	1	1	1
8	1	1	1	1
9	-1	-1	-1	1
10	1	-1	-1	1
11	-1	1	-1	-1
12	1	1	-1	-1
13	-1	-1	1	1
14	1	-1	1	1
15	-1	1	1	-1
16	1	1	1	-1

i	k1	k2	k3	k4
17	α -	0	0	0
18	α	0	0	0
19	0	α -	0	0
20	0	α	0	0
21	0	0	α -	0
22	0	0	α	0
23	0	0	0	α -
24	0	0	0	α
25	0	0	0	0

- design core

- star points

- central points

i – number of configurations
 k₁ – pressing pressure
 k₂ – pressing temperature
 k₃ – material moisture content
 k₄ – size of entering fraction

These designs belong to the most commonly used composite designs (Horníková, 2009). They consist of three parts:

1. Core of the design – composed of a total factorial experiment of 2^k (k =number of examined factors). If $k \leq 4$, it is necessary to use as a core the total experiment 2^k . In our case, $2^4=16$.
2. Star points – points that lie on the coordinate axes with a distance of $\alpha > 0$ from the center of the design. Their quantity is $2k$. In our case, it is $2k=2 \cdot 4=8$.
3. Central points – points in the center of design. In our case, we would suggest one central point

An appropriate selection of α and the number of central points can affect the properties of the designs.

Table 3.2: Set parameter levels (Křižan & Svátek, 2007; 2008).

factor	unit	- α	-1	0	1	α
pressure	(MPa)	63	95	127	159	191
temperature	(°C)	55	85	100	115	130
humidity	(%)	5	8	10	12	15
size	(mm)	0.5	1	2	4	4<

For specific and exact evaluation of the measured data, it was necessary to set the values for each examined parameter. The levels were defined with respect to compaction and the pressing stand. The experiment was performed with a total factorial of 2^4 (Wimmer & et al., 2001) according to Tab. 3.1 (orange fields—design core). To obtain the most precise experiment, the interval of validity is constricted. From the originally set levels of examined parameters (Tab. 3.2), the experiments on levels listed in Tab. 3.3 (where specific values of examined parameters are listed) were set.

3.2.2 Pressing Procedure and Apparatus Used

The aim of the experiments was to observe the quality of the briquette in relation to the examined factors (pressing pressure, pressing temperature, material moisture content, and the size of entering fraction). The quality of the briquette was evaluated according to resulting density. Due to the complexity of the experiments, only one raw material, pine sawdust, was considered. The experiments were carried out on the constructed pressing stand. At each set level, according to the centered composite

Table 3.3: Centered composite design with specific value of examined parameters (Križan & Svátek, 2007; 2008).

i	p (MPa)	T (°C)	w (%)	L (mm)
1	95	85	8	1
2	159	85	8	1
3	95	115	8	4
4	159	115	8	4
5	95	85	12	1
6	159	85	12	1
7	95	115	12	4
8	159	115	12	4
9	95	85	8	4
10	159	85	8	4
11	95	115	8	1
12	159	115	8	1
13	95	85	12	4
14	159	85	12	4
15	95	115	12	1
16	159	115	12	1

design, seven briquettes were made. For the purpose of further evaluation of the measured data (analysis of diameters, variance analysis, effect method, etc.), the literature indicates a minimum of six values to be obtained at each configuration level. The experimental procedure for compaction was as follows:

1. Material preparation

Because in the experimental plan there exist several levels of size and water content, a sieving analysis was conducted to ensure various values of fraction and moisture. For the sieving analysis, a sieving apparatus with different sizes of openings 0.5 , 1, 2, and 4 mm (Fig. 3.8) was employed.

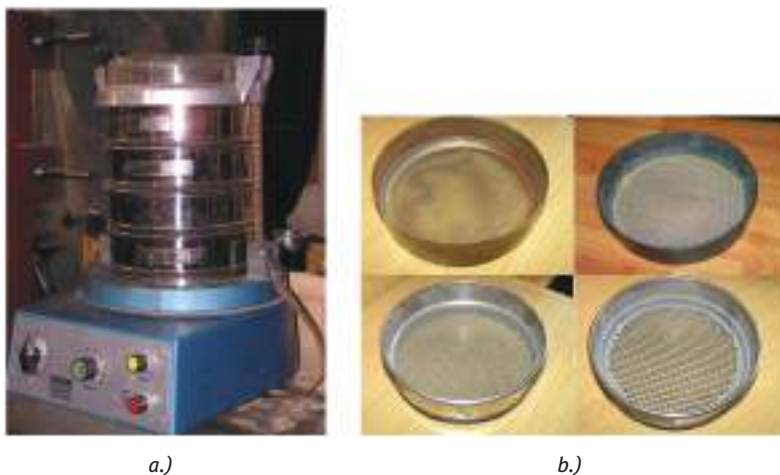


Figure 3.8: Tools used in sieving analysis– a.) sieving apparatus; b.) laboratory sieves.

Different moisture content values in the sawdust were obtained by drying in the laboratory (Fig. 3.9) and controlling specific values with the aid of a digital humidity meter GMH 3830 (Fig. 3.10).

2. Dosage and filling of the pressing chamber

Because the volume of the opening in the pressing chamber and the ratio of compaction are known, the amount of sawdust needed to fill the opening can be determined. This is how filling the opening with the same dosage of weighed sawdust was done. The pressing chamber is of cylindrical shape with a diameter of 20 mm. The principle of pressing is vertical; therefore, when filling the chamber, it is important to use a backpressure plug, which prevents free sawdust from exiting the chamber during pressing.



Figure 3.9: Laboratory dryer BINDER ED 23.



Figure 3.10: Digital humidity meter GMH 3830 with GSF 38 K probe for granular materials– a.) digital humidity meter; b.) measuring humidity.

3. Pressing procedure

The pressing process can begin when the material, which is prepared prior to loading according to the desired fraction size and moisture content, is inside the pressing chamber. During pressing, the experimental plan and configuration of the defined value levels of the examined parameters are closely followed. The control panel of the heating apparatus must indicate that the temperature inside the chamber is equal to the set temperature before filling the pressing opening with sawdust can begin (Fig. 3.11a.). Heating the pressing chamber is used to facilitate the plasticisation of lignin in the material's cellular structure, and mainly because it enables the needed decrease of pressure for obtaining the required quality of the briquette. After filling the pressing opening with sawdust, a pressing pressure according to the experimental plan is set (Fig. 3.11b.). After releasing of the pressure, the pressing opening is filled to the edges (Fig. 3.11c.). Subsequently, the sawdust is pressed into a compact form with a set pressing force according to the experimental plan (Fig. 3.11d.). After releasing the pressure, the backpressure plug is released, and the briquette is pushed outside of the pressing chamber (Fig. 3.11e.). In this way, one briquette is made at a time; for each configuration, according to the centered composite design, at least six briquettes for each setting has to be made. In order to fulfil this condition, seven briquettes per configuration were made.

4. Briquette dimensions, weight, and stability

After pressing the briquette, its diameter and length are measured. Using two separate callipers for each dimension prevents correlation of measured values, which would appear while calculating inaccuracies of the measurement. With the aid of a digital scale, the briquette is weighed (Fig. 3.12). The briquette is allowed to sit for

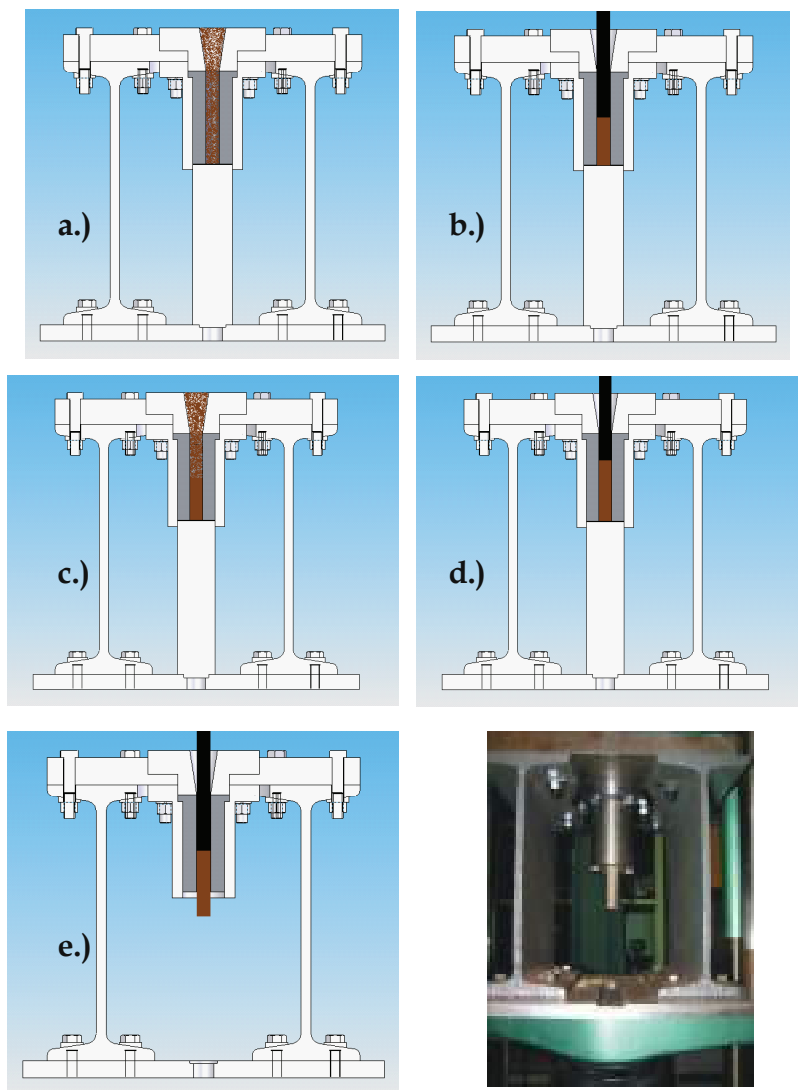


Figure 3.11: Pressing phases of wooden sawdust

a defined length of time to dilate and stabilize. After the stabilization period, the diameter, length, and weight of the briquette is measured again. These values are the foundation for determining the density according to DIN 52182. Densities obtained at one configuration according to the experimental plan are averaged and are used when evaluating the measured data.

The described procedure was repeated according to the designed experimental plan and centered composite design.



Figure 3.12: Used instruments– a.) digital caliper 1; b.) digital caliper 2; c.) digital scale KERN.

3.3 Processing Methods and Experiment Evaluation

With the procedure described in previous section, estimates were obtained for the measured and calculated values of density, which are listed in Tab. 3.4. In the left part of the table, the values of the parameters at which the experiments were performed according to the centered composite design are given. In Tab. 3.4, the values of measured parameters can be seen as $\rho_1, \rho_2, \dots, \rho_j$, where $j = 1, 2, \dots, r$ (in our case, $r = 7$), for experiments 1 to 16. When evaluating, only measured values which characterize the core of the experiment for the centered composite design $2^4 = 16$ are used (Tab.3.1). From these measured values, the selection averages are calculated (Chudý & et al., 1999; Palenčár & Jankura, 2005):

$$\bar{\rho}_j = \frac{\sum_{j=1}^r \rho_j}{r} \quad (3.1)$$

for $j = 1, 2, \dots, r$ (in our case, $r = 7$).

These selection averages are also listed in Tab. 3.4 and are the basis for experimental evaluation.

In this chapter, the processing methods and evaluation of the experiment are described. Initial information about the effect of a particular factor is obtained

Table 3.4: Table of experiment design and estimates of measured parameters for each value level of the experiment.

Number of measurement	A	B	C	D	ρ_1 (kg.dm ⁻³)	ρ_2 (kg.dm ⁻³)	ρ_3 (kg.dm ⁻³)	ρ_4 (kg.dm ⁻³)	ρ_5 (kg.dm ⁻³)	ρ_6 (kg.dm ⁻³)	ρ_7 (kg.dm ⁻³)	$\overline{r_j}$ (kg.dm ⁻³)	$s^2(p)$ (kg.dm ⁻³)
1	95	85	8	1	1,17716	1,16662	1,08542	1,16056	1,14979	1,13905	1,09569	1,13919	0,001258
2	159	85	8	1	1,08537	1,17830	1,19467	1,17326	1,14581	1,13802	1,18045	1,156558	0,001384
3	95	115	8	4	1,14225	1,20398	1,07769	1,21126	1,13990	1,21333	1,18382	1,167468	0,002513
4	159	115	8	4	1,18110	1,23233	1,22386	1,13300	1,22964	1,21867	1,18254	1,200171	0,001332
5	95	85	12	1	0,86704	0,82048	0,89251	0,85791	0,79096	0,68229	0,68661	0,79969	0,00727
6	159	85	12	1	1,13135	0,99180	1,10075	1,08047	0,90114	0,95539	0,89003	1,007282	0,009572
7	95	115	12	4	1,18522	1,13034	1,21985	1,13904	0,94520	1,13079	1,14360	1,127726	0,007584
8	159	115	12	4	1,18664	1,08373	1,18725	1,19630	0,97648	1,14318	1,17217	1,135111	0,006393
9	95	85	8	4	1,09419	1,11017	1,11536	1,07802	1,10997	1,10358	1,00922	1,088649	0,001384
10	159	85	8	4	1,11654	1,11338	1,13230	1,12979	1,05443	1,00981	1,01213	1,081203	0,002974
11	95	115	8	1	1,21957	1,21403	1,21330	1,19424	1,17393	1,24748	1,07308	1,190808	0,003207
12	159	115	8	1	1,25719	1,26455	1,24496	1,25577	1,21456	1,24060	1,17151	1,235596	0,001062
13	95	85	12	4	0,60222	0,65262	0,69318	0,80187	0,72564	0,79672	0,62350	0,699423	0,006349
14	159	85	12	4	0,95193	0,93156	0,93512	0,92283	0,75913	0,95180	1,01321	0,923658	0,006146
15	95	115	12	1	1,17740	1,05525	1,18092	1,16053	1,15152	1,26143	1,22984	1,173846	0,004268
16	159	115	12	1	1,27086	1,24116	1,20838	1,23363	1,17060	1,27736	1,25231	1,236333	0,001377

Labelling of measured parameters:

- A – pressing pressure p (MPa)
- B – pressing temperature T (°C)
- C – material moisture content w_r (%)
- D – size of entering fraction L (mm)

mostly with the aid of the multivariation diagram. In a multivariation diagram, the maximum and minimum values of a certain group are shown. The variability in one group is characterized by a certain range that corresponds to the length of the segment connecting both points. This diagram allows us to monitor level changes and variability at the same time. The multivariation diagram for our experiments is illustrated in Fig. 3.13.

When the multivariation diagram indicates differences in averages and variability on several levels, it is possible to use statistical tests for further verification. 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). Both these methods assume normal distribution of the observed character and the same variability for some groups, which indicates an insignificant difference between selection distribution and range. The second of these assumptions verifies whether the Bartlett's test, typically used in relation with ANOVA or by the control diagram of range (R-diagram) or for variance (s^2 -diagram), correspond by their character to ANOM.

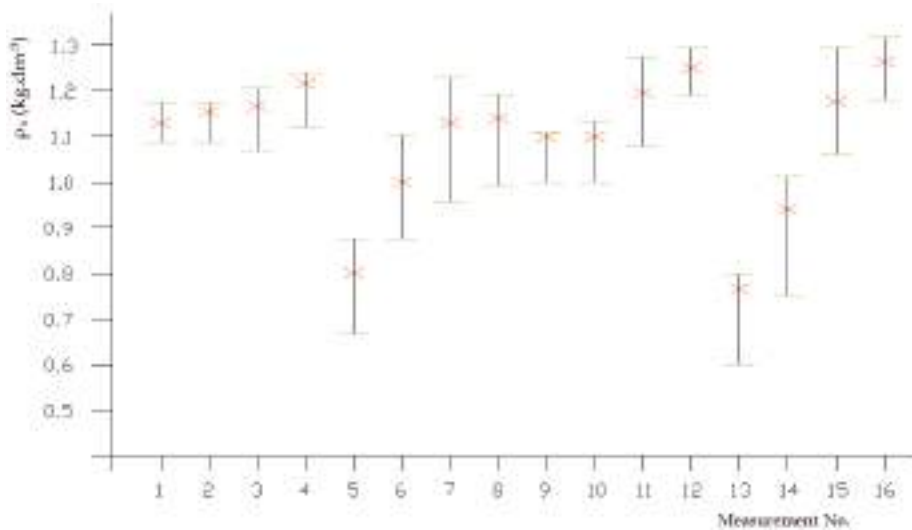


Figure 3.13: Multivariation diagram.

3.3.1 Average Analysis and Variance Equality Test Using Bartlett's Test

The first step in evaluation is to perform an average analysis and test the variance equality hypothesis using Bartlett's test (Palenčár & et al., 2001). This test can be employed when differences between selection variances s_i^2 or

the range R_i are insignificant. This test is based on k independent selections $y_{i1}, y_{i2}, \dots, y_{in_i}$, where $n_i > 6$; $i = 1, 2, \dots, k$.

Represented by:

$$s_i^2 = \frac{1}{n_i - 1} \cdot \left(\sum_{j=1}^{n_i} y_{ij}^2 - n_i \bar{y}_i^2 \right) \quad (3.2)$$

Where:

$$\bar{y}_i = \frac{1}{n_i} \sum_{j=1}^{n_i} y_{ij}, \quad i = 1, 2, \dots, k \quad (3.3)$$

$$s^2 = \frac{1}{n - k} \sum_{i=1}^k (n_i - 1) \cdot s_i^2 \quad (3.4)$$

$$C = 1 + \frac{1}{3 \cdot (k - 1)} \left(\sum_{i=1}^k \frac{1}{n_i - 1} - \frac{1}{n - k} \right) \quad (3.5)$$

$$\text{and } n = \sum_{i=1}^k n_i$$

In the following steps, the variance equality hypothesis is tested $H_0: \sigma_1^2 = \sigma_2^2 = \dots = \sigma_k^2$ by means of Bartlett's testing statistic:

$$B = \frac{1}{C} \cdot \left[(n - k) \ln s^2 - \sum_{i=1}^k (n_i - 1) \ln s_i^2 \right] \quad (3.6)$$

Hypothesis H_0 is rejected at a significance level of α , if: $B \geq \chi^2(k - 1, \alpha)$ resp. $B \geq \chi_{1-\alpha}^2(k - 1)$

When using this test, it is necessary that the selection ranges are sufficiently large. In the relevant literature, a condition is usually mentioned where its validity is $n_i > 6$ for each $i = 1, 2, \dots, k$. In this case, 16 measurements with 7 repetitions were made, meaning $n_i = 7$ for each $i = 1, 2, \dots, 16$. For this degree of calculation, with respect to the

large nature of the data, the program *STATGRAPHIC S Plus* was used. The results of the analysis are the sigma values for each measurement.

Outputs from STATGRAPHIC S Plus:

n.m.	n.values	average value ρ_j (kg.dm ⁻³)	variance $s^2(\rho)$ (kg.dm ⁻³)
Column_1	7	1.13919	0.00125764
Column_2	7	1.15656	0.00138368
Column_3	7	1.16747	0.00251308
Column_4	7	1.20017	0.001332
Column_5	7	0.79969	0.00727022
Column_6	7	1.00728	0.00957224
Column_7	7	1.12773	0.00758404
Column_8	7	1.13511	0.00639349
Column_9	7	1.08865	0.00138447
Column_10	7	1.0812	0.00297365
Column_11	7	1.19081	0.00320678
Column_12	7	1.2356	0.00106233
Column_13	7	0.6994	0.00634858
Column_14	7	0.923658	0.00614595
Column_15	7	1.17385	0.00426814
Column_16	7	1.23633	0.00137731
112*		1.08517**	0.026123**

(* means sum; ** means average;)

The next step was the evaluation of the hypothesis based on Bartlett's test.

Variance control results – *STATGRAPHIC S Plus* program output:

Cochran's test: 0.149394 P - value = 0.339438

Bartlett's test: 1.30317 P - value= 0.0650303

Hartley's test: 9.0106

The results of Bartlett's test obtained using the program *STATGRAPHIC S Plus* is the value P – the level of calculated significance, according to which the hypothesis H_0 is accepted or rejected on the level of specified significance $\alpha = 95\%$.

This means that, in order to accept the variance equality hypothesis, a value of P larger than 0.05 is necessary (Palenčár & et al., 2001). The analysis results returned values of $P = 0.55001$, which indicates that the hypothesis is acceptable. In this way, we satisfy one of the conditions for additional variance analysis using the method ANOVA. The selection can be regarded as homogeneous with respect to variability. This fact, based on the results, was also confirmed by the value of Cochran's tests.

3.3.2 Experiment Evaluation by the Variance Analysis Method (ANOVA)

The affecting factors are seen through the significant differences between selection averages (Chudý & et al., 1999; Palenčár & et al., 2001). This means that the fluctuation of the selection averages over the total average is larger than the fluctuation of each result within the selection. Variance analysis, contrary to selecting averages, compares two variance estimates describing the variability type. For this analysis, the same assumptions apply for the averaging analysis. The role of the variance analysis is to find the effect of one or more factors on the average value of independent, normally divided, random variables with the same variance.

The test was made in two steps:

- In the first step, the hypothesis $H_0: \mu_1 = \mu_2 = \dots = \mu_k$ is tested on a set level of significance α if H_0 is rejected.
- The groups that differ from each other are identified.

Table 3.5: Variance analysis table (Chudý & et al., 1999).

Variability source	Sum f squares	Degree of freedom	Average square	ratio F
Factor A	$S_A = \{1\} - \{3\}$	$l - 1$	$S_A^2 = S_A / (l - 1)$	S_A^2 / S_e^2
residual	$S_e = \{2\} - \{1\}$	$n - 1$	$S_e^2 = S_e / (n - 1)$	S_A^2 / S_e^2

After substituting in the formulas from Table 3.5, specific values can be obtained. In the table for the ANOVA method (F values), for the defined significance α , a certain number of degrees of freedom and critical value $F_{1-\alpha}$, are found. In another step, the ratio S_A^2 / S_e^2 is compared with the critical value $F_{1-\alpha}$ from the table. If the value of the ratio S_A^2 / S_e^2 is larger than that of the critical value of $F_{1-\alpha}$, the effects of this factor are considered to be significant (Palenčár & et al., 2001).

For this analysis, the program *STATGRAPHIC S Plus* was used once again. The range of examined parameters for the next step is defined as follows:

Table 3.6: Examined parameter range table.

Factor	Unit	Range
Pressure	(MPa)	95 - 159
Temperature	(°C)	85 - 115
Moisture content	(%)	8 - 12
Fraction size	(mm)	1 - 4

The next step was the evaluation process itself and the determination of each factor's significance with the aid of the program *STATGRAPHICS Plus*. The analysis results are listed in Table 3.7.

The effect of each parameter or its significance is given by the value of P – level of calculated significance, which we compared. The effect of the parameter is considered important at a significance level of P^* (95%); when the value of P is less than $(1-P^*)$, this means $P < 0.05$. From this, and based on the analysis results listed in Table 3.7, it is implied that the order of significance for each of the parameters are as follows: pressing temperature, material moisture content, pressing pressure, fraction size.

Table 3.7: Variance analysis table– results (Križan & Svátek, 2007; 2008).

Variability source	Sum of squares	Degrees of freedom	Average square	Ratio F	Value P	significance
A – pressure „ p “	0.0210523	1	0.0210523	3.40	0.0923	3 rd
B – temperature „ T “	0.13857	1	0.13857	22.37	0.0006	1 st
C – moisture content „ w_r “	0.0709649	1	0.0709649	11.46	0.0061	2 nd
D – fraction size „ L “	0.0107137	1	0.0107137	1.73	0.2152	4 th
Residual	0.0681389	11	0.00619445			
Sum	0.30944	15				

3.3.3 Experiment Evaluation with Factor Effect Method

For a closer determination, not only of the effect of each parameter but also of their mutual interactions, a factor effect method is used (Jarošová & Žaludová, 1997). The degree of influence of a factor on an observed variable is known as the factor effect, which is defined as the magnitude of response change caused by a change in the factor level. It is often referred to as the main factor effect, unlike the interaction effect.

If factor A' has two levels, then the main factor effect "A" is equal to the difference between the average response at one level and the average response at the second level (Jarošová & Žaludová, 1997). If factor "A" has more than 2 levels, we usually use the effect of (i) level term of factor "A," which means the difference between the average response at level " A_i " and the average of all the results from all the trials.

If the difference between response values at two levels of the factor "A" are the same for different levels of factor "B," this implies that there is an interaction between factors from "A" and "B" (referred to as "AB")(Jarošová & Žaludová, 1997).

The aim of this analysis is to determine the magnitude of each parameter's effect on the process. Proceeding based on the design matrix in Tab. 3.9, the parameters and interactions are assigned values of "+" and "-." The next step is the sum of each of the examined parameter values (in our case, its density) with the aforementioned signage to a certain column, separated by a separator, which at experiment 2^N is generally $2^{N-1}r$. The following experiment matrix considers the effect of each of the examined factors (p, T, w_p , L), as well as their interactions (pT, pw_p , pL, Tw_p , TL, w_pL , p Tw_p , pTL, pw_pL , Tw_pL , p Tw_pL). Factors with interactions are referred to according to the caption in Table 3.4, which is the established denotation.

According to the design matrix in Table 3.9, the magnitude of the main effects and their interaction are estimated. Estimated (calculated) values of each of the effects are shown in Table 3.9, and Fig. 3.14 illustrates the effect differences.

Table 3.8: The effect value of each parameter and their interaction.

B	C	BC	A	D	ABC	AC	AB
0.0465	0.0333	0.0254	0.0181	0.012938	0.012881	0.012065	0.008587
BCD	AD	ABCD	CD	ABD	ACD	BD	
0.00387	0.002897	0.002367	0.002005	0.000963	0.000661	0.000396	

The magnitude of each effect expresses the weight of significance, magnitude of each parameter effect, and their interaction. It is obvious that the largest effects are the pressing temperature, material water content, and their interaction, followed by the pressing pressure and the fraction size, and finally by several mutual interactions between the examined parameters.

In relation to the proposed design, it is necessary to decide which of the listed examined parameters and interactions are to be considered in the mathematical model.

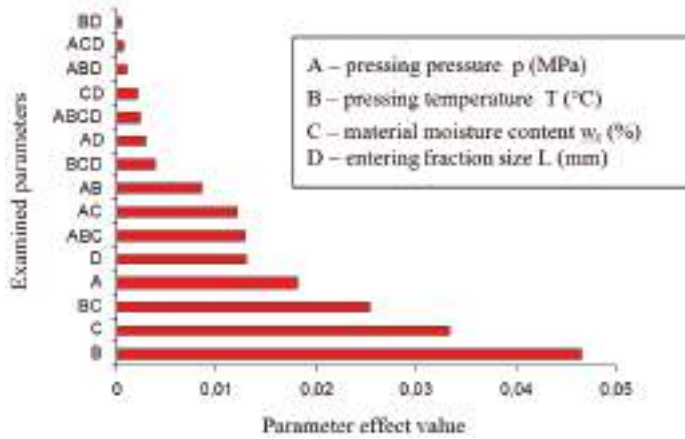


Figure 3.14: Pareto's effect diagram.

Table 3.9: Main factors with interaction table–design matrix.

Number of measurement	A	B	C	D	AB	AC	AD	BC	BD	CD	ABC	ABD	ACD	BCD	ABCD	ρ_j ($\text{kg} \cdot \text{dm}^{-3}$)
1	-1	-1	-1	-1	1	1	1	1	1	1	-1	-1	-1	-1	1	1.13919
2	1	-1	-1	-1	-1	-1	-1	1	1	1	1	1	1	-1	-1	1.156558
3	-1	1	-1	1	-1	1	-1	-1	1	-1	1	-1	1	-1	1	1.167468
4	1	1	-1	1	1	-1	1	-1	1	-1	-1	1	-1	-1	-1	1.200171
5	-1	-1	1	-1	1	-1	1	-1	1	-1	1	-1	1	1	-1	0.79969
6	1	-1	1	-1	-1	1	-1	-1	1	-1	-1	1	-1	1	1	1.007282
7	-1	1	1	1	-1	-1	-1	1	1	1	-1	-1	-1	1	-1	1.127726
8	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1.135111
9	-1	-1	-1	1	1	1	-1	1	-1	-1	-1	1	1	1	-1	1.088649
10	1	-1	-1	1	-1	-1	1	1	-1	-1	1	-1	-1	1	1	1.081203
11	-1	1	-1	-1	-1	1	1	-1	-1	1	1	1	-1	1	-1	1.190808
12	1	1	-1	-1	1	-1	-1	-1	-1	1	-1	-1	1	1	1	1.235596
13	-1	-1	1	1	1	-1	-1	-1	-1	1	1	1	-1	-1	1	0.699423
14	1	-1	1	1	-1	1	1	-1	-1	1	-1	-1	1	-1	-1	0.923658
15	-1	1	1	-1	-1	-1	1	1	-1	-1	-1	1	1	-1	1	1.173846
16	1	1	1	-1	1	1	-1	1	-1	-1	1	-1	-1	-1	-1	1.236333

3.4 Mathematical Model Design

Based on the measured data obtained from experiment, we sought to find a relationship between a dependent variable, such as the briquette density ρ , and some relevant independent variables, such as the pressing pressure p , pressing temperature T , material moisture content w_r , and entering fraction size L .

We supposed that the dependency is given by a continuous function of four variables $\rho=f(p,T,w_r,L)$, which is expanded into a Taylor's series. A mode, which includes linear variables and the interaction of independent linear variables, is assumed. The model can be simplified into a functional dependence:

$$\rho = f(A, B, C, D, AB, AC, AD, BC, BD, CD, ABC, ABD, ACD, BCD, ABCD) \quad (3.7)$$

Where:

A – Pressing pressure p (MPa)

B – Pressing temperature T (°C)

C – Material moisture content w_r (%)

D – Entering fraction size L (mm)

When the measured data represent a column vector of measured variable ρ and a column vector of configured values of variables A, B, C , and D , then the dependency (3.7) can be described in the shape of a multiple linear regression model.

$$\begin{aligned} \rho_i = & \beta_0 + \beta_1.A_i + \beta_2.B_i + \beta_3.C_i + \beta_4.D_i + \beta_5.A_iB_i + \beta_6.A_iC_i + \beta_7.A_iD_i + \beta_8.B_iC_i + \\ & + \beta_9.B_iD_i + \beta_{10}.C_iD_i + \beta_{11}.A_iB_iC_i + \beta_{12}.A_iB_iD_i + \beta_{13}.A_iC_iD_i + \beta_{14}.B_iC_iD_i + \beta_{15}.A_iB_iC_iD_i + \varepsilon_i \end{aligned} \quad (3.8)$$

for $i = 1, 2, \dots, 16$

where:

β_j – regressive parameters, for $j = 0, 1, \dots, 15$

ε_i – measurement error

Then for estimating the coefficients of the regression function, the following applies:

$$\hat{\beta} = (x^T x)^{-1} x^T \rho \quad (3.9)$$

where:

$\hat{\beta}$ – variable parameter polynom estimation vector

x – plan matrix

ρ – measured density value vector

In this relation (3.9), the weight of each of the measurements is not taken into consideration. This is therefore necessary to expand the relation by a covariance matrix $U(\rho)$. The previous relation then becomes:

$$\hat{\beta} = (x^T U^{-1}(\rho) x)^{-1} x^T U^{-1}(\rho) \rho \quad (3.10)$$

Covariance matrix $U(\rho)$, where m states the number of measurements, is as follows:

$$U(\rho) = \begin{pmatrix} u^2(\rho_1) & u(\rho_1, \rho_2) & u(\rho_1, \rho_3) & \cdot & \cdot & \cdot & u(\rho_1, \rho_m) \\ u(\rho_2, \rho_1) & u^2(\rho_2) & u(\rho_2, \rho_3) & \cdot & \cdot & \cdot & u(\rho_2, \rho_m) \\ \cdot & \cdot & \cdot & \cdot & \cdot & \cdot & \cdot \\ \cdot & \cdot & \cdot & \cdot & \cdot & \cdot & \cdot \\ \cdot & \cdot & \cdot & \cdot & \cdot & \cdot & u(\rho_{m-1}, \rho_m) \\ u(\rho_m, \rho_1) & u(\rho_m, \rho_2) & u(\rho_m, \rho_3) & \cdot & \cdot & u(\rho_m, \rho_{m-1}) & u^2(\rho_m) \end{pmatrix} \quad (3.11)$$

For a closer description of designing the model from the measured data, it is necessary to decide which of the variables in the relation (3.7) should be included in the model and which can be disregarded.

3.4.1 Regression Model Evaluation Criteria

Some variables have a negligible effect on the density (Fig. 3.14) and can consequently be disregarded. A valid criterion is necessary to select use for the evaluation and comparison of variables in the model and regression model, respectively. Currently no theories exist that provide a precise specification of variables in a model. Nevertheless, there are several criteria that are suggested for use when selecting variable specifications for evaluating and comparing several regression models.

Initially, the criteria used are based on the coefficient of multiple determination R^2 and corrected coefficient R^2 , referred to as R^2_{adj} . Coefficients are defined as follows (Palenčár & et al., 2001):

$$R^2 = \frac{SS_A}{SS_T} = 1 - \frac{SS_R}{SS_T} \quad (3.12)$$

$$R^2_{adj} = 1 - \frac{SS_R / (n - k - 1)}{SS_T / (n - 1)} \quad (3.13)$$

where:

SS_A – sum of squares between groups (regressive) (kg.dm³)

SS_R – sum of squares inside the groups (residual) (kg.dm³)

SS_T – total sum of squares (kg.dm³)

$(n-k-1)$ a $(n-1)$ – degrees of freedom

Coefficients of multiple determination R^2 and R^2_{adj} are based on the single factor variance analysis, which is a test of regression significance, or a test for determining if there is a linear relationship between variables y and the subset of regressive variables (plan matrix columns) x_1, x_2, \dots, x_k . Testing hypothesis $H_0: \mu_1 = \mu_2 = \dots = \mu_k = 0$, against the alternative H_1 : there results in at least one nonzero value of μ_j . The rejection of $H_0: \mu_1 = \mu_2 = \dots = \mu_k = 0$ means that at least one of the regressive variables x_1, x_2, \dots, x_k contributes significantly to the model. Significance test of regression is a general procedure used in linear regression. The total sum of squares of the measured variable SS_T is divided into 2 components:

$$SS_T = SS_A + SS_R \text{ (kg.dm}^3\text{)} \quad (3.14)$$

But first, it is necessary to calculate the sums of squares from the sum of values on the (i^{th}) selection of y_i and from the average values of this selection. The sum of squares between the groups is:

$$SS_A = \sum_{i=1}^k \frac{y_i^2}{n_i} - \frac{1}{n} y^2 \dots \text{ (kg.dm}^3\text{)} \quad (3.15)$$

Sum of squares inside the groups (residual) equals:

$$SS_R = \sum_{i=1}^k \sum_{j=1}^{n_i} y_{ij}^2 - \sum_{i=1}^k \frac{y_i^2}{n_i} \text{ (kg.dm}^3\text{)} \quad (3.16)$$

The total sum of squares equals:

$$SS_T = \sum_{i=1}^k \sum_{j=1}^{n_i} y_{ij}^2 - \frac{1}{n} y^2 \dots \quad (3.17)$$

The results needed for testing H_0 are listed in Tab. 3.10. Hypothesis H_0 is reject on the level of significance α if $F > F(k-1, n-k, \alpha)$.

Other criteria also exist that can help in deciding whether including an additional explanatory variable would improve the model; in other words, other criteria can

help determine which of the models we should prefer. One such criterion is known as Akaike's information criteria (AIC).

Table 3.10: Single factor variance analysis (Palenčár & et al., 2001).

Variability source	Sum of squares (kg.dm ⁻³)	Degrees of freedom	Average square (kg.dm ⁻³)	Ration F
Regressive sum	SS_A	$k - 1$	$MS_A = \frac{SS_A}{k - 1}$	$F = MS_A / MS_R$
Residual sum	SS_R	$n - k$	$MS_R = \frac{SS_R}{n - k}$	$F = MS_A / MS_R$
Total sum	SS_T	$n - 1$	-	-

AIC is not a test of models on which basis we test the hypothesis; AIC is a tool for model selection (Akaike, 1981; Beck & Arnold, 1977). The measured group of values or the designed models can be arranged and evaluated according to AIC with a distinction that the model with smallest AIC is the most appropriate. On the basis of AIC, it is possible to evaluate, for example, whether the best models are similar and whether they are much better than the rest of the models. It is not possible to set a value according to which a model is rejected using AIC.

Generally, AIC is calculated as follows (Akaike, 1981; Bozdogan, 1987):

$$AIC = 2.k_m - 2.\ln(L_p) \quad (3.18)$$

where:

k_m – number of parameters in the model

L_p – maximum value of the probable function for the estimated model

It is considered that model errors are normal and independently distributed. This method (AIC) tries to find a mode that best describes and explains the measured values and also includes at least 3 parameters. AIC decides how a model best describes the measured values. The AIC value only sorts and defines which model is best compared to the possible alternatives in the considered set of models.

AICc is AIC with a second degree of correction for samples with a small number of values and is calculated by (Akaike, 1981; Bozdogan, 1987):

$$AICc = AIC + \frac{2k(k+1)}{n-k-1} \quad (3.19)$$

Where: $n-k$ = degrees of freedom.

The quality of estimated models is usually also measured by the RMSE criterion (Root mean squared error). RMSE is a very good test for model accuracy. The RMSE value is the root of the average sum of squares of the residuals (Palenčár & et al., 2001). The average sum of squares of the residuals is the sum of squares divided by the difference between the number of observations and the number of estimated parameters. Then, the best model from the designed alternatives is the model that has the smallest standard deviation of the residuals.

Based on the three previously described criteria, we will compare and evaluate the designed models with the variables.

3.4.2 Regression Model Creation and Selection

This section will focus on the creation and selection of an appropriate regression model based on the criterion mentioned in previous chapter. For the selection and evaluation of models, the software JMP 8 is used. This software calculates the values for each of the described criteria for each designed mode. Based on the results obtained from the factor effect method selected, the parameter effect values in Model 1 and Models 2 and 3, respectively, are shown in Fig. 3.15..

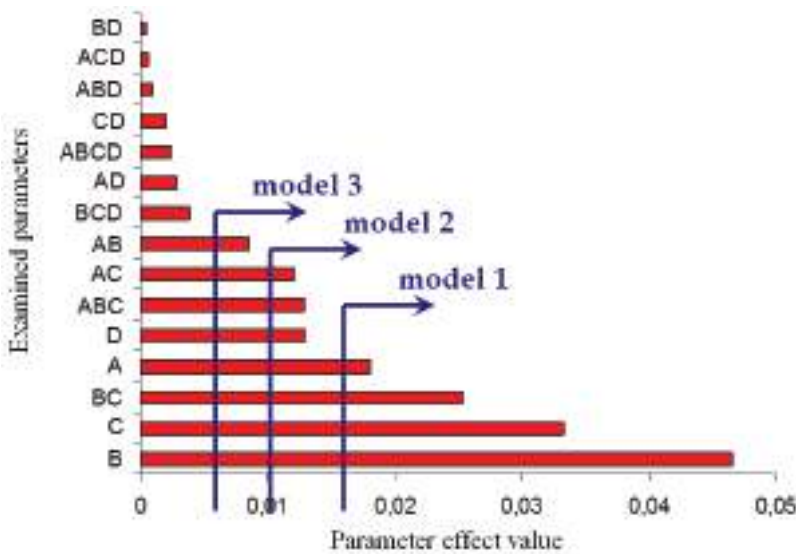


Figure 3.15: Variable and regression model design selection example.

With the aid of JMP 8, we have calculated the values for the observed criteria (Tab. 3.11). Model 1, which considers 4 factors and is based on measurement results having the largest effect, is described by the list of values of measured data with 87.9 % accuracy. This accuracy cannot be disregarded, but to improve accuracy, more parameters were added to the model (Model 2). The model's accuracy was indeed increased with the addition of more parameters. Additionally, the criterion *RMSE* implies that Model 2 is better than Model 1. However, the *AICc* criterion implies that Model 2 is worse compared to Model 1. An additional parameter (Model 3) was thus added, and this designed model was evaluated by all three criteria. These three criteria indicate that Model 3 is the most accurate..

Subsequently, this simplified approach for design was also applied to select a regression model that considers all the variables according to Equation 3.7. The Software JMP 8 created models from all possible combinations of variables from Equation 3.7 and calculated the values of the examined criteria for each mode. In Tab 3.11, the models and their coefficients are listed. Appropriate models were selected based on the highest values of the coefficient R^2 , the lowest values of *AICc*, and the lowest values of the coefficients for *RMSE*.

Table 3.11: The best models calculated with JMP 8 on the basis of the evaluating criterion.

Model	Parameters	Number of parameters	R^2	<i>AICc</i>	<i>RMSE</i>
model 1	B,C,BC,A	4	87.9%	-30.1238	0.058448
model 2	B,C,BC,A,D,ABC,AC	7	96.8%	-24.8346	0.035139
model 3	B,C,BC,A,D,ABC,AC,AB	8	99.3%	-32.7996	0.017764
model 4	C,AB,AC,BC,ABCD	5	94.3%	-35.5702	0.041982
model 5	B,C,AB,AC,BC, ABCD	6	96.1%	-33.239	0.036413
model 6	B, C, BC, A, D	5	91.3%	-28.8272	0.05183

All of the mentioned models are solved with a certain accuracy describing the measured data. Each of the mentioned models could be used depending on specific conditions described by the models. For selecting a model, which will be discussed later, it is necessary to define the initial conditions; if the simplest model from a statistical point of view is desirable, the model with the least number of parameters would be chosen (Model 1).

Since the model is designed for the densification process (pressing), the model must include all of the examined parameters (A, B, C, D) because the final model must describe the effects of all four examined parameters on the resultant density of the

briquette. In terms of the densification process, the models should also include the mutual interaction of the parameters, which have a significant effect on the resultant density of the briquettes. Selecting an appropriate model requires an objective evaluation and comparison of each of the values of the examined criterion for each group of models. That is why that, from the six models, Model 3 is ultimately chosen. Model 3 describes the list of measured data with 99.3 % accuracy, and the value of the *RMSE* coefficient is the lowest. The *AICc* criterion is not the lowest for this model, but considering the values of the coefficients R^2 and *RMSE*, it is still the best choice. Model 3 includes all four examined parameters (A, B, C, D) and their interactions, which have a significant effect on the density of the briquette (BC). This model also includes the interaction of some important parameters (ABC, AC, and AB). Based on what has previously been discussed, the dependency in Equation 3.7 can now be rewritten to yield the following form:

$$\rho = f(A, B, C, D, AB, AC, BC, ABC) \quad (3.20)$$

Equation 3.8 can also be rewritten to have a multiple linear regression model form:

$$\begin{aligned} \rho_i = & \beta_0 + \beta_1.A_i + \beta_2.B_i + \beta_3.C_i + \beta_4.D_i + \\ & + \beta_5.A_iB_i + \beta_6.A_iC_i + \beta_7.B_iC_i + \beta_8.A_iB_iC_i + \varepsilon_i \end{aligned} \quad (3.21)$$

for $i = 1, 2, \dots, 16$

where:

β_j – are regressive parameters, for $j = 0, 1, \dots, 8$

ε_i – measurement error

3.4.3 Mathematical Model Design

This section will deal with the calculation or estimation of regressive parameters β_j , for $j = 0, 1, \dots, 8$. Once again, the JMP 8 software was employed, with which the values of the regressive parameters β_j are calculated for each regression model. For Model 3, and according to Equation 3.21, the regressive parameters have estimated values shown in Tab. 3.12. In the left column, there are variables that contain the designed and selected model. In the second column from the left, regressive parameters attributed to variables in the first column are listed. In the third column from the left, specific estimated values of the regressive parameters are shown. Also here we see the standard estimated values of the regressive parameters and the values of the upper and lower limit of 95% the reliability interval. Considering the values of the regressive parameters, we can rewrite Equation 3.21 into the form 3.22.

Table 3.12: Estimated regressive parameters values using JMP 8 software.

Model variables	Regressive parameters	Estimated value	Standard error	Lower limit 95,0% interval	Upper limit 95,0% interval
	β_0	4.98371	0.236318	4.52053	5.44688
A	β_1	-0.0261781	0.00189126	-0.029885	-0.0224713
B	β_2	-0.0410292	0.00228295	-0.0455038	-0.0365547
C	β_3	-0.620594	0.0235536	-0.666758	-0.574429
D	β_4	-0.015446	0.00152757	-0.01844	-0.012452
AB	β_5	0.000228845	0.0000185268	0.000192533	0.000265157
AC	β_6	0.0031851	0.000188348	0.00281595	0.00355426
BC	β_7	0.00528717	0.000230218	0.00483595	0.00573839
ABC	β_8	-0.0000273004	0.00000185582	-0.0000309378	-0.000023663
sigma	sigma	0.00586314	0.00110664	0.00405013	0.00848773

$$\begin{aligned} \rho_i = & 4,98371 - 0,0261781.A_i - 0,0410292.B_i - 0,620594.C_i - \\ & - 0,015446.D_i + 0,000228845.A_i.B_i + \\ & + 0,0031851.A_i.C_i + 0,00528717.B_i.C_i - 0,0000273004.A_i.B_i.C_i + \varepsilon_i \text{ (kg.dm}^3\text{)} \end{aligned} \quad (3.22)$$

for $i = 1, 2, \dots, 16$

This equation is not the final mode; it is a multiple linear regression model with estimated values of regressive parameters. It is necessary to further design the mathematical model. For calculating and designing a mathematical model, JMP 8 software is once again utilized, which designed the model in the following form:

$$\rho = e^{\left(\begin{array}{l} 4,98371 - 0,0261781.A - 0,0410292.B - 0,620594.C - 0,015446.D + 0,000228845.A.B + 0,0031851.A.C + \\ 0,00528717.B.C - 0,0000273004.A.B.C \end{array} \right)} \text{ (kg.dm}^3\text{)} \quad (3.23)$$

Substituting the notation of the variables or the examined parameters we can rewrite the relation to:

$$\rho = e^{\left(\begin{array}{l} 4,98371 - 0,0261781.p - 0,0410292.T - 0,620594.w_r - 0,015446.L + 0,000228845.p.T + \\ 0,0031851.p.w_r + 0,00528717.T.w_r - 0,0000273004.p.T.w_r \end{array} \right)} \text{ (kg.dm}^3\text{)} \quad (3.24)$$

Equation 3.24 can be rewritten into the following form:

$$\ln \rho = 4,98371 - 0,0261781.p - 0,0410292.T - 0,620594.w_r - 0,015446.L + 0,000228845.p.T \\ + 0,0031851.p.w_r + 0,00528717.T.w_r - 0,0000273004.p.T.w_r \quad (3.25)$$

Then, using the designed mathematical model (according to 3.24), the density values at levels according to the experimental plan in (Tab. 3.13) are calculated and compared with the densities of the briquettes obtained from experiments at these configurations.

The following graph (Fig. 3.16) illustrates the comparison of the experimentally measured density values with the calculated density values. In this graph, it can be seen that the characters for both are almost identical; this comparison proves that the proposed procedure was appropriate when selecting a regression model based on the criteria R^2 , $AICc$, and $RMSE$.

Table 3.13: Comparing briquette densities obtained from the experiments and calculated with model.

n.m.	p (MPa)	T (°C)	w (%)	L (mm)	ρ_n experiment (kg.dm ⁻³)	ρ_n from model (kg.dm ⁻³)
1	95	85	8	1	1.139	1.138
2	159	85	8	1	1.157	1.152
3	95	115	8	4	1.167	1.163
4	159	115	8	4	1.200	1.201
5	95	85	12	1	0.799	0.797
6	159	85	12	1	1.007	1.007
7	95	115	12	4	1.128	1.125
8	159	115	12	4	1.135	1.176
9	95	85	8	4	1.089	1.087
10	159	85	8	4	1.081	1.100
11	95	115	8	1	1.191	1.218
12	159	115	8	1	1.236	1.258
13	95	85	12	4	0.699	0.761
14	159	85	12	4	0.924	0.961
15	95	115	12	1	1.174	1.178
16	159	115	12	1	1.236	1.231

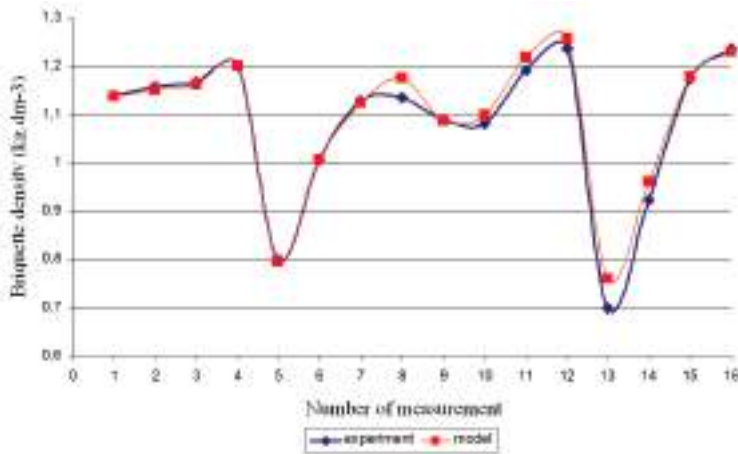


Figure 3.16: Graphic comparison of briquette densities obtained from experiments and calculated from the regression model.

3.4.4 Measurement Errors

Measurement error is a parameter related to the results of the measurement, which characterises the variance of measured values. Error is included in the measurement results, the values taken by the used instrument, the values of used constants, and so on. Each effect registered in the measurement process or somehow affecting the process brings a certain level of inaccuracy to the measurement itself. These inaccuracies cause errors in the resultant density of the briquette. We can say that the error of the measured result reflects defects in the perfect knowledge of the density value. Phenomena that contribute to error so that the results cannot be characterized by one value are called error sources.

The basic quantitative error characteristic is the standard error u . Standard error is equal to the standard deviation of the respective probability distribution as it is considered that the measured values and errors have a probability distribution (Chudý & et al., 1999).

Errors from each source can be evaluated using two basic methods (Chudý & et al., 1999):

- Errors from statistical methods from measured data are known as Error defined by method A (Errors type A) – u_A ;
- Errors from any other source, defined as errors by method B (Errors type B) – u_B .

Contrary to errors evaluated by method A, where the causes of the errors are generally considered as unknown, the errors evaluated by method B are from known or

unidentifiable sources. A characteristic sign of the errors evaluated by method A is that with more measurements, their values decrease. Errors evaluated by method B are independent to the number of measurements. It is necessary to remember that the errors evaluated by both methods are equivalent and differ only in the manner of evaluation; however, in terms of magnitude, they differ and some can be neglected.

Combining the standard error evaluated by method A and standard error evaluated by method B yields the total (combined) standard error u_c (Chudý & et al., 1999). Standard errors constitute an interval covering the real value of the measured variable with a relatively small probability (around 66%). Usually, we require a value of error that creates an interval with a larger probability of covering the real value. That is why the expanded error U is used:

$$U = k \cdot u_c \quad (3.26)$$

Procedure for evaluating type A

Evaluation type A of standard error, as has been mentioned before, is a method of evaluation with the aid of statistical analysis of the measured data. The statistical evaluation depends on the model of measurement. In the case of repeated direct measurements, it is about the statistical processing of values of repeated direct measurements. Most frequently, we consider that the measured values are obtained with independent measurements under the same conditions. This means that, if we have n measured values x_1, x_2, \dots, x_n , the measured values are the realization of n independent equally precise measurements of one variable. Then the following is true:

- estimated values of measured variables according to the relation:

$$\bar{x} = \frac{1}{n} \sum_{i=1}^n x_i \quad (3.27)$$

- standard error is determined by method A; this estimation is equal to the standard variation of the arithmetic average. Thus:

$$u(x) = s(\bar{x}) = \frac{s(x)}{\sqrt{n}} = \sqrt{\frac{1}{n(n-1)} \sum_{i=1}^n (x_i - \bar{x})^2} \quad (3.28)$$

Procedure for evaluating type B

As was mentioned before, the evaluation of error using method B is everything but the statistical analysis of measured values. Amongst the most frequent way of evaluating by type B are:

1. If the certificates, documentation from the manufactures or other sources, list the expanded error U and the expansion coefficient k , then method B for evaluating the standard error $u(z_i)$ from the given source Z_i lies in the equation:

$$u(z_i) = \frac{U}{k} \quad (3.29)$$

2. If $2U$ is the length of the interval with a confidential probability (95%, 99%, 99.73%), and it can be said that, when determining this interval, the normalized normal distribution was considered, standard error (z_i) from the source Z_i is determined from the following equation:

$$u(z_i) = \frac{U}{k_p} \quad (3.30)$$

where k_p is the expansion coefficient, which equals the quantile of normalized normal distribution for a probability of P_p ($k_p=1,96$ pre $P_p=95\%$; $k_p=2,58$ pre $P_p=99\%$; $k_p=3$ for $P_p=99.73\%$).

3. It is possible to estimate only the boundaries in which the values of the variable are located under the effect of the given source at almost 100 percent. For this estimation, we proceed as follows:
 - estimate the rate of change (error) $\pm z_{i\max}$ of the nominal value of the variable related to the considered source Z_i , which is very unlikely (practically impossible);
 - the probability distribution of the error is evaluated in this interval, and its approximation is determined;
 - standard error of the considered source $u(z_i)$ is calculated from the relation:

$$u(z_i) = \frac{z_{i\max}}{k} \quad (3.31)$$

where k is the value related to the selected approximation of the probability distribution:

- for normal distribution (N) $k = 3$ ($k = 2$);
- for equal (right-angled) distribution (R);
- for triangular distribution (S) $k = 2,45$;
- for bimodal distribution (U) $k = 1$.

The approximation with normal distribution is used if deviations from nominal values of small values may occur more often, and the resulting increase in probability decreases value.

The approximation with equal distribution is used if the probability of occurrence of any error in the interval is the same $\pm z_{imax}$. This approximation is the most frequently used because it common to not have enough knowledge about the probability of error occurrence distribution, and thus some of the errors cannot be biased. This distribution is considered in this work.

Triangular distribution approximation is used just like the normal one.

Bimodular distribution approximation can be used, for example, for measurement instruments, which the manufacturer sorts into several accuracy classes (classification of measurement instruments).

The aforementioned methods of evaluation (Type B) of the standard error of course do not constitute all possibilities. Measurements in practice can vary, and it is very important for each situation to closely consider all circumstances that can act as an error source.

While using a numerical measurement instrument, the source of error is the last valid resolution number. This means that, despite the fixed nature of the value after repeated measurements, the error is not zero. Error estimation in this case is based on the consideration of equal probability distribution in the interval restricted by the resolution $\delta(z_p)$ of the given instrument. In the case of analog measurement instruments, the readability is usually given by the value of the segments scale. Usually, it has also a significant effect on the instrument hysteresis.

Determining standard errors when measuring

We considering the case when the value of Y (output value), which interests us, is a function f of values X_1, X_2, \dots, X_m . Values X_1, X_2, \dots, X_m (input values) represent a directly measured value (affecting values, corrections, physical constants, etc.), so values that can be directly measured or have known estimations, errors, and covariances from other sources. We can write:

$$Y = f(X_1, X_2, \dots, X_m) \quad (3.32)$$

where f is a known function.

Estimate y of output value Y is determined from the equation:

$$y = f(x_1, x_2, \dots, x_m) \quad (3.33)$$

where x_1, x_2, \dots, x_m are the estimates of the input values X_1, X_2, \dots, X_m .

Estimate y of value Y error indicates that the estimates x_1, x_2, \dots, x_m are uncorrelated. From the relation (if we linearize the function f by expanding it into a Taylor's series and neglect higher terms) we get:

$$u^2(y) = \sum_{i=1}^m A_i^2 u^2(x_i) \quad (3.34)$$

Where, for coefficients (sensitivity, conversion) A_i , it is true that:

$$A_i = \left| \frac{\partial f(X_1, X_2, \dots, X_m)}{\partial X_i} \right|_{X_1=x_1, \dots, X_m=x_m} \quad (3.35)$$

The designed mathematical model in this manuscript (Equation 3.24) can be written in its basic form as $\rho = f(p, T, w_p, L, pT, pw_p, Tw_p, pTw_p)$, where ρ is the density of the briquette, and the values for p, T, w_p, L and their interactions are configured and therefore known to affect the resultant observed value of ρ . The observed value ρ is calculated according to a known equation for density (2.13) and an equation for calculating the cylinder volume. Based on what has been mentioned, and after adapting the relations to our case, we are able to rewrite relation (3.32) into the following form

$$\rho = \frac{m_n}{V_n} = \frac{m_n}{\pi \cdot \frac{d_n^2}{4} \cdot L_n} \quad (3.36)$$

Throughout the experiments, after pressing, values m_n, d_n , and L_n , are measurable, which means that we make an indirect measurement of one value. The measurements do not consider covariances as the measured value estimates are uncorrelated because the values of m_n, d_n , and L_n are measured with a different instrument. With these considerations, Equation 3.34 and Equation 3.35 can be written for this case as follows:

$$u\rho = \sqrt{\left(\frac{\partial \rho}{\partial m_n}\right)^2 u_{m_n}^2 + \left(\frac{\partial \rho}{\partial d_n}\right)^2 u_{d_n}^2 + \left(\frac{\partial \rho}{\partial L_n}\right)^2 u_{L_n}^2} \quad (3.37)$$

where:

u_{m_n} is the total combined standard error of the instrument (scale) for measuring the value m_n (weight of briquette)

u_{dn} is the total combined standard error of the instrument (calliper 1) for determining the value d_n (briquette diameter);

u_{Ln} is the total combined standard error of the instrument for measuring the value of the mold L_n (briquette length);

Sometimes, it is advantageous to determine the errors of estimates y of output value Y by methods A and method B separately. Then, the total (combined) standard error will be given with the relation (if we do not consider the dependency between the sources of error evaluated by method type A and method type B):

$$u(y) = \sqrt{u_A^2(y) + u_B^2(y)} \quad (3.38)$$

Based on Equation 3.38 for this case, it is true that:

$$u_{mn}^2 = u_{Amn}^2 + u_{Bmn}^2 \quad (3.39)$$

$$u_{dn}^2 = u_{Adn}^2 + u_{Bdn}^2 \quad (3.40)$$

$$u_{Ln}^2 = u_{ALn}^2 + u_{BLn}^2 \quad (3.41)$$

Since there were 7 measurements at 16 different configurations, Equation 3.27 can be written as follows:

$$\bar{m}_n = \frac{1}{7} \sum_{i=1}^7 m_{ni} \quad (3.42)$$

$$\bar{d}_n = \frac{1}{7} \sum_{i=1}^7 d_{ni} \quad (3.43)$$

$$\bar{L}_n = \frac{1}{7} \sum_{i=1}^7 L_{ni} \quad (3.44)$$

Then, considering relations 3.42-3.44, we can then rewrite the standard errors determined by method A based on Equation 3.28 and calculate it according to the following equations:

$$u_{Amn} = \sqrt{\frac{1}{7(7-1)} \sum_{i=1}^7 (m_{ni} - \bar{m}_n)^2} \quad (3.45)$$

$$u_{Adn} = \sqrt{\frac{1}{7(7-1)} \sum_{i=1}^7 (d_{ni} - \bar{d}_n)^2} \quad (3.46)$$

$$u_{ALn} = \sqrt{\frac{1}{7(7-1)} \sum_{i=1}^7 (L_{ni} - \bar{L}_n)^2} \quad (3.47)$$

For the standard error determined by method B, it is true that:

$$u_B^2(y) = u_{B1}^2(y) + u_{B2}^2(y) + \dots \quad (3.48)$$

where:

- $u_{B1}^2(y)$ is the instrument error, whose value is according to a certification less than or equal to δ_{dov} . Assuming equal distribution to be

$u_{B1}^2(y) = \frac{\delta_{dov}^2}{\sqrt{3}}$, according to Equation 3.31.

- $u_{B2}^2(y)$ is the error of readability and so on, but this error is neglected.

We determine the standard error type B from the following Equations 3.49-3.51. The values δ_{dov} of each instrument was listed by the manufacturer in the attached certificates. Then, the standard error type B will have values as follows:

$$u_{Bmn}^2 = \frac{\delta_{dov}^2}{\sqrt{3}} = \frac{0,02}{\sqrt{3}} = 0,00013333 \quad (3.49)$$

$$u_{Bdn}^2 = \frac{\delta_{dov}^2}{\sqrt{3}} = \frac{0,025}{\sqrt{3}} = 0,00020832 \quad (3.50)$$

$$u_{BLn}^2 = \frac{\delta_{dov}^2}{\sqrt{3}} = \frac{0,02}{\sqrt{3}} = 0,00013333 \quad (3.51)$$

Sensitivity coefficients coming from relation 3.37 are used to calculate according to relation 3.35, and they will have the following form:

$$A_{mn} = \frac{\partial \rho}{\partial m_n} = \frac{4}{\pi \cdot d_n^2 \cdot L_n} \quad (3.52)$$

$$A_{d_n} = \frac{\partial \rho}{\partial d_n} = - \frac{8.m_n}{\pi.d_n^3.L_n} \quad (3.53)$$

$$A_{L_n} = \frac{\partial \rho}{\partial L_n} = - \frac{4.m_n}{\pi.d_n^2.L_n^2} \quad (3.54)$$

On the basis of previous Equations 3.42-3.54, all of the coefficients and values needed for expressing the standard error type A and B are calculated according to Equations 3.39-3.41. Subsequently, the errors of estimation of the observed values ρ_n at each of the 16 configurations according to Equation 3.37 are calculated. The calculated errors are listed below in the table of errors.

Table 3.14: Table of calculated measurement errors for each configuration.

n.m.	ρ_n (kg.dm ⁻³)	u_ρ (kg.dm ⁻³) .10 ⁻³
1	1.134889752	0.0482210
2	1.156558042	0.0562181
3	1.16746774	0.186039
4	1.205601254	0.0466928
5	0.799690152	0.306733
6	1.007281758	0.184125
7	1.127725754	0.0832159
8	1.135110519	0.12598
9	1.088648941	0.156747
10	1.081203369	0.187254
11	1.190807876	0.187356
12	1.235596	0.212271
13	0.754723313	0.103315
14	0.960492782	0.201452
15	1.173846102	0.132002
16	1.236332914	0.197956

4 Design of Application Software for Setting Technological Parameters and Briquette Quality Estimation According to the Configured Parameters

To design a simple mathematical model that is also user-friendly, it was necessary to design a user friendly tool that the model would use. To fulfill this requirement, simple application software was created to function based on the derived mathematical model. The basic requirements for the software include: an overview of the environment, user-friendliness, report generation, and an easy interface with a user's manual.

The application software was designed in *DELPHI* and with minimal hardware requirements for using the application software (running on Windows 95 or 98, 128 MB of memory (RAM) and 5MB of free disk space required). The designed application software operates under Windows XP or Windows 7 with no problem. The user environment for the application software with basic descriptions can be seen in the Figures 4.1 and 4.2. The software environment constitutes several basic parts (Fig. 4.2).

Component part description of the application software:

1. Application control– contains options such as “start”(after entering inputs, the function starts analysis), “print protocol”(prints protocol), “Quit”(quits the application software), and basic information about the application software.
2. Material configuration – enables the selection of input material. Currently, the configuration is limited only to pine. As previously described, this raw material was used in the enclosed experiments and the foundation to which the mathematical model was constructed. Expansion of this mathematical model to include spruce, oak, beech, and other wood materials has been planned.
3. Entering input parameters – enables the entrance of input values of the examined parameters in listed units. In this part of the interface, the desired density value of the briquette is displayed after running the analysis. The values of each of the parameters can be selected only from intervals, which are listed in the information part.
4. Information part – here, the value intervals for each of the examined parameters, from which the user can select, are listed. Momentarily, the listed intervals of values are those which are valid for the mathematical mode and for the executed experiment. In the future, the interval will be expanded.
5. Error messages– warns the user about incorrect input values. For example, if the input value is outside the interval range, the density cannot be calculated.
6. Illustrative display of the entering fraction (sawdust) and product. Interestingly, the pictures change according to the configured value for the fraction size.
7. Illustrative display of the experimental press stand with the heating device.





Figure 4.1: Application software view (Křižan, 2009).

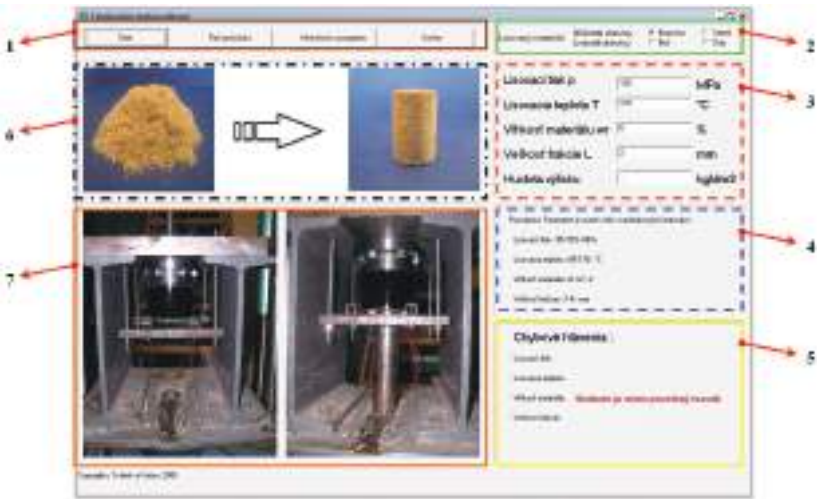


Figure 4.2: Basic overview of the designed software (Křižan, 2009).

The designed application software can operate in two basic modes. In the first mode, the values of the examined parameters (pressing pressure, pressing temperature, moisture content of the material, and the size of the raw material input fraction) are configured, and the application displays the estimation of the resulting density values after the analysis.

In the second mode, the application functions in a reversed manner. The density values of the briquette, along with three or four of the examined parameters, are set. The application will display the estimates for the remaining parameters after the analysis. The application software can be a useful tool for estimating the quality of briquettes according to the configured parameters or for optimizing structural parameters.

In the first case for using this software, the application can be a useful tool for briquette producers. It is estimated that most producers typically use pressing machines on which it is not possible to change the pressing pressure. This parameter is constant and is one of the input parameters. On this application, the producer can also enter the pressing temperature (depending on the pressing machine, control may or may not be possible), the moisture content of the input fraction, and its size. After analysis with the aid of the designed application, the user receives the estimated values for the density of the briquette before commencing the process itself. Subsequently, the user can use the application for predicting the values for examined parameters. Another possible input value is the briquette density that the user wishes to achieve. If the producer uses a pressing machine on which it is not possible to control the pressing temperature, then these parameters can be constant, fixed input as well. However, if the temperature can be regulated, the value must be set. Material parameters (material humidity and fraction size) can be easily obtained according to the technology requirements. In summary, there are 2-3 fixed inputs and 2-3 controllable inputs. The application is able to control the parameters and estimate optimal parameters for the pressing process to achieve the desired density.

The second case for using the designed application is the use of the software as a tool for optimizing technology parameters. Pressing machine constructors can use this application for predicting the pressing pressure and temperature. By setting fixed values for the briquette density and material parameters (moisture content and fraction size), the application is able to gradually regulate the pressing pressure and temperature and estimate the necessary values for these parameters. These optimal values are then the input values when designing the construction and structural analysis of the pressing chamber, pressing tool, and drive that facilitate the pressing process.

Using this application, blueprints for creating illustrative graphical dependencies of the density from the examined parameters (pressing press, material moisture content, and fraction size) can be found for several temperature levels. These dependencies are shown and described in Figures 4.3-4.5. It is necessary to understand that the listed dependencies and examples for using the application software are

valid for pressing pine sawdust only. Expanding the usability of this application for other types of materials and expanding the interval values requires conducting more experiments. The results from these experiments would alter the mathematical model, and thus the designed application using this model would need modification.

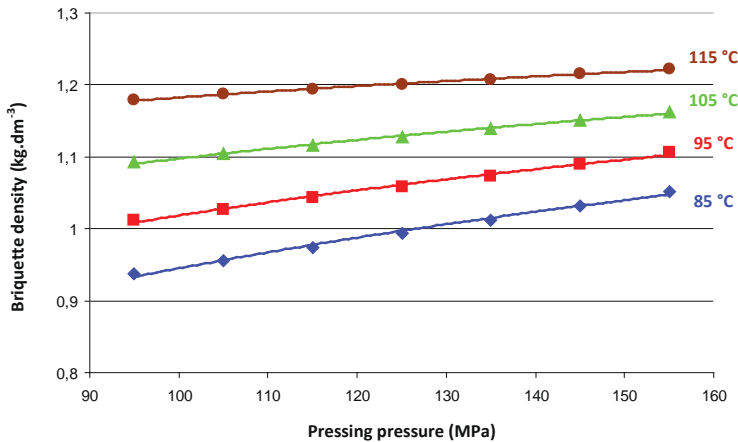


Figure 4.3: Briquette density dependence with respect to various pressing temperatures for pine sawdust ($w_r = 10\%$; $L = 2\text{mm}$) (Križan, Šooš & Vukelić, 2009; Križan & Matúš, 2008).

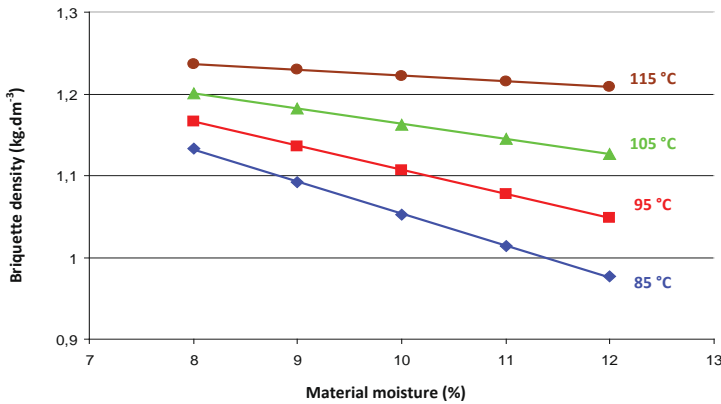


Figure 4.4: Briquette density dependence with respect to material moisture at various pressing temperatures for pine sawdust ($p = 155\text{ MPa}$; $L = 2\text{mm}$) (Križan, Šooš & Vukelić, 2009; Križan & Matúš, 2008).

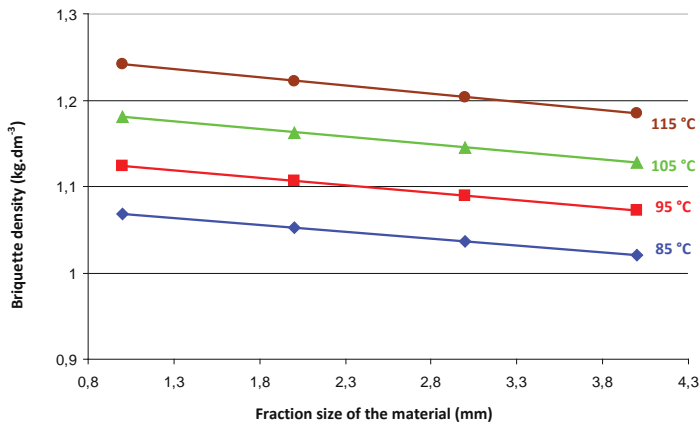


Figure 4.5: Briquette density with respect to the material fraction size at various pressing temperatures, for pine sawdust ($w_f = 10\%$; $p = 155$ MPa) (Križan, Šooš & Vukelić, 2009; Križan & Matúš, 2008).

Based on the dependencies shown, the four examined parameters have a large impact on the quality of the briquette as evaluated by the resultant briquette density during the pressing process. Fig. 4.3 shows that the briquette density increases with increasing pressing pressure and temperature. This dependency shows a change in briquette densities at pressing pressures from 95 to 159 MPa and in the temperature interval from 85 to 115 °C. The pressing temperature and pressure are parameters that mutually affect each other and thus affect the resultant density. At a given pressing temperature, it is necessary to obtain a briquette with a certain density at a lower pressure. The dependency shown in Fig. 4.3 is a depiction of this principle. Consequently, pressing machine builders must either strive for lower pressing pressures at higher pressing temperatures in their designs, or vice versa. However, the pressing temperature has a larger impact on density than the pressing pressure per the present study's findings, and so we recommend that pressing machines should be constructed with a focus on regulating temperature.

Fig. 4.4 shows the dependence between briquette density and moisture content of the raw material at various pressing temperatures. The dependency shows a change in briquette density in a range of material water content from 8 to 12 % and in an interval of pressing temperatures from 85 to 115 °C. The dependency shows how the moisture content of the material combined with pressing temperature affects the resultant briquette density. We can see that, with decreasing values of water content and increasing pressing temperature, the briquette density increases. This results in better plasticisation of the lignin within the material at higher temperatures and smoother connections between the plasticized lignin and the material particles when under pressure. The dependency seen in Fig. 4.4 is different from that in Fig. 2.40. It

is necessary to note that the experimental results are valid within an interval of 8 to 12%. More experiments are needed to increase and expand this interval.

The effect of the fraction size combined with the change in pressing temperature on the resultant briquette density is shown in Fig. 4.5. The dependency shows the change in briquette density with fraction sizes from 1 to 4 mm and an interval of pressing temperature from 85 to 115 °C. We can see that, with a decreasing size of fraction and increasing pressing temperature, the briquette density increases. Small fractions obtain a better bond amongst the material particles and the lignin when solid particles in the material contact each other. Thus, there is more contact area and number of atoms in the interphases between particles. Decreasing the size of the fraction shrinks the pores between solid particles and thus restricts the motion of the particles.

Thanks to the derived mathematical model, we are able to describe the effects of parameters and their mutual interactions in the pressing chamber. Another goal of this study was to define the optimal configuration of these parameters to obtain a high-quality briquette. To identify optimal configurations, density calculations using the derived mathematical model were performed for each parameter setting under different conditions (various parameter settings). This experimental study showed the most suitable as well as least suitable configurations within a given interval. These results created an applicable boundary interval for the use of the mathematical model for each monitored (affecting) parameter. Definitive values of the boundary settings can be observed in the following graphs. These graphs illustrate the applicable region that the mathematical model uses.

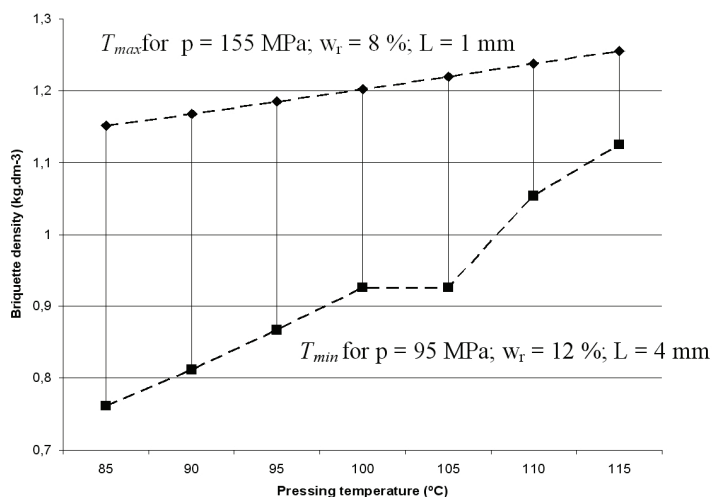


Figure 4.6: Briquette density for various settings of pressing temperature (Križan & et al., 2010).

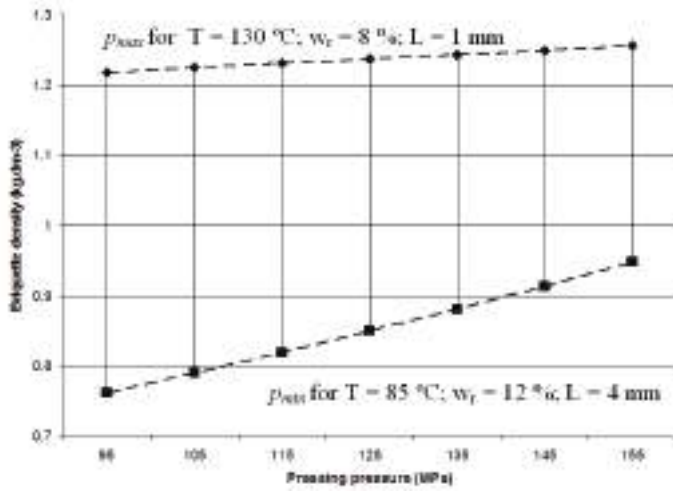


Figure 4.7: Briquette density for various pressure settings (Križan & et al., 2010).

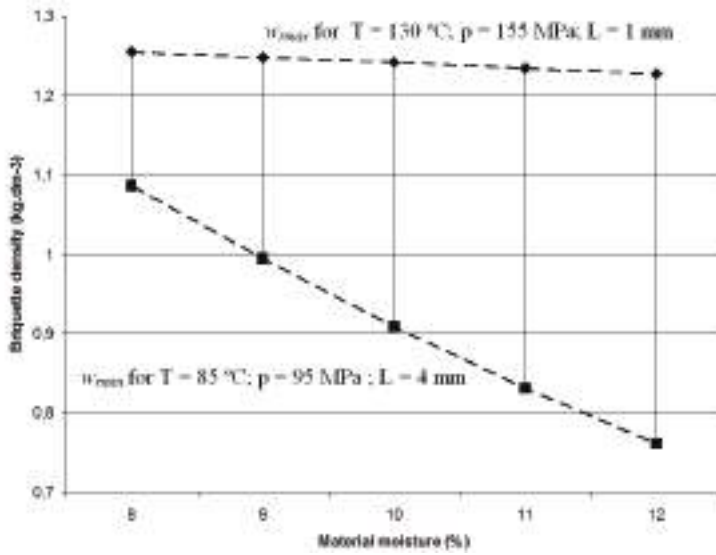


Figure 4.8: Density of briquette for various settings of input fraction moisture content (Križan & et al., 2010).

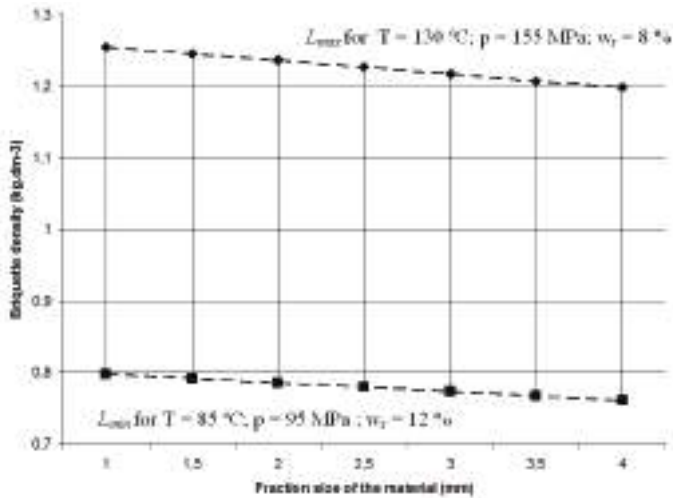


Figure 4.9: Briquette density for various settings of input fraction size (Križan & et al., 2010).

The goal of this experimental research was to provide a tool for the accurate estimate of briquette quality with respect to some set parameters through a mathematical model. When pressing any kind of material in specific conditions, it is possible to obtain an optimal setting of the effecting parameters. Since the most important factor that defines the quality of compacted wood biomass is the briquette density, this parameter was set as the criterion for the affecting parameters. Using the mathematical model derived in this study, it is possible to define regions of optimal parameters that yield a high-quality briquette according to DIN Plus. This certification scheme was chosen because it has the most strict density values (1.12 kg.dm^{-3}) for wood material briquettes. The following figure illustrates where to find a specific region. This figure was created using individual sets of monitored parameters. Each set is defined by a closed interval for which an experiment was performed. These intervals are identical to those calculated by the mathematical model.

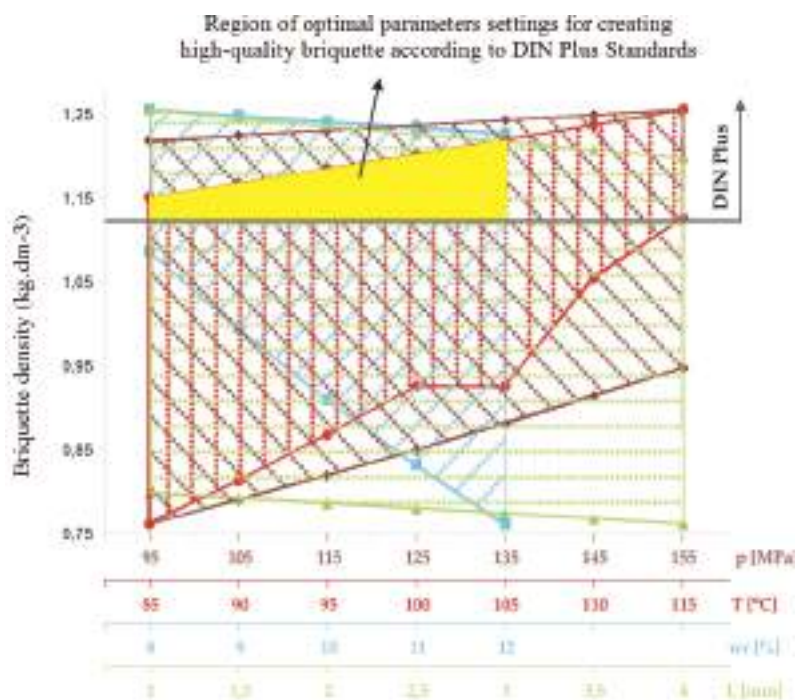


Figure 4.10: Illustrative representation showing regions of optimal parameters settings with respect to briquette quality (Križan & Svátek 2007; Svátek & Križan, 2013).

5 Experimental Research in the Densification of Wood Waste

In modern times, the used waste biomass from forests comes in different forms, like chips and shavings, sawdust, logs after logging, waste from manipulation in forest storage, tree stumps, roots, and other forms of waste. All these forms of waste are useful in generating thermal energy, i.e. the combustion in furnaces. For a more effective production of energy, the wooden waste should be processed into the more appropriate form, a compacted biofuel. With the modern boom in densification technologies, an important question to be answered is the question of material need. At the present time, we can already see that wood and wooden waste, of which there is an abundance, is lacking in practicality as biofuels. Furthermore, modern manufacturers of biofuels nowadays utilize rarely-used types of wood and often question the possibility of using other types of materials as a source of biofuel. These modifications include several factors to consider that the manufactures cannot ignore. The material properties of raw materials as well as the technological requirements of the production process itself must be considered. The input material must be disintegrated, dried, and pressed to secure optimal technological parameters that enable the production of briquettes with a desired density. If we look more closely on this process, it can be seen that each material demands its own procedure.

When pressing various types of material, different parameters (pressure, temperature, and others) are manipulated. There are operating differences between pressing soft woods and pressing hard woods. Of course, there can be differences between pressing soft woods of various types, and likewise with hard woods. Differences in chemical composition are not only present between deciduous trees and coniferous trees (Tab. 1.4), but also between each of the specific woods (Tab. 1.5). Using different types of raw material, and operating at different input conditions, the resulting briquettes possess different properties of various types (density, mechanical durability, thermal potential, water content, ash content).

To produce high-quality briquettes from each type of wood, it is necessary to understand the raw material behaviour. It is therefore very important to understand the behaviour of various materials during the process of densification and what technological parameters are to be controlled to produce a briquette with a certain standard of quality. Because each material has its own mechanical and thermal properties and a unique chemical composition, separate but similar experiments must be conducted for each of the wood types.

From the previous results and analysis, it is clear that the type of raw material used will have a significant influence on the pressing process. In the next phase of the present study, several experiments of similar character were conducted on several types of wooden materials. To maintain experimental accuracy, one material was

tested at a time. In this chapter, the results from the experimental study of several types of wooden raw materials are discussed.

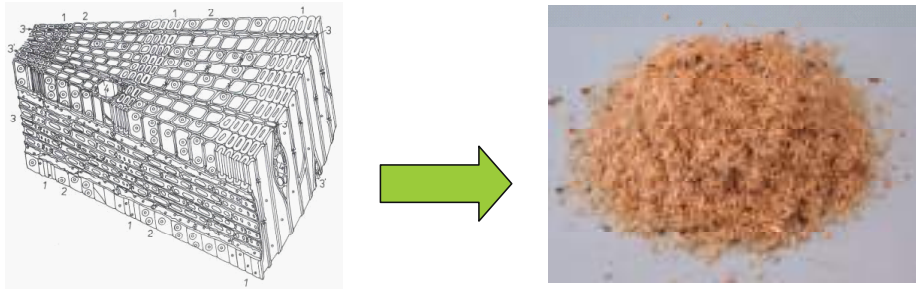


Figure 5.1: Spatial figure of softwood section (left) and spruce sawdust prepared for pressing (Požgaj & et al., 1997; Križan & et al., 2009).

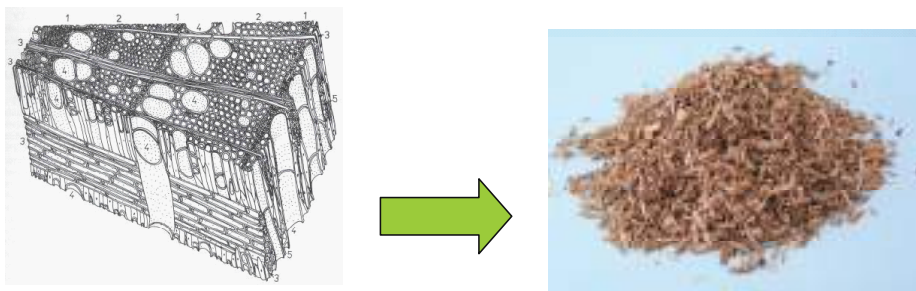


Figure 5.2: Spatial figure of hardwood (right) and beech wood sawdust prepared for pressing (Požgaj & et al., 1997; Križan & et al., 2009).

In the figures above, the difference between soft and hard wood is clearly seen. In Figure 5.1, the spruce sawdust is of 2 mm fractions. In comparison with Figure 5.2, which shows the same fraction size of beech wood sawdust, it can be seen that the structure and shape of the two types of wood particles are different. In this case, spruce sawdust has a very fine structure, resembling a cluster of fine particles, or flakes. Beech wood sawdust shows the character of hard, long flakes. These differences cause different behaviours in the raw material when compressed.

5.1 Experimental Research on Selected Types of Wood

All experiments were conducted in a similar manner on an experimental pressing stand. As previously mentioned, on this stand, it is possible to conduct experiments to determine the effect of pressing temperature, pressing pressure, material moisture, the size of input fraction, and the effects of some of the structural parameters. The experiments were conducted at ambient temperature, around 23°C. The experimental plan was identical to the plan of the pine sawdust experiment. In this phase of the study, spruce sawdust, beech wood sawdust, and oak sawdust were used. The input fractions of the mentioned materials, compared to that of the pine sawdust, are shown in the following figure.



Figure 5.3: Input fraction (sawdust) and briquettes of various materials (Križan & et al., 2009; 2011a).

The next figure shows the relationship between the effects of the pressing temperature and pressing pressure on the resultant density of the spruce briquettes. Pressing pressure is a factor that mainly affects the strength of the briquette. Briquette strength is higher when the acting pressure is higher during compression. It increases to the

ultimate tensile strength of the compressed material. The briquette strength affects the durability of the briquettes because the increased strength decreases the tendency of the final product to absorb atmospheric humidity when stored.

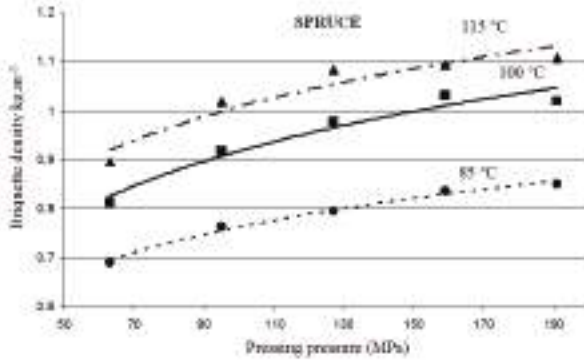


Figure 5.4: Dependence of briquette densities of spruce on the pressing pressure at different pressing temperatures. ($w_r = 10\%$; $L = 2$ mm) (Matúš & Križan, 2010; Menind & et al., 2012; Križan, 2007).

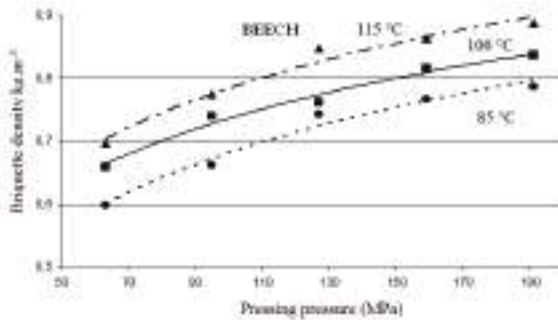


Figure 5.5: Dependence of briquette density of beech on the pressing pressure at different pressing temperatures. ($w_r = 10\%$; $L = 2$ mm) (Matúš & Križan, 2010; Menind & et al., 2012; Križan, 2007).

The depicted relationship proves that, for hardwood raw material (beech, oak), with increasing pressing temperature improves the density of the resultant briquettes. This trend is also true for increasing pressing pressure.

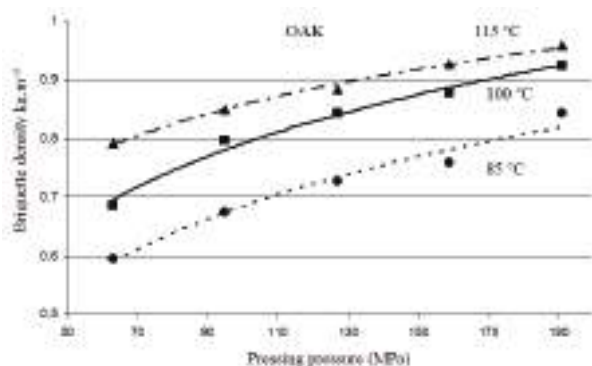


Figure 5.6: Comparison between briquette density of oak and its dependency on pressing pressure at various pressing temperatures ($w_f = 10\%$; $L = 2$ mm) (Matuš & Križan, 2010; Menind & et al., 2012; Križan, 2007).

The aforementioned dependencies prove the affect of pressing temperature on various materials. We observe that each material behaves differently; thus, it is crucial to set the conditions properly to meet the product requirements during the densification process. The higher the pressing temperature, the lower the needed pressing pressure needed to achieve a standard briquette quality. Pressure and temperature mutually influence each other during the densification process; if the pressure is increased, we can slightly decrease the temperature, and vice-versa.

We can observe that, while pressing hardwood (beech, oak), achieving a briquette density of 1 kg.dm^{-3} failed. This failure is perhaps due to the fact that these woods do not have a large lignin content as softwood does. For softwood, the lignin content is much higher, which causes better bonding between the material particles and thus better compression. It is necessary to note that, for hardwood, a negative factor is the fact that the pressed fraction is of size 2mm. By reducing the size of the fraction, it is possible to achieve the standard briquette density for all examined materials. Additionally, the mutual interaction between the pressing temperature (increase) and the moisture change of the pressed material has, according to the presented results, the most significant effect on the densification process of wooden materials. The measured results from spruce, beech, and oak sawdust, were evaluated and incorporated into a mathematical model. The designed mathematical models are valid only for the specific materials and observed parameters that can be changed in intervals in which the experiments were conducted.

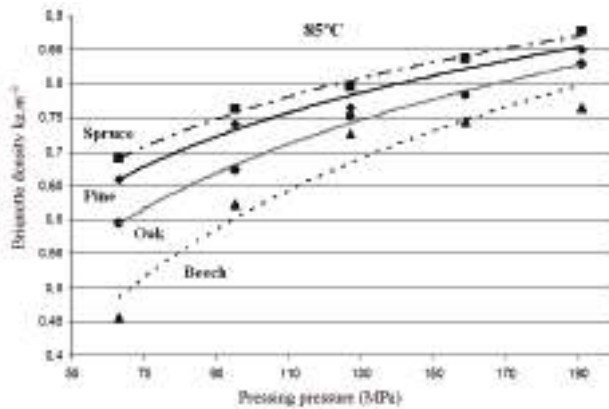


Figure 5.7: Comparison between briquette density depending on various materials to pressing pressure at a pressing temperature of 85°C ($w_f = 10\%$; $L = 2$ mm) (Matúš & Križan, 2010; Menind & et al., 2012; Križan, 2007).

In Fig.5.7, the measured dependencies of each of the woods at a pressing temperature of 85°C are shown. It is clear from the graph that the temperature is too low for the optimal plasticization of lignin and for bonding of the lignin to the material particles under the effect of an external load. The resulting briquettes, even at a pressing pressure of 191 MPa, do not meet the standard density. It can also be observed that the dependencies have an expected course with respect to the wood compression parameters; at low temperatures, the bonding and attracting forces between the particles are significant. The function of lignin is similar to that of glue, and although it is not negligible, in this case it is secondary. From the graph, the behaviour of spruce and pine during the process of compression can be observed. The dependencies suggest that a higher content of lignin affects the briquette density.

In Fig. 5.8, it can be seen that the increase of pressing temperature has an effect on the higher value of briquette density at a lower pressing pressures. For spruce and pine, we notice a significant increase in density at higher pressures. Even though the pressing temperature is not yet optimal, a higher lignin content is present in these materials. For oak and beech, the increase of density is slower due to the much lower content of lignin. Even at a temperature of 100°C combined with a higher pressing pressure of 191 MPa, optimal plasticization of lignin is not reached in hardwood.

In the next figure, the dependencies of briquette density from various materials on the pressing pressure (at a pressing temperature of 115 °C) show that, for softwood, the role of higher lignin content improves the bonding of material particles for better compression. For hardwood, in which lignin is less abundant, lower briquette density values are achieved. The figure not only depicts the difference between soft and hard wood but also between each wood type as well. A solution to increase briquette

density from the point of view of biofuel production technology is the use of additives. Technical standards permit the use of glues, but of course only the ones based on biologically friendly materials. Here, we associate the chemically unmodified products with agricultural and forest biomass (for example, whole grain flours, cornstarch, and rye flour). These can be mixed with basic materials to produce wooden pellets and simplify the pressing process while also improving the energy balance, density, and mechanical resistance of the resulting briquettes.

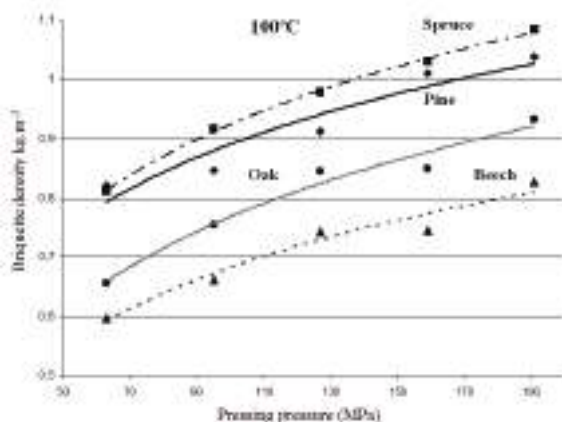


Figure 5.8: Comparison between the dependencies of briquette densities and pressing pressures at a pressing temperature of 100°C of various materials ($w_f = 10\%$; $L = 2$ mm) (Matúš & Križan, 2010; Menind & et al., 2012; Križan, 2007).

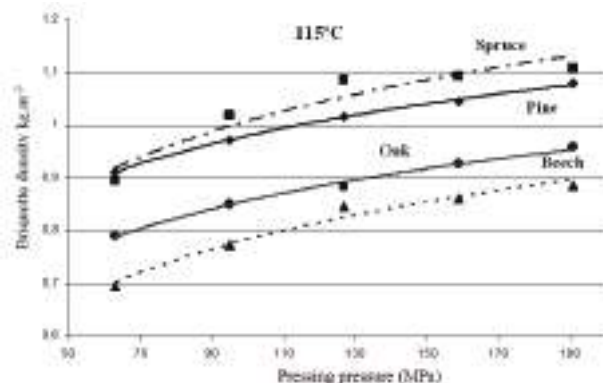


Figure 5.9: Comparison of briquette density, dependencies of various materials to the pressing pressure at a pressing temperature of 115 °C ($w_f = 10\%$; $L = 2$ mm) (Matúš & Križan, 2010; Menind & et al., 2012; Križan, 2007).

We see that the increase in pressing temperature has a positive effect on the performed experiments. The most significant effect on the process is the pressing temperature. Although the evaluations mentioned in this monograph were discussing the compression of pine sawdust only, it is highly probable that the pressing temperature will have the most significant influence on the briquette density for various wooden materials.

5.2 Experimental Research on the Effect of Parameters on Briquette Dilation

Experiments in the field of biomass densification indicate that briquette dilation is a very important factor to be considered (Križan & et al., 2014; Simanov, 1995). Briquette dilation is a phenomenon during which, under the effect of internal briquette parameters and external process parameters, the dimensions of the briquette (diameter, length, and briquette weight) change. Regarding the process and evaluation of the experiments, it is not possible to measure briquette dimensions directly after pressing. Dilation thus directly affects the given briquette parameters. The process of dilation arises under the effect of pressure and in relation to mutual interaction of pressing temperature, input fraction size, and material moisture content. For the purposes of experimentation, we distinguish two basic types of briquette dilation (Križan & et al., 2014; Križan, Šooš & Matúš, 2011):

- dilation with the effect of decreasing briquette density – as the briquette diameter increases, the length of the briquette increases and the weight of the briquette decreases (the accompanying phenomena cause the formation of cracks in the briquette, where material particles are separated or the briquette falls apart).
- dilation with the effect of increasing briquette density – as the briquette diameter decreases, the briquette length decreases and the weight increases (the accompanying phenomena is the briquette retracting and hardening).

Both these basic types of dilation are, of course, significantly affected by the types of compressed material, the moisture content of the input fraction, its size, the pressing temperature, the pressing pressure, and the length of briquette relaxation time.

Briquette stabilization period is the time interval during which dilation of the briquette occurs and during which the briquette is stabilizing (Križan & et al., 2014; Simanov, 1995). The stabilization period of the biomass briquette takes around 24 hours depending on the type of material and technology used, although it can take even longer. After pressing, the briquette is placed into stable environmental conditions. During this stabilization period, the diameter, length, and weight are measured multiple times and evaluated. If, during the course of the 24 hour period, the weight of briquette changes by a maximum of 0.1 % of its weight, the state of the briquette is considered stabilized (DIN 52182).

The stabilization period (briquette dilation) is significant mainly regarding the change in briquette density. Briquette density is one of the basic mechanical indicators of briquette quality, which is why the final product densities are determined after each experiment. The density is important regarding the sufficient coherence of material particles in the briquette. Therefore, it is important to consider the dilation during experiments and obtain data over a period from the fresh briquette to the stabilized briquette. During biomass densification, lignin is released, which acts as a glue. At the same time, it creates a layer or thin film on the surface of the product that prevents moisture from being absorbed from the surroundings. The effect of dilation is well known for briquette producers. During real operations, non-acceptable effects of briquette dilation may occur and lead to crumbling. In technological production lines, the standard stabilization duration is shortened by cooling the briquettes. During laboratory experiments, dilation can significantly distort the results.

5.2.1 The Effect of Pressing Pressure on Briquette Dilation

All relations presented in this monograph from the included experimental results are shown after the stabilization period. Results can be evaluated and processed only after briquette stabilization. On the following figure, the differences between pinewood briquettes before and after stabilization are shown. The difference is significant. To obtain a correct result, it is necessary to work with results obtained only after stabilization.

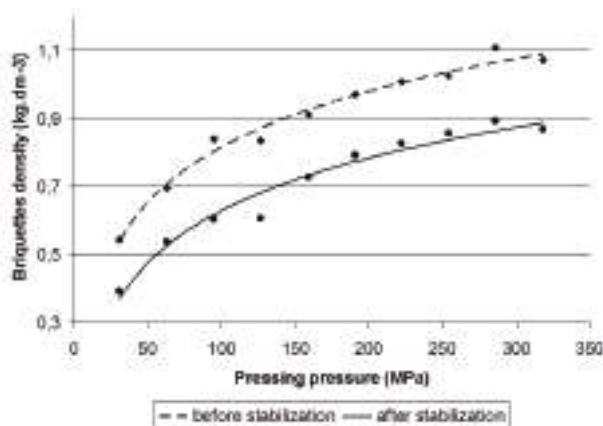


Figure 5.10: The briquette density of Pine sawdust, dependence of the pressing pressure ($w_r = 10\%$; $L = 2$ mm) (Križan & et al., 2014).

An experimental objective was to quantify and define the effect of basic technological parameters on briquette dilation. The experimental research was conducted on

a designed experimental pressing stand according to the procedure described and mentioned in the previous chapter. The briquette density was chosen as an indicator of quality. For each point shown in the graphical dependencies, ten briquettes were created. This number of briquettes was chosen to obtain more accurate data and decrease the result error. The experiments were performed with pine sawdust with a fraction size 2 mm and moisture content of 10%. These material parameters were sustained throughout all of the experiments. On the basis of analysis conducted in our workplace and on the basis of information gained from several and various international conferences and symposiums, we defined the effect of pressing pressure and the effect of pressing temperature on briquette dilation.

In the first step, the effect of pressing pressure was defined. In the following table, the average values of briquette density compressed at various pressures are listed. Calculated briquette densities and the differences before and after stabilization are also shown. The biggest difference in briquette density was noted at lower pressing pressures. With the increase of pressing pressure, these differences slightly diminished. The trend shows that, the higher the pressing pressure is during the process, the lower the briquette dilation. In Fig. 5.11, the difference in briquette density before and after stabilization for each briquette is shown at various pressing pressure settings.

In Tab. 5.1, the average density values are listed. These average values were calculated from a set of 10 densities. These are shown separately in Fig 5.11.

Table 5.1: Briquette density from Pine sawdust in relation to pressing pressure ($w_r = 10\%$; $L = 2 \text{ mm}$) (Križan & et al., 2014).

Briquette no.	Pressure (MPa)	$\rho \text{ (kg/dm}^3\text{)}$		Density difference
		Before stabilization	After stabilization	(%)
1.	31	0.664	0.350	47%
2.	63	0.699	0.535	23%
3.	95	0.841	0.605	28%
4.	127	0.836	0.625	25%
5.	159	0.913	0.729	20%
6.	191	0.967	0.792	18%
7.	222	1.066	0.813	24%
8.	254	1.026	0.856	17%
9.	286	1.113	0.893	20%
10.	318	1.074	0.869	19%
				$\Delta \bar{\rho} = 24\%$

The relationships in the previous figures prove the tendency of briquette dilation to decrease under increasing pressing pressure. It was also demonstrated that selecting a high number of briquettes for one setting decreases the resulting error. It can be seen that some of the measured values are significantly different than that of the averaged one.

5.2.2 The Effect of Pressing Temperature on Briquette Dilation

A similar experiment was performed to determine the effect of the pressing temperature on briquette dilation. The configuration and conditions of the experiments were left the same as in the previous case, but we observed changes not only at a temperature of 25 °C (laboratory conditions), but also at temperatures of 80 °C and 115 °C.

In the previous figure, the effect of pressing temperature on briquette density can be observed. With increasing pressing pressure and pressing temperature, the briquette density increases. It is important to note that the values presented on this figure are measured after stabilization. The effect of pressing temperature and pressing pressure on briquette density was defined and quantified just like in previous chapters. And, as it was mentioned before in this monograph, pressing temperature during densification of wooden materials goes hand in hand with the pressing pressure. In the next figure, the dependency between the pressing temperature and pressing pressure regarding the briquette density are shown.

Temperature significantly affects the bonding mechanism acting between the solid particles of the pressed material. A more thorough bonding of cellular structure of pressed material and the required plasticization of lignin is possible only under the active pressing temperature. In the case of pressing at an improper temperature, Van der Waals attraction forces form between the solid material particles. On the basis of decreasing the gaps between the solid particles of the material, and because the moisture within the pressed material is contained mainly on the product surface, fluid bridges start forming. Particles get closer to each other and are pressed as a result of the acting pressing pressure, and on the basis of their shape, they connect and bind together to create deformed bonds. Wood is an elastic material; to overcome its elastic limit, optimal conditions for bonds to form between particles must be achieved. At pressing conditions, where the pressing temperature was maintained at 25 °C, these bonds were not created; after releasing pressure, the briquette changed its dimensions and “inflated.” This phenomenon causes a lower briquette density after stabilization than before stabilization (Fig. 5.10). The average density change of pinewood briquettes at a pressing temperature of 25 °C was 24 % (Tab. 5.1).

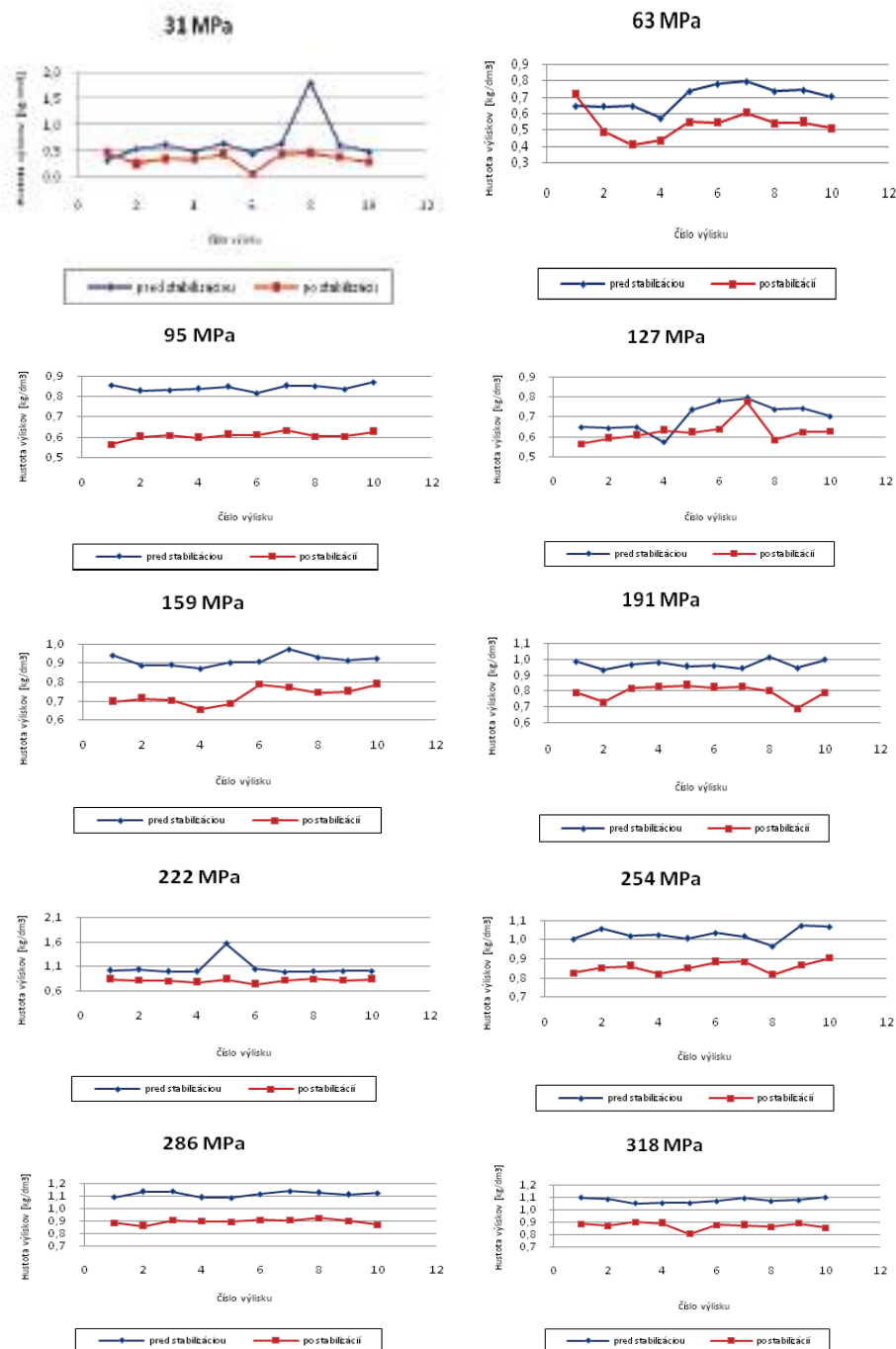


Figure 5.11: Dilation process, briquette density values at various pressures (relation description: axis x → briquette number; axis y → briquette density (kg/dm³); blue color → before stabilization ; red color → after stabilization); (Križan & et al., 2014; Križan, Šooš & Matúš, 2011).

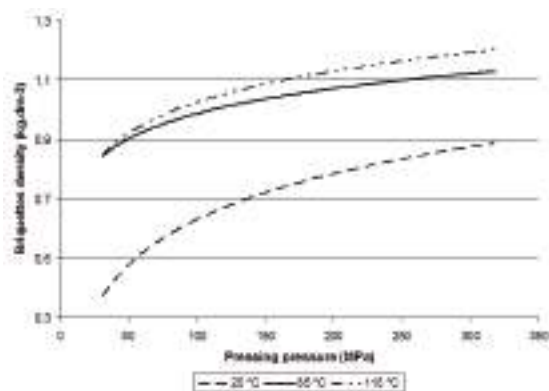


Figure 5.12: Density of Pinewood briquettes, dependence of the pressing temperature at various pressing temperatures. ($w_f = 10\%$; $L = 2$ mm) (Križan & et al., 2014; Križan, Šooš & Matúš, 2011).

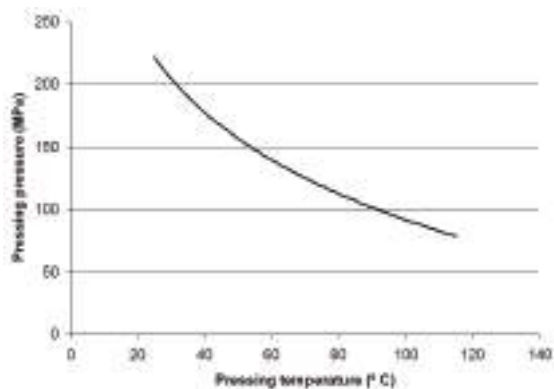


Figure 5.13: Pressing pressure, dependence of the pressing temperature when pressing Pine sawdust. (Križan & et al., 2014; Križan, Šooš & Matúš, 2011).

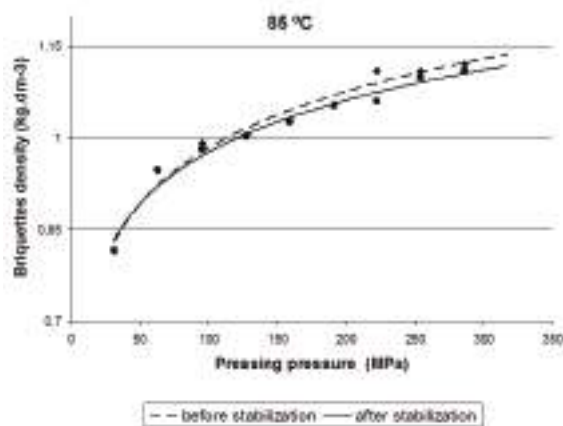


Figure 5.14: Briquette density of Pine sawdust, dependence of the pressing pressure at 85 °C ($w_f = 10\%$; $L = 2$ mm).

In the previous figure, the dependence of briquette density on the pressing pressure at a temperature of 85 °C is shown. The differences between the curves, which compare the states before and after stabilization, decrease. The briquette density before stabilization decreased by 1.5 % compared to the state before stabilization. This is a significant difference compared to briquettes pressed at 25 °C. This can be attributed to the effect of higher pressing temperature. In this case, better bonding between material particles and a certain phase of lignin plasticization has been reached. However, the briquette still lacks sufficient internal strength, which prevents its disintegration and thus decreases the briquette density.

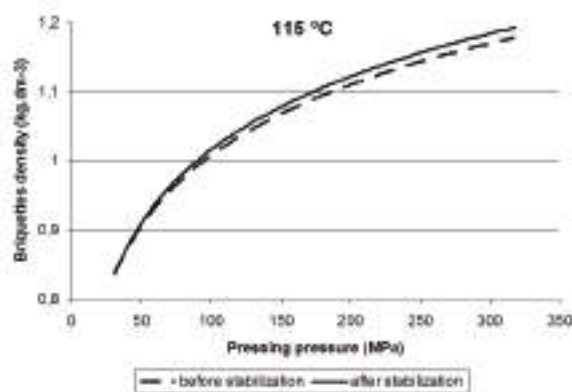


Figure 5.15: Briquette density of Pine sawdust, dependence of the pressing pressure at 115 °C ($w_t = 10\%$; $L = 2$ mm).

The dependence of the briquette density on the pressing pressure at a pressing temperature of 115 °C is shown in the previous figure. The difference between the curves before and after stabilization is once less than that of the previous cases. An interesting phenomenon is that, after stabilization, a higher value of density than that before stabilization is reached. For briquettes compressed at this temperature, there is no expansion or disturbance to the briquette's structure occurring during dilation; on the contrary, the briquettes are compacted, and after stabilization, a higher density is reached.

The previous results indicate the effect of pressing temperature on briquette dilation. Thus, with increasing pressing temperature, the briquette density decreases. Interesting is the comparison at which pressure the density of 1.12 kg·dm⁻³ (stated by the standard DIN Plus) was reached. At a pressing temperature of 25 °C (Fig. 5.10), this value was not reached; at a pressing temperature of 85 °C (Fig. 5.14), the desired density was reached at a pressure of 220 MPa. Finally, at a pressing temperature of 115 °C (Fig. 5.15), the given density was reached at a pressure of 190 MPa. Thus, if we press at a higher temperature, we can use less pressure to achieve the required briquette

density. A higher pressing pressure is more appropriate, since better molecular bonding is achieved in the material because of the lignin plasticization.

Of course, the listed pressure values are very high. It is necessary to press briquettes at a lower pressing pressure. It is therefore important to define and quantify the optimal conditions for several wooden materials and thus decrease the requirements for constructing the pressing equipment. One method might be by increasing the pressing temperature to a maximum value of around 130 °C. Increasing the temperature higher than that results in the loss of volatile substances and lignin.

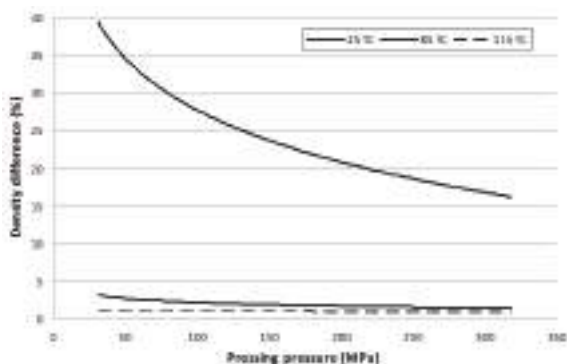


Figure 5.16: Density differences, dependence of the pressing pressure at observed temperatures. (Pine sawdust; $w_f = 10\%$; $L = 2$ mm) (Križan & et al., 2014; Križan & et al., 2011b).

In the previous figure, the dependence can be seen between different densities of pine sawdust briquettes on the pressing pressure at three observed temperatures (25 °C, 85 °C, and 115 °C). These dependencies clearly show the effect of increasing pressing pressure together with temperature on briquette dilation. We can see that increasing the pressing pressure without the effect of temperature positively affects the briquette dilation.

5.2.3 The Effect of Fraction Moisture on Briquette Dilation

In the next phase of the experimental research, the objective was to define the effect of fraction moisture on briquette dilation. In this case as well, pine sawdust of size 2 mm was used. The experiments were conducted in a similar manner as before. Since the objective was to determine the effect of moisture content, 8 levels of configuration for monitored parameters were equally distributed over a range of 5% to 30 %. For each level, 10 briquettes were pressed. The pressing pressure was constant and set at 286 MPa. Since we were pressing without increasing temperature, this pressing

pressure was selected to achieve briquette compactness. In the following figure, we can see the dependency of briquette density on moisture fraction. The two curves represent the states before and after stabilization. The briquette density difference is significant before and after stabilization, and the average difference in pine sawdust briquettes is around 9 %.

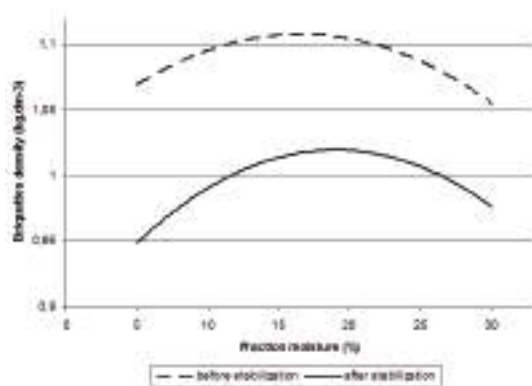


Figure 5.17: Briquette density, dependence of Pine sawdust on the moisture content in the fraction ($p = 286 \text{ MPa}$; $L = 2 \text{ mm}$) (Križan & et al., 2014; Križan & Matúš, 2008).

In the following figure, the dependence of briquette density on the moisture content of input fractions is shown. We can observe that, with increasing moisture, the density has a tendency to decrease. It is necessary to note that these results were obtained while pressing without any input temperature. It was thus interesting to compare the results of density change after using higher temperatures. As was previously quantified in the experimental research (Fig. 3.14), the interaction between pressing temperature and moisture fraction has a significant effect on the resultant briquette density.

Other than that, increasing the pressing temperature positively increases the briquette density and also tends to decrease the negative effect of moisture fraction on the briquette density. This statement also proves the listed results in Fig. 4.4. Under a temperature of 85°C , the increase in briquette density when changing the moisture content from 12% to 8% results in an almost tenfold difference, as when operating at a temperature of 115°C . Based on this, we can assume that the listed interaction (pressing temperature \leftrightarrow fraction moisture) will have an effect on briquette dilation. Thus, in the future it is necessary to perform experimental research with the objective of defining the effect of the interaction (pressing temperature \leftrightarrow fraction moisture) on briquette dilation. Also, it will be important to perform research on various types of wooden materials, since the effect of the given interaction is related to the lignin and cellulose content in the materials.

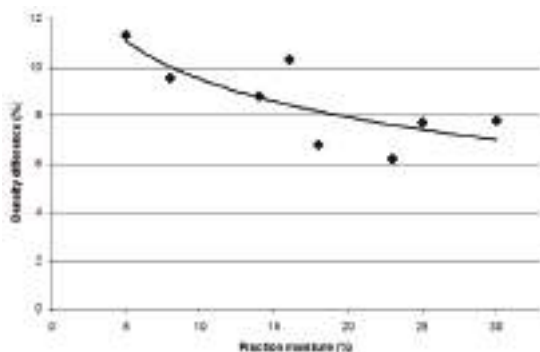


Figure 5.18: The difference of briquette density on moisture content of input fraction ($p = 286$ MPa; $L = 2$ mm) (Križan & et al., 2014; Križan & et al., 2011b).

5.2.4 The Effect of Fraction Size on Briquette Dilation

A significant part of the experimental research was also studying the effect of fraction size on briquette dilation. The experimental results in Fig. 4.5 have shown that decreasing the fraction size can positively influence the increase in briquette density. Therefore, it was important to also define the effect of fraction size change on briquette dilation. The experimental conditions were similar to those of the previous experiment when observing the effect of fraction moisture on briquette dilation. In the following figure, the dependence of briquette density on fraction size is shown. Again, the two curves characterize the state before and after stabilization. The briquette density difference before and after stabilization is very significant; the average change in density of pinewood briquettes is around 18 %.

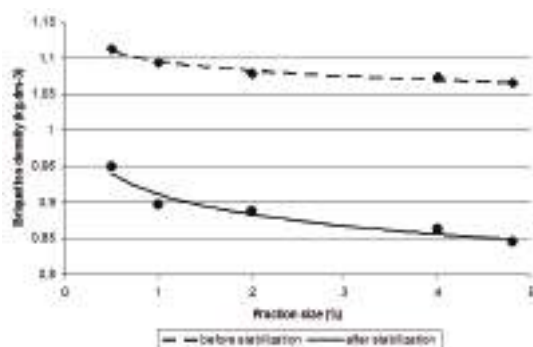


Figure 5.19: Dependence of Briquette density (Pine sawdust) on fraction size ($p = 286$ MPa; $L = 2$ mm) (Križan & et al., 2014; Križan & et al., 2011b).

In the figure, it can be observed that, with increasing fraction size, material density changes. Also, it is important to note that, for these experiments, the measurement was performed while pressing without any increase in temperature during the process and at a constant pressing pressure. It is interesting to compare the results of briquette density change obtained by pressing at higher temperatures and with various levels of pressure.

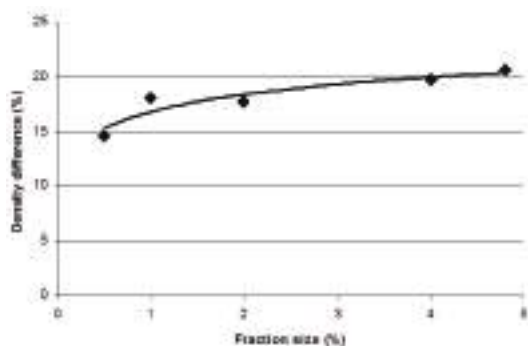


Figure 5.20: The difference in briquette density depending on fraction size ($p = 286 \text{ MPa}$; $L = 2 \text{ mm}$) (Križan & et al., 2014; Križan & et al., 2011b).

The sawdust compressibility factor size is also an important parameter. The compressibility factor characterizes one of the basic properties of disintegrated sawdust – compression. The sawdust compression is defined as the tendency of bulk sawdust to change volume under the effect of an external force. This means that the magnitude of this compressibility or compression factor is affected by the acting pressure. The results of the experiments show that increasing the pressing pressure for sawdust of equal fraction size increases the briquette density. Also, it is shown that, by increasing the fraction size, the briquette density decreases (Fig. 5.21). The listed results imply that important parameters affecting the compression factor are the pressure and the fraction size. Also, experimental research determined that, by increasing the size of the fraction under the same pressure, the compression factor decreases. Thus, it is important to conduct experimental research with the objective of defining the affect of the interaction (pressing pressure \leftrightarrow fraction size) on briquette dilation in the near future (Matúš & et al., 2014). Also, it will be very important to realize this experiment on various wooden materials, in addition to on various moisture levels. The relations shown in the following figure were created using the designed application software, which operate on the basis of the designed mathematical model.

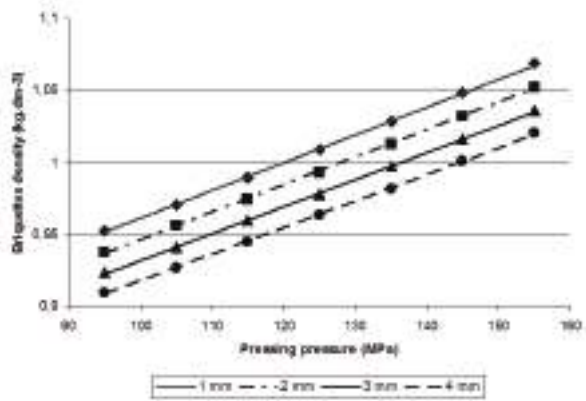


Figure 5.21: Dependence of briquette density on pressure at various fraction sizes ($T = 85\text{ }^{\circ}\text{C}$; $L = 2\text{ mm}$).

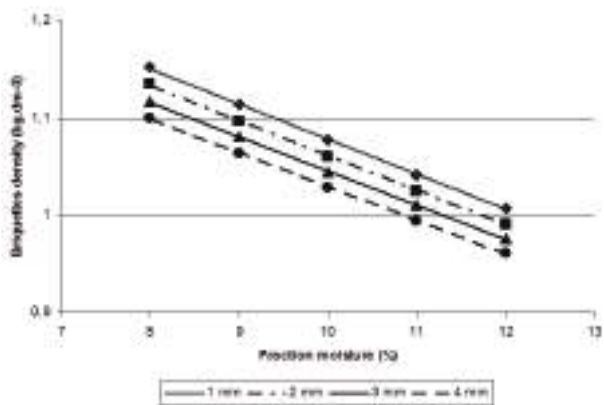


Figure 5.22: Dependence of briquette density on moisture content at various fraction sizes ($T = 85\text{ }^{\circ}\text{C}$; $p = 159\text{ MPa}$) (Šooš, Matúš & Križan, 2010; Križan, 2009).

6 Describing the Significance of the Results to Optimize the Structure of the Pressing Chamber

In this section, the significance of the conducted experiments and results are discussed as well as their effects on optimizing the construction of the pressing chamber and pressing structure itself. During the experiments and during the evaluation of the experiments, the results are significant regarding the construction of the pressing machines and pressing chambers. This section will briefly describe the significance of the results with respect to the construction of pressing chambers and machines.

6.1 Effect of Pressing Temperature

The pressing temperature has the largest impact on the whole pressing process. The pressing temperature affects the plasticization of lignin during the pressing process. Lignin, when in liquid state, can bind better to the pressed material. The effect of the pressing pressure, which is followed by cooling, creates a solid briquette. Based on the experimental results, it can be argued that the optimal pressing temperature, from a quality point of view, is more important than the pressing pressure. From practical experiences, it is also recognized that obtaining the optimal temperature in a pressing machine is cheaper than reaching an optimal value of pressure. It is then logical, when constructing pressing machines, to use a device with ability to regulate temperature. Another effect of the pressing temperature on the process is the experimental observation that, during the pressing process and with the use of the optimal pressing temperature, less force is required for removing the briquette from the pressing chamber when compared to that from pressing with no applied heat in the process. When pressing under higher temperatures, plasticization of the lignin occurs, followed by its subsequent mixing within the material particles, which creates a smooth shiny film on the surface of the briquette. This film causes a smaller friction coefficient between the briquette and chamber wall.

6.2 Functional Dependence $\rho = f(p, T)$

Finding this functional dependence is the “alpha and omega” of the whole pressing process for the optimal construction of a pressing machine. Not one manufacturer of these machines can say with certainty what the appropriate pressing pressures for making a quality briquette according to standards are. Too often these pressing machines are oversized and thus economically demanding. Finding the functional dependence can help when constructing a pressing machine, sizing the drive, designing the mechanism, and sizing of the shape and dimensions of the pressing



chamber, etc. Of course, it is necessary to conduct many more experiments because the results of our experiments are valid only for pine and only for certain intervals of pressing temperature, pressing pressure, material water content, and fraction size. To obtain better knowledge about the behaviour of the pressed material within the densification process, it is necessary to repeatedly perform experiments and supplement the results with respect to other materials. It is important to know that each material type is necessary to experiment with separately. In practice, the demands for optimal parameters are ever-increasing for the densification of varying material mixtures (for example: 30% pine, 30% spruce, 30% fir); therefore, experiments utilizing such mixtures are necessary to perform in order to obtain the optimal parameters for their use.

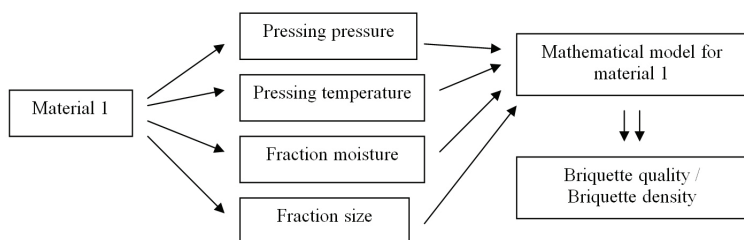


Figure 6.1: Methodology for creating the mathematical model in the first stage of the experiment (Križan, Matúš & Svátek, 2012; Svátek & Križan, 2013).

In Fig. 4.3, the briquette density change due to changes in temperature and pressure is shown. If we look closely at the change of briquette density, pressed at 95 MPa, then only under the effect of the change of temperature from 85 °C to 115 °C the density changes by around 25.8 % from 0.9378 kg.dm⁻³ to 1.1797 kg.dm⁻³. If we press a material of the same characteristics with a pressure of 159 MPa, then, under the effect of temperature from 85 °C to 115 °C, the density changes by around 15.6 % from 1.0604 kg.dm⁻³ to 1.2255 kg.dm⁻³. This means that, although we pressed with a 67.4 % higher pressure than in the first case, we achieved very small gains in density at the mentioned temperature values. At a temperature of 85 °C, the change in briquette density is around 13 %, while at a temperature of 115 °C, the change in density is no more than 4%. This proves the assumption that pressing at lower pressure and higher temperature is more effective. The following figure shows the effects of interacting pressing pressure and temperature on the density of the pine briquette at various moisture levels.

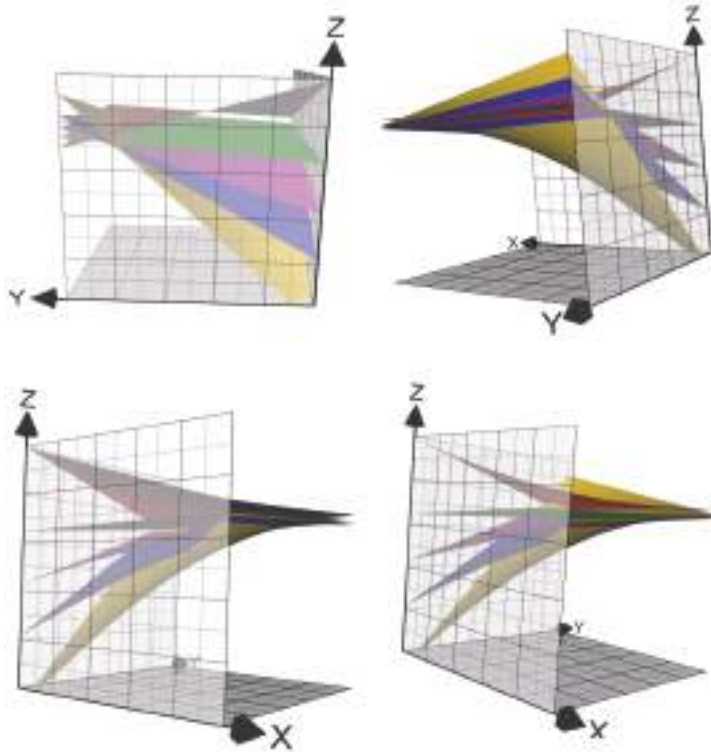


Figure 6.2: Surface response of the density of pine briquettes at a constant fraction size 2 mm / Labeling: x-axis → pressing pressure (MPa); y-axis → pressing temperature (°C); z-axis → briquette density ($\text{kg} \cdot \text{dm}^{-3}$); colour legend → levels of material moisture: red = 5%; green = 8%; purple = 10%; blue = 12%; yellow = 15%.

6.3 Back Pressure Acting on the Pressed Plug in the Pressing Chamber

The back pressure acting on the plug in the pressing chamber is a very important parameter during the pressing process. It prevents material from escaping the pressing chamber before pressing and facilitates pressing in the chamber. The back pressure can be attained using horizontal and vertical pressing configurations and forms as a single back pressure plug at the end of the pressing chamber with a controllable choke, friction coefficient between the briquette and the wall of the pressing chamber, shape of the pressing chamber (conicity), and the length of the crimp column. The value of this parameter is very useful to know while sizing a pressing chamber. According to the functional dependency $\rho = f(p)$, it is possible to find this value. Based on Equation 2.32, it is possible to calculate the value of the back pressure exerted on the plug in the

pressing chamber. The value of the back pressure, together with the value of the radial pressure, acting over the length of the pressure chamber can be determined. The next example demonstrates the usefulness of the dependency $\rho = f(p)$ and Equation 2.32 for calculating the back pressure acting on the plug in the pressing chamber. The calculation is applied to the design of the experimental stand. The back pressure is calculated according to the following equation:

$$p_G = p_k \cdot e^{\frac{4 \cdot \lambda \cdot \mu \cdot H}{D_k}}$$

where :

p_G – is the back pressure in the chamber (MPa)

p_k – axial pressure of cylinder (MPa) – in our case pressing pressure used to make a briquette of standardized quality $p_k = 120$ MPa (see Fig. 4.3).

λ – primary stress ratio σ_r / σ_m (see Fig. 2.46). For disperse materials it is: $0 < \lambda < 1$.

μ – friction coefficient between the pressed material (wood) and the wall of the chamber (steel). The coefficient is $\mu = 0.35$.

H – length of the plug (mm)

D_k – diameter of the pressing chamber (mm). In our case $D_k = 20$ mm.

For calculating the length of the plug, we will start from the dimensions of the pressing chamber and from the compression ratio. An example of the calculation is shown in Fig. 6.3.

The dimensions of the pressing chamber are $D_k = 20$ mm and $L_{kl} = 140$ mm. The volume of the pressing chamber before the first compression is $V_{kl} = 43982$ mm³. As mentioned before, calculating the length of the plug uses the compression ratio for wood. This ratio compares the volume before and after compression. Using the application software and Fig. 4.3, we know that the density of a briquette pressed at a pressure of 120 MPa and a temperature of 105 °C was 1.12152 kg. dm⁻³. The bulk density of the sawdust is around 0.140 kg.dm⁻³. This means that the compression ratio in our case is $1.12152/0.140 \cong 8$. Then, the volume of the plug is $V_{kl} / 8 = 5497$ mm³. Since the diameter of the plug does not change, through the use of a basic formula for calculating the volume of a cylinder, the length of the plug can be calculated as $H_1 = 17.5$ mm. For calculating the second plug, we need to correct for the height of the pressing chamber. Since, in our case, we are not dealing with an interim process of pressing, the chamber is always closed. The first plug is still in the pressing chamber, and its dimension decreases the volume of the pressing chamber. By subtracting H_1 from the initial length of the chamber L_{kl} , we obtain $L_{k2} = 122.5$ mm. The procedure is repeated for calculating the rest of H_i . Table 6.1 lists all calculated values for back pressure. Subsequently, we can then calculate the value of the back pressure in relation to the pressed column.

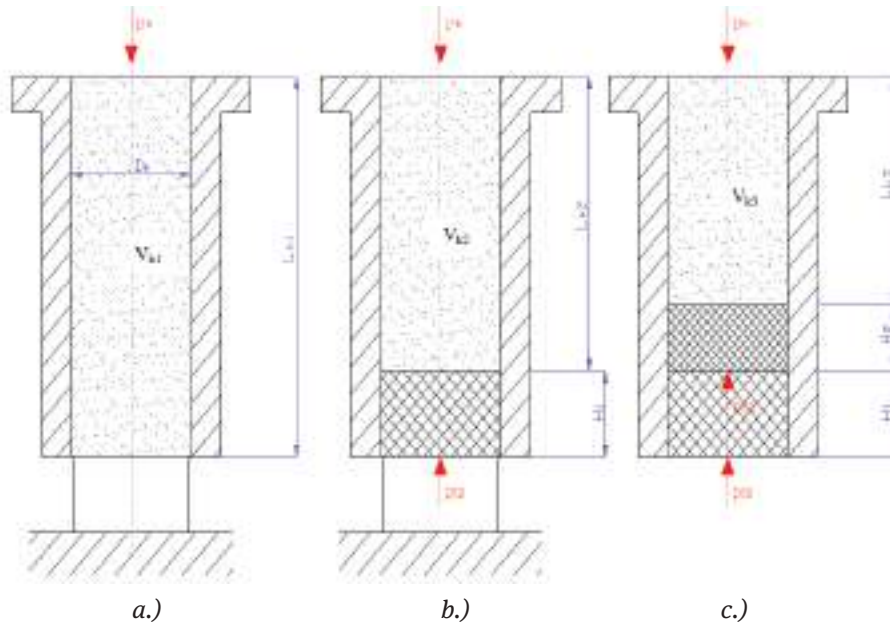


Figure 6.3: An example for calculating the length of the pressed plug (at each pressing stage) – a.) filling of the chamber; b.) pressing the first plug + filling the chamber; c.) pressing the second plug + filling the chamber.

Table 6.1: Table of calculated parameters for the back pressure.

i	1	2	3	4	5	6	7	8
L_{ki} (mm)	140	122.5	107.19	93.79	82.07	71.81	62.83	54.98
H_i (mm)	17.5	15.31	13.40	11.72	10.26	8.98	7.85	6.87
p_{Gi} (MPa)	39.84	45.74	51.59	57.35	62.87	68.15	73.18	77.84

As we can see from the previous table, the back pressure value increases with decreased plug length. In a closed pressing system, the pressed plug is shorter after each pressing because after each pressing, the volume of the pressing chamber decreases.

Based on this calculation, we can assume that the back pressure value will decrease with increasing length of the pressed column. Since Equation 2.32 allows for the calculation of back pressure depending on the length of the pressed column, it is possible to apply these results for the continuous pressing process. The explanation can be visualized by Figure 6.4. The principle of continuous pressing is different, meaning that the length of the pressed plug will be the same after each pressing,

whereas earlier plugs are shifted toward the exit of the chamber. The calculations consider the parameters of the experimental stand but with the assumption that the pressing process is continuous. Figure 6.4 describes situations and effects of the backpressure during continuous pressing.

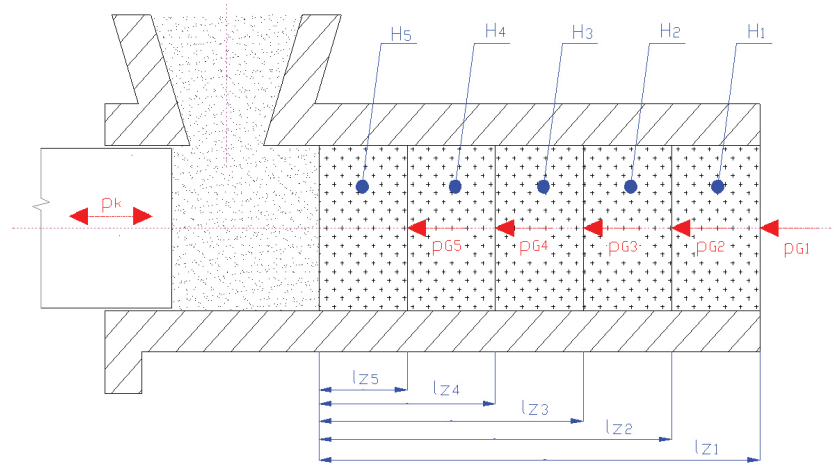


Figure 6.4: Back pressure in the continuous pressing process.

Table 6.2: Table of calculated values for the back pressure in continuous pressing process.

i	1	2	3	4	5
l_{zi} (mm)	87.5	70	52.5	35	17.5
p_{Gi} (MPa)	0.48	1.45	4.39	13.23	39.84

Based on the calculated values, we observe that the back pressure decreases as the pressed column length increases (increased length of the pressing chamber). Obviously, further experiments are necessary to verify this observation. Furthermore, it is necessary to find the optimal length of the pressing chamber depending on the pressing pressure when the desired density is achieved. This will depend also on the radial pressure. The radial pressure is possible to calculate based on the ratio between primary stresses λ (radial/axial). It would be very useful if experiments determining the radial pressure were performed.

Figure 6.5 shows the dependence of the back pressure acting on the plug within the pressing chamber on the length of the pressed column. This dependence was defined from the calculated results by means of Equation 2.32 and their interpretation

using figure 6.4. To define the dependence between the pressure on the overall length of the pressing chamber, it is necessary to understand, according to Equation 2.37, the values of residual radial pressure $p_{r,r}$ and material constant A , which gives the degree of elastic properties within the pressed material. However, these values are found only through experiments.

As above, the calculation of the backpressure uses Figure 4.3 to define the pressure at 120 MPa and at a temperature of 105 °C. In my case, 35mm briquettes were pressed which, based on the calculated parameters, should return a backpressure of approximately 13.23 MPa. This would mean that, at the values of backpressure, it should be possible to press a briquette with a density reflecting standards.

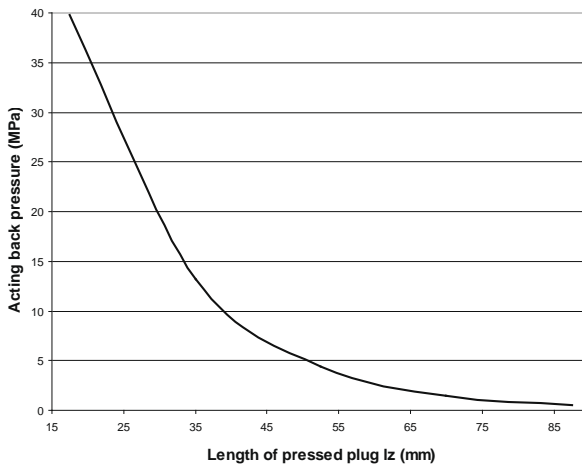


Figure 6.5: Dependence of the back pressure acting on the plug in the pressing chamber on the length of the pressed column.

6.4 Radial Pressure Acting in the Pressing Chamber

The radial pressure, pressing pressure, temperature, and back pressure work together in the densification process. The magnitude of acting pressure p_r is a factor that determines whether the briquette may exit the chamber or not. If the radial pressure acting on the surface of the plug after compressing is large, the briquette could crumble after exiting the chamber due to residual radial stresses. It is therefore necessary to keep the briquette within the chamber until the residual radial stresses decrease below the strength value of the briquette. For designers, it is very interesting to know the dependence of radial pressure on the overall length of the pressing chamber; on the other hand, such information is very difficult to obtain. It is possible to calculate them; however, without the previous calculation for back pressure, it would not be possible.

When pressing bulk materials, we are usually talking about anisotropic pressure (higher pressures are in the perpendicular direction). Equation 2.30 represents the ratio of primary stresses (radial σ_r / axial σ_m). From before, we know how to calculate p_G separately for each pressed plug. If, according to Figure 2.46 we define $p_G = p_m$, we should be able to determine the value of radial pressure using the following relations for any point in the pressing chamber.

$$\lambda = \frac{\sigma_r}{\sigma_m} = \frac{p_r}{p_m} = \frac{p_r}{p_G} \Rightarrow p_r = \lambda p_G \quad (6.1)$$

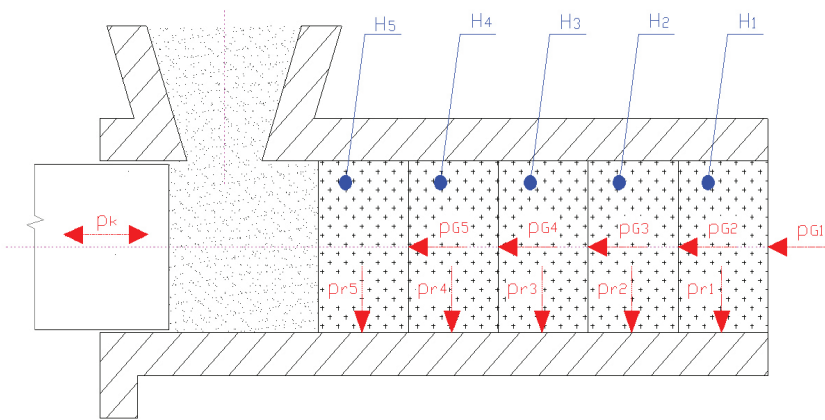


Figure 6.6: Behaviour of radial pressure during continuous pressing.

Since $0 < \lambda < 1$, we set $\lambda = 0.9$. Then, from the values of p_{Gi} in Tab. 6.2, we calculate the values for p_{ri} :

Table 6.3: Table of the calculated radial pressures.

i	1	2	3	4	5
$p_{mi} = p_{Gi}$ (MPa)	0.48	1.45	4.39	13.23	39.84
p_{ri} (MPa)	0.43	1.31	3.95	11.91	35.86

Based on the calculated values, we can see that the radial pressures decrease with increasing length of the pressing chamber.

6.5 Length of the Pressing Opening – Pressing Chamber

If we know the value of axial pressure acting on the plug for which the briquette will not crumble when it leaves the chamber, based on Equation 2.45, we can calculate the optimal length of the pressing opening (area in the pressing chamber where the given pressing pressure is acting on the plug). After modification of Equation 2.45, we get:

$$L_k = \frac{\ln \frac{p_m}{p_{m0}} \cdot r}{2 \cdot \mu \cdot G \cdot \nu_{RL}} \quad (6.2)$$

where:

p_m – pressing pressure on the plug (MPa);

p_{m0} – initial pressing pressure (MPa); in our case, the pressing pressure which gives the briquette a quality defined by standards $p_k = 120$ MPa (see Fig. 4.3).

r – radius of the pressing chamber (mm); in our case $r = 10$ mm.

μ – friction coefficient between the pressed material (wood) and the material of the chamber (steel); coefficient of friction between wood/steel is $\mu = 0.35$.

G – ratio of the elasticity module for lateral and radial directions in the fibre bundles (E_L/E_R); for pine sawdust $E_R = 320$ MPa; $E_L = 6634$ MPa.

ν_{RL} – Poisson's constant; for pine sawdust $\nu_{RL} = 0.016$.

Based on the calculated results in Table 6.2 and the conclusions described by the assumption that when applying a pressure $p_m = 13.23$ MPa on the plug, the briquette should not crumble upon exiting the chamber, this value on the plug will be considered when calculating the length of the press opening L_k . If we substitute the defined values into Equation 6.2, the calculated length of the press opening is $L_k = 94.97$ mm. In this way, we can calculate the length of the pressing chamber for the previously calculated values of p_m . It is necessary, though, to remind the reader that it is very important to interpret the results appropriately. Improper interpretation can result in a very large error in sizing the pressing chamber. This means that calculated values for L_k are values representing the length of the pressing chamber. However, they represent areas of the pressing chamber which produce pressure on the plug. For simplicity, an illustration is given in Figure 6.7, which is based on Figure 2.47.

Table 6.4: Table of the calculated pressing chamber lengths.

I	1	2	3	4	5
$p_{mi} = p_{Gi}$ (MPa)	0.48	1.45	4.39	13.23	39.84
L_k (mm)	237.80	190.19	142.48	94.97	47.49

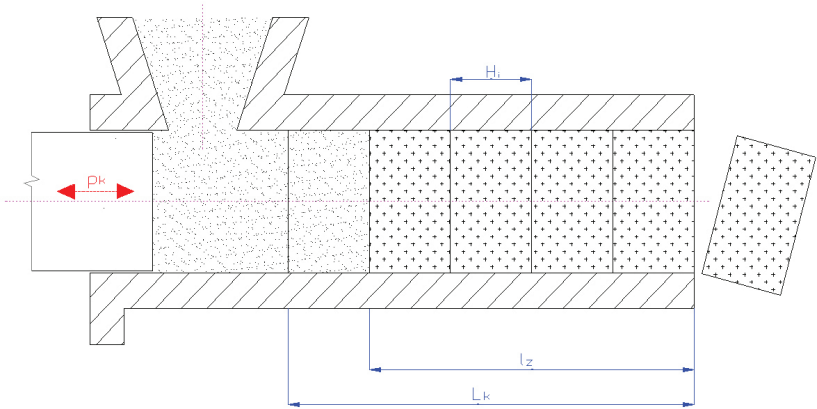


Figure 6.7: Calculation of pressing chamber length.

7 Summary of the Experimental Research and Results with Suggestions for Practice

Each designer has to adhere to certain knowledge when designing a new construction of a machine or innovating existing machines and machine parts. This knowledge refers to certain structural nodes, which describe the process taking place in the proposed machine. If the knowledge gained is comprehensive and accurate, it can be very useful and helpful in optimizing the equipment/machinery and output quality of the produced briquette as well as minimizing the input power requirements for the process. These factors significantly affect the economic requirements for operation a pressing machine.

At first glance, the densification process is a very simple and transparent process. If we devote more attention to this process and look at it in more detail, we find out that the densification process can be characterized by a large diversity of materials providing great opportunities for the resulting products applications. However, this process has not been sufficiently researched, and the experience gained is mainly used during the design of the pressing machine. It prolongs the design and manufacture process and makes the technology itself and products more expensive. However, densification machines belong among the devices that help to protect the environment by recovering various types of waste. This is the main reason why the debate has been reopened recently in this area, with the recommendation to support research and development and broaden the use of densification machines and products of these machines.

We desired to contribute to this field by optimizing the design of pressing machinery and the densification process through experimental research in our department. To describe the densification process and thus give recommendations for the design and construction of a pressing machine, a number of measurements must support the research.

This monograph presents the results of an **analysis of wood matter during the densification process**. It consists of the analysis of particulate material characteristics – disintegrated wood matter, analysis of parameters affecting the densification process, and the analysis of existing mathematical models that are in some way related to the process of densification. These analyses were very important for the design of an experimental pressing stand and creation of an experimental methodology. The experimental purpose was to define the behaviour of disintegrated wood matter and identify the impact of individual parameters when pressing this matter. An emphasis on determining the impact of a certain type of pressed material was defined by theoretical analysis. Each material has different chemical composition, and the lignin content in the raw material significantly affects the behaviour of the wood matter during the densification process. The type of pressed material decides the scale and nature of the pressing pressure, temperature, moisture content, and size



of the input fractions. These parameters are of major importance in the densification process.

The main contribution of the experimental research performed at our department was the design of a mathematical model in the process of uniaxial pressing and construction of an experimental pressing stand. Systematic work in area of densification resulted in **the design of an experimental pressing stand** used to measure the impact of select parameters affecting the densification process. The stand allowed certain parameters to be set and measured. Therefore, the necessary dependences and interactions of the examined parameters can be determined within a desired range. The effects of the studied parameters were evaluated with respect to the briquette density. After pressing the briquette with the set parameters, measurements were made and the resulting briquette density was calculated. The designed pressing stand allows us to find the effects of the examined parameters through an indirect method. The experimental pressing stand is designed in a way that allows for future experiments to determine the size of the radial stresses in the pressing chamber and to determine the impact of other design parameters (for example, the influence of chamber length, the impact conicity (taper ratio), the effect of the friction, etc.).

The **methodology for the design and evaluation of the experiment** has been shown within the experimental research. The complete experimental plan was used to measure the effects of pressing temperature, pressure, material moisture content, and fraction size. The variance analysis method (ANOVA), average analysis method, the test of equal variances (Bartlett's test), and affecting of factors method was used to detect the significance of these parameters. The mathematical models, which have different compositions of regression variables, have been proposed from the measured and processed data. One mathematical model was chosen based on evaluation using three different criteria for assessing the regression models. After computation of the regression parameters, the designed mathematical model of a densification process acquires real contours. The proposed methodology for the design and evaluation of experiments is a contribution for future experimental research in the field of densification.

Future ambitions include the **proposal of methodology to create a complex mathematical model**, which should take into account the nature of the material. It is possible to design a mathematical model for each specific type of material in certain closed intervals of validity by methodology presented in this monograph. However, this would require lengthy and laborious experimental research. The idea for designing a mathematical model that takes into account the nature of the material is as follows. It is known that there is a large set of materials that can be pressed and subsequently energetically recovered. For the experimental research, we would select 7-10 types of materials that would evenly represent the entire set of usable materials. For the selected 7-10 kinds of materials, experiments must be performed according to the methodology presented in this monograph. This would result in 7-10 individual and valid mathematical models. These models will be the basis for

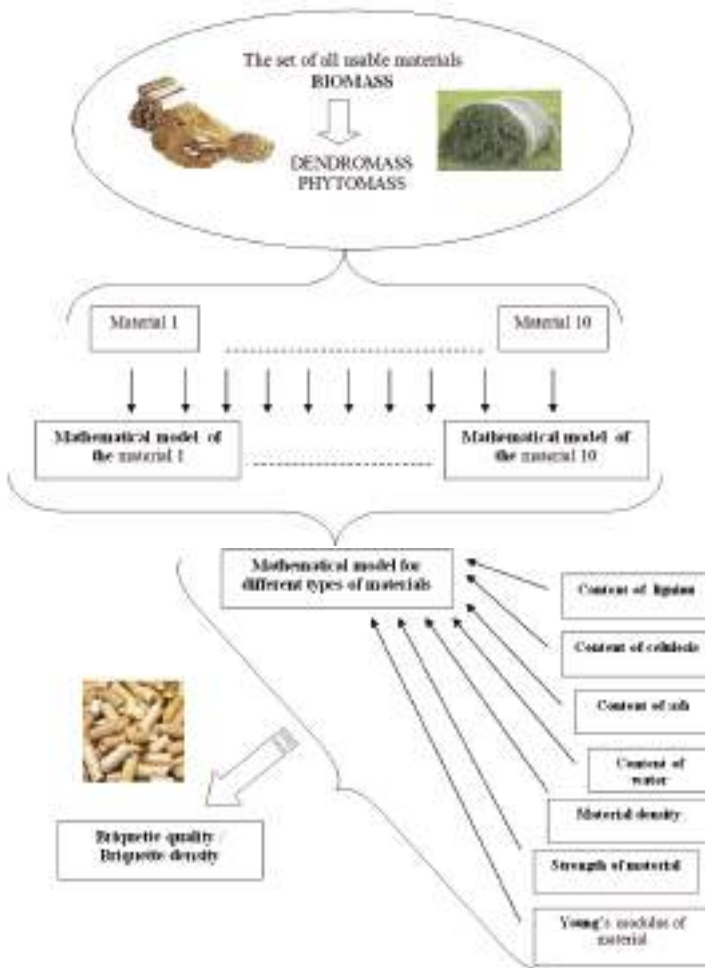


Figure 7.1: Scheme illustrating the design methodology for the development of a comprehensive mathematical model that takes into account the type of material being pressed (Križan, Matúš & Svátek, 2012; Svátek & Križan, 2013).

creating one comprehensive model by means of known statistical methods. It is also necessary to know the basic properties of relevant materials such as lignin, cellulose content, material density, strength, Young's modulus, ash and water content, heating potential, etc. A new mathematical model will work based on mathematical models for selected types of materials and will take into account the aforementioned material parameters (Figure 7.1). These models will define a set of materials where the new mathematical model is valid. When using a new mathematical model for material that has not been experimentally explored, it will be necessary to know only the material

parameters (mentioned above). Based on these parameters, the model will display the result (prediction of pressings density) even for any other kind of material that has not been subject to experimental research.

Based on the results of the experiments, it can be said with confidence that the **pressing temperature** has the greatest influence on the densification process. The results give a clear recommendation to designers to consider this fact and reflect it into designed densification machines in the form of a heating device for the pressing chamber. Most pressing machines are manufactured without a heating unit (Figure 7.2). Of course, pressing devices with added heating elements do exist (Figure 7.3). The heating unit ensures an optimum pressing temperature in the pressing chamber. This leads to an improvement in the quality of the final product, meaning an increased briquette density. It is also therefore possible to achieve the desired pressing density at lower pressing pressures. This reduces load and increases the durability of the pressing machine.

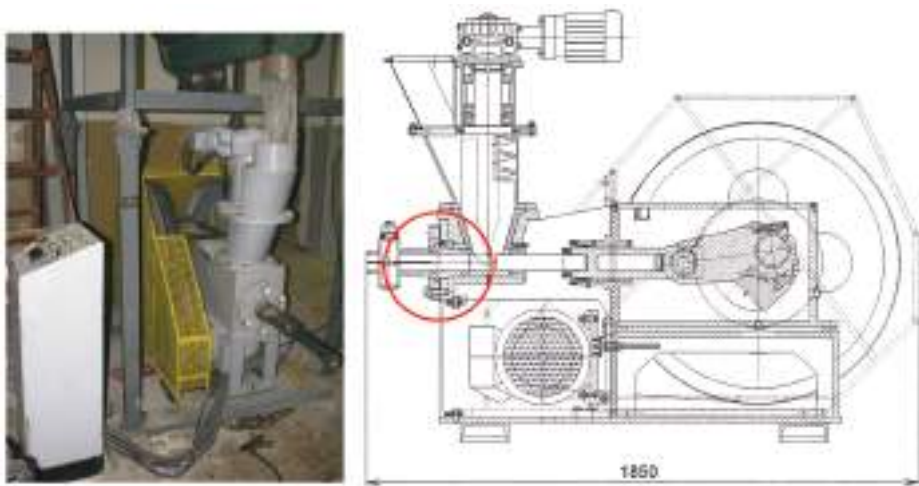


Figure 7.2: The mechanical (crank) briquetting press (red circle marks the area for mounting a heating unit).



Figure 7.3: An example of installing a heating unit on the mechanical briquetting press.

The practical contribution of this work is the design of a mathematical model describing the uniaxial pressing process. The monograph provides a tool for the effective and rapid prediction of resulting density of briquettes, values of pressing temperature and pressure, and values of input fraction moisture content and size. Experiments confirm the expected effects of the pressing temperature, pressure, moisture, and size of input fractions. The effects of the parameter interactions were confirmed as well. The mathematical model can be used as a basis for designing the optimal structure for a densification machine.

Another practical contribution is the **design of application software** for optimization of technological parameters and estimation of briquette quality according to the set parameters. This application will be useful for manufactures. Another use of the application is as a tool for optimizing technological parameters. Designers of pressing machines can use this application to predict pressing pressure and temperature.

The effect of the monitored parameters (pressing pressure, temperature, fraction moisture, and size) on the **dilatation of briquettes** was defined within the experiments. It was discovered that the dilatation of briquettes is strongly influenced by parameter settings and their mutual interactions. Dilatation is an undesirable phenomenon, but it must be considered. Reducing the impact of dilation can be achieved with optimal parameter settings or by shortening the stabilization time using a cooler. As seen in the following Figure 7.4 (pellet press) and Figure 7.5 (briquetting press), briquettes exiting from the pressing chamber (or matrix) are influenced strongly by the temperature, which rises in the densification process due to friction. It is necessary to shorten the stabilization time by cooling in production lines (Figure 7.6), mainly due to time constraints.

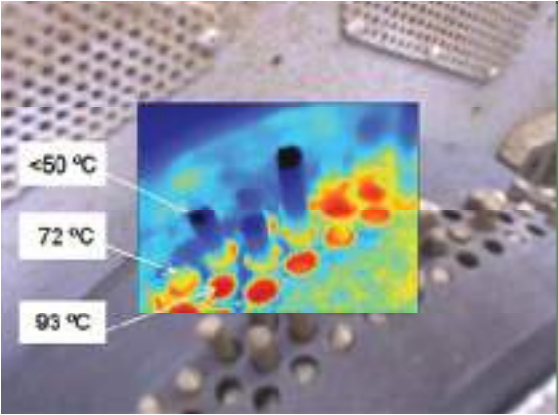


Figure 7.4: Infrared image of a pressing matrix in a pellet press (Stelte, 2011).

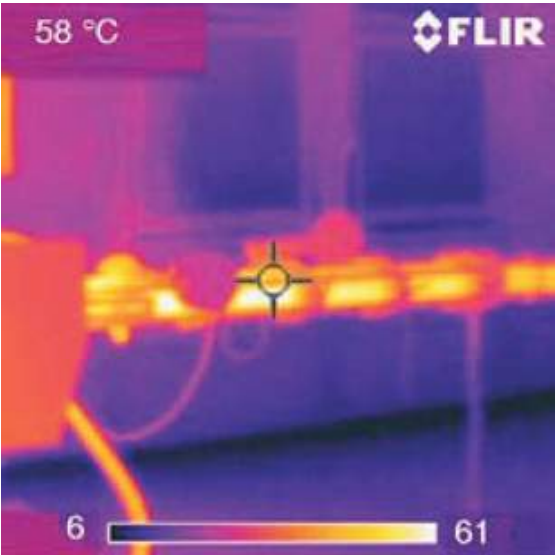


Figure 7.5: Infrared image of the pressing chamber with cooling channel (briquette press) (Holm & et al., 2005).



Figure 7.6: A cooling device installation on a briquette press.

Analysis of the existing mathematical models was performed; however, none of the analysed models clearly defines the relationship between resulting briquette quality (briquettes density) and pressing pressure, which is considered an important interaction in the densification process. The significance of the experiments was defined and quantified with respect to **optimizing the pressing chamber design** of a pressing machine. The mathematical models used to optimize the dimensions of the pressing chamber can be analysed in combination with the results of research performed at our department. It is very important to define the expression $\rho = f(p)$ for applicability of models into practice. Important design parameters of the densification machine that also affect the final quality of briquettes can be calculated by using published mathematical models. Calculation of design parameters such as optimal length of the pressing chamber, aperture diameter of the pressing chamber, radial pressure acting in the pressing chamber, and the back-pressure acting on plug in the pressing chamber are important to properly design the pressing area of the machine. The desired output quality of briquettes and increased efficiency of the densification process can be achieved by the correct design of the pressing chamber parameters. The idea of the pressing chamber in a pressing machine is to optimize the pressing to ensure that the resulting briquette is solid, compact, and achieves a certain quality given by Standards (Figure 7.7). The effects of the mentioned parameters can be computed using analysed mathematical models. However, it will be important to conduct experimental research of structural parameters impacting the resulting briquettes density in the near future.



Figure 7.7: A compacted briquette leaving the pressing chamber.

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