Universitatea
Transilvania
din Brașov

INTERDISCIPLINARY DOCTORAL SCHOOL

Faculty: FURNITURE DESIGN AND WOOD ENGINEERING

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CONTRIBUTIONS TO THE PRODUCTION, TESTING AND QUALITATIVE EVALUATION OF BRIQUETTES AND PELLETS FROM WOODY AND VEGETABLE BIOMASS

SUMMARY

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Doctoral field: Forestry engineering

BRAȘOV, 2023

LIST OF NOTATIONS AND ABBREVIATIONS

Title	Definition (reference)
Additive	Material that improves biofuel quality, reduces emissions of harmful elements or helps increase productivity and efficiency of the manufacturing process.
Biomass	Biodegradable fraction of products, waste and residues of biological origin from agriculture (including vegetal and animal substances), forestry and related industries including fisheries and aquaculture, and biodegradable fraction of industrial and municipal waste. (Directorate 2009/28/EC, Official Journal of the European Union, L140/27, 5.6.2009)
Biofuel	Fuel directly or indirectly produced from biomass. (Marian G. 2016)
Briquettes	Product obtained by briquetting small or powdery material into regular geometric shapes (rectangular, cylindrical, ovoid, etc.) for transport, use or further processing (DEX 1996).
Ash	Solid mineral residue left after complete combustion of fuel.
Calorific density	The multiplication of the superior calorific value of briquettes/pellets and the density of briquettes/pellets.
Pellets	Compressed wood biomass of cylindrical shape, with or without additives. (Marian G. 2016)
Calorific value	The amount of heat contained in fuel, determined by measuring the heat produced by complete combustion of one unit of combustible mass, excepting the energy obtained from condensation of the vapour obtained.
Torrefaction	Method for improving the properties of biomass used as fuel. The treatment consists in slowly heating the biomass in an inert atmosphere to a maximum temperature of 300°C.

INTRODUCTION

Agricultural activities generate a significant amount of residues that can be valued by producing solid biofuels. Pellets are one of the most widespread types of densified solid biofuels, but also they ask particular quality requirements. For this reason, many agricultural residues cannot be processed into qualitative pellets by traditional methods used today in the solid biofuels industry (Gudima etc. 2017).

In our days, biomass accounts for 15% of all energy sources worldwide. Although it is an abundant resource, the use of energy contained is not consistent developped.

This study is an special approach on the field of wood and vegetable biomass processing.

Given the existence of many researches reviewing the issue of briquettes and pellets plant and forest based resources (Grîu, 2014, Gudîma, 2018), some aspects - such as making, testing and evaluating these solid biofuels - are the subject of controversy, which creates the need for deepening.

The production of briquettes and pellets from wood and vegetable biomass, as well as their use for obtaining energy, given existing conditions in our country, is an important element to consider, both in terms of the possibilities of uninterrupted supply and the low cost.

In light of the above, the motivation of the doctoral thesis acquire significance and reveals the importance of focusing on the research for new solutions for the realization, testing and evaluation of wood and vegetable biomass briquettes and pellets, created for the purpose of producing green energy.

The doctoral thesis "Contributions to the realization, testing and qualitative evaluation of wood and vegetable biomass briquettes and pellets" is structured on 6 chapters.

Chapter I, "*Current state of research in the field*", is a selective bibliographic summary of the database and information on the raw material used for the production of lignocellulosic briquettes and pellets.

Bibliographic synthesis is useful for understanding and study the original content of doctoral research and helps to highlight current issues, treated in the chapter 2 of the thesis, "Objectives of the thesis".

Chapter 3 "*Theoretical research*" presents the installations used, the materials used for experimentating and software used. Here we present the methods and measuring instruments used, adapted to the subject of thesis.

Chapter 4 "*Experimental research*" includes experimental research on the production and testing of wood and vegetable biomass briquettes and pellets.

Chapter 5 "*Quality of briquettes and lignocellulosic pellets*" covers theoretical research and experiments with different methods and applications for measuring the quality of briquettes or pellets.

In Chapter 6 "Final *Conclsions. Own contributions. Valorization of results. Future research directions*" are synthesized the defining elements of doctoral research, personal contributions to the theme and perspectives for continuing research in the future.The study is the result of studies conducted at the Doctoral School of the "Transilvania" University of Brasov.

CHAPTER 1. CURRENT STATE OF RESEARCH IN THE FIELD

1.1. Raw material base

By biomass, as a renewable energy resource, we understand the biodegradable part of bioresources generated by plant photosynthesis. Biomass is the main component of biofuels, a raw material for energy generation.

Residues are originating from the processing of raw materials to produce goods, in technological or biological processes. The value of residues is small compared to the finished product produced, but it is not negligible.

Spring wheat, sunflower, maize, barley and autumn wheat are the main agricultural crops generating biodegradable waste in the form of biomass.

Vegetal residues presents important potential energy, with the lowest production costs, because they require only collection, transport and storage expenses (Salaru etc.,2013).

1.2. Lignocellulose briquettes

Wood waste is a renewable energy source that plays an important role in the global energy market. It is a substantial part of biomass and was the main alternative source of energy. The woodworking industry is an inexhaustible source of wood waste, which must be evaluated in order to find the most efficient methods of energy valorisation.

There are two methods of torrefaction: wet (hydrothermal treatment) and dry (torrefaction). Dry heat treatment takes place between 200 and 300°C with a duration of approximately 30 min for sawdust (Almeida etc.,2010) or approximately 72 h for timber (Chiaramonti etc., 2010). The torrefaction process of rapid heat treatment is most effective in increasing the carbon content of wood (Almeida et al.,2010; Chen et al.,2011a, b; Sarvaramini et al.,2014).

Torefaction has several advantages, such as dimensional stabilization of the wood product (Pelaez-Samaniego etc., 2013) and reduction of hydrophilia. Four species of wood sawdust were used in the experiments: beech (Fagus sylvatica), oak (Quercus robur), spruce (Picea abies) and larch (Larix decidua).

Spîrchez et al.(2018) obtained wood briquettes with a hollow core made from spruce and oak wood waste. A comparison was made with the classic types of briquettes in order to be able to identify the their advantages and disadvantages. The results shows that there are few differences between the characteristics of hollow core wood briquettes and the classic ones.

These differences depend on the pressing method and equipment, compared with other briquettes without a hollow core. Given their economical type, it is completely possible to manufacture these briquettes suitable for cooking and heating (Spîrchez et al.,2018).

The briquettes obtained could successfully replace wood sawdust briquettes or even wood, due to their good density and very good calorific value. The ash content of 2.4% is similar to that for European wood species. Burning briquettes obtained from sunflower husks produces clean energy, used for stoves or ovens (Spîrchez, etc.,2019).

The briquettes obtained from larch chips have a density of 1024.8 kg/m^3 and a high calorific value of 19375 kJ/kg. Even though they have lower mechanical resistances, briquettes obtained from sunflower seed husks are calorifically efficient, with calorific values of 19265 kJ/kg, close to wood briquettes. The ash content of 2.34% is within the range of briquettes obtained from plant resources and wood waste. Briquettes from sunflower seed pods are becoming a viable alternative to wood briquettes.

Paulownia is one of the fastest growing woody species in the world. Making good quality briquettes from large sawdust paulownia lumber cutting is a big challenge.
Bamboo species are perennial plants, and their growth harmonizes with the principles of sustainability, due to the rapid growth and annual production of stems (Pereira and Beraldo,2016). For that briquettes, 4 species of bamboo were analyzed. After carrying out the experiments, the results showed variable figures: compressive strength 4.68 - 5.82 MPa; volatile content 79.01 - 82.25%; fixed carbon content 15.26 - 20.18%; ash content 0.38% - 2.49%; gross calorific value 4571 - 4716 kJ/kg.

For the production of lignocellulose briquettes wood waste was also used. Based on the results obtained, it was found that the moisture content of wood waste used for the production of briquettes ranged from 10.7-14.8%, while their calorific value was within the limits of 15.5-16.3 MJ/kg.

Agro-forestry industries such as sugar-alcohol, food and logging produces large amounts of waste, used to generate energy from direct combustion (International Energy Agency France,2018; Maroušek,2014). The application of other processes, such as torrefaction and briquetting, can increase the profits for agro-industrial waste used for energy. Four types of biomass were used, waste from pine and eucalyptus wood, waste from cutting coffee and torrefied sugar cane. Roasting increased the fixed carbon content, ash and calorific value and reduced the volatile matter content and hygroscopic equilibrium moisture of the biomass.

Eucalyptus and pine processing wastes, Arabica coffee and sugarcane, without bark, leaf or root residues, were air-dried to a moisture content of about 20%. The torrefaction process reduced the biomass moisture for those from nature. Comparing the materials without roasting, the Arabica coffee residue had a higher fixed carbon content, while the pine residue had a higher calorific value. A reduction of 17 times over the density of the material was achieved.The gray and volatile values for sugarcane waste were 3.9% and 87.7%, while for starch they were 0.09% and 94.65%. The gross calorific value was 3,593 MJ/kg for bagassa and 3,637 MJ/kg for starch.

All mixtures were made according to the established recipe, processed in the form of briquettes with 12% moisture. The carbon content of the briquettes produced ranged from 41.85% to 43.84%, and the sulfur was below 0.1%. The calorific value of the produced briquettes ranged from 3359.4 to 4028.3 kcal/kg and the ash was below 6%. The use of coffee husks or isolated branches and leaves, as well as the mixture of coffee husks with 50% or more branches of leaves allows the production of briquettes with a calorific value higher than about 4000 kcal/kg, which complies the quality parameters. The briquetting of coffee crop residues is viable and sustainable from an energetic point of view (Oberdan et al.,2014).

1.3. Lignocellulose pellets

Pelletization of forestry and agricultural materials is an alternative in obtaining value-added products for energy generation, since many residues from both industries can be used in this process. A method of making sawdust more efficient is torrefaction followed by pelletization (Lunguleasa et al., 2017; Lunguleasa et al., 2019). Pelletisation is widely used to improve the mechanical qualities or physico-chemical characteristics of wood biomass in the form of chips, sawdust, chips, solid wood, briquettes and pellets.

Lunguleasa et al. (2019) analyzed four wood species, beech, spruce, larch and oak, which were thermally treated as sawdust waste at temperatures of 200, 220, 240, 260 , 280 and 300°C for different periods of 3, 5 and 10 min. The results indicated an increase in calorific value and density with temperature. Economicaly speaking, pellets obtained from torrefied sawdust had better properties than untreated ones.

Three composite samples were used to produce the pellets: corn cobs, corn stalks, and a 50:50 composite of corn cobs and corn stalks. Each of the composite samples was thoroughly mixed with a locally produced cassava starch using a kneading process. (Sulaiman et al., 2019).

A mixture of rice husks (Oryza sativa L.) and wood particles from Gmelina arborea was used to produce the pellets (Baetge et al.,2018). Using rice husk as a raw material to produce fuel pellets, a large volume of this biomass could be used (Missagia et al.,2011). In general, except for the ash content, the physical and mechanical properties of these pellets were within acceptable limits to be used for industrial heat applications (Bajo and Acda,2017).

In order to reduce the amount of ash from combustion and raise the melting point at the same time, use of waste paper for the production of wheat straw pellets was analyzed with proportion from 10% to 30%. Reference pellets were 1:1 wheat straw and waste paper were produced, for comparison. With a 10% waste paper content, the lower calorific value decreased by about 10% compared to the calorific value in the reference pellets. The positive effect of waste paper was manifested by its ability to increase the melting point of ash from plant biomass pellets. Pelletized waste paper would increase the melting temperature and ash content, but decrease the lower calorific value (Nosek,2020).

Kachel et al., 2020, realised an analysis of selected qualitative characteristics of pellets produced from rape seed straw obtained from fertilised crops and from straw mixtures selected for testing with crude glycerol obtained as a by-product of biodiesel production. The obtained results indicated that the different treatment schemes applied in the spring rape crops had a significant impact on the physico-chemical qualities of the straws. Furthermore, in all the mixtures analyzed, the addition of 10% crude glycerol improved the mechanical characteristics of the produced straw pellets. Starch proved to be a good additive in the production of pellets (Găgeanu et al.,2017).

Using materials such as corn cobs, corn husks, alfalfa straw in combination with woody biomass has ensured that by-products from agriculture, which we would otherwise waste, are valorised and transformed into energy. The combination in the sample (wheat straw + alfalfa straw + corn cobs + corn husk) leads to the highest pelleting time and energy consumption. The combinations between woody biomass and agricultural biomass showed good results in terms of lower calorific value, ash content, bulk density (Găgeanu et al.,2017).

Gudima (2018) used a series consisting of pellet samples, for which he determined the calorific value and ash content, with variable mixtures of straw, energy willow and acacia.

The analysis of the study data, respectively of the values of the ash content resulting from the burning of the samples, highlighted the different influence of compositions with woody biomass (energy willow and acacia), respectively with straw, on the calorific values of the pellets manufactured according to the recipes. The increase in the calorific value for all the samples reached 16.5 MJ/kg, which allows the pellet studied to be registered in the EN plus standard compliance category. (Gudîma,2018).

1.4. Quality assessment of briquettes and pellets

Quality represents the measure of the material value of the products, regardless of the product or the purpose for which it was created. Qualitologist Juran defined quality as that characteristic that is relevant to users.

The quality of briquettes and pellets can be assessed by the estimated value method (Lunguleasa,2015), the European standardization and the distance method from the limiting value. (Spîrchez et al.2016)

1.5. Critical analysis of the current state and conclusions

A consistent and generally valid method of qualitative analysis of a batch of briquettes or pellets has not been realized or identified.

No combinations or recipes of forest and agricultural species were found to significantly improve those made from plant resources alone.

No correlation was made between the sizes of the lignocellulosic assortments introduced during briquetting and the characteristics of the briquettes obtained.

Household and industrial energy needs are covered significantly and increasingly, especially in rural areas, by sources using briquettes and pellets. Studies on the use of straws for briquettes are not consistent and do not highlight their advantages and disadvantages.

Even if they have lower mechanical resistance, briquettes obtained from sunflower seed husks can be used in their natural state, without additional processing, because they are calorifically efficient.

Using fast-growing wood species such as Paulownia can create compact and durable briquettes and pellets during storage and handling. This species could be used in the near future in briquettes and pellets, being cheaper and of higher quality.

The result of use of the four bamboo species in briquettes presented satisfactory physical and energetic properties.

Waste from the market can be successfully used to obtain briquettes and pellets. To add value to these materials, there were produced torrefied briquettes.

When the fuel used has low density, natural binders and additives can be successfully used: cassava starch, gum arabic, cow dung and corn starch.

Using wood waste of various forms and species, like poplar sawdust, poplar bark, oak shavings and oak bark to produce lignocellulosic briquettes can lead to obtain wanted biofuels, favorable to achieving the objectives of the sustainable agricultural policy.

Briquettes produced with residues from the coffee culture have physico-chemical characteristics that prove to be a viable source for energy generation.

Banana leaf briquettes showed similar thermal comportment and physical and chemical characteristics to other biomass already used as fuel for power generation.

Combining sawdust with paper, barnyard rice and cow dung in various ratios, can result in briquettes that have improved characteristics.

The combination of peanut shells, rice husks and sawdust by Daniella Oliveri and starch as a binder demonstrated that the ratio of starch used can influence the burning properties.

By adding 1% corn starch to the biomass materials, the lower calorific value of the product pellets was increased; pellet moisture decreased compared to non-starch samples, leading to better storage and combustion.

Straw pellets with woody biomass- 30% energetic willow and 30% acacia- increased in calorific value by 9.6%, up to the value of 18.49 MJ/kg.

Combinations of woody biomass and agricultural biomass showed good results in terms of lower calorific value, ash content, bulk density.

Recipe changes can lead to increased production, reduced energy requirements per unit of product and also improved quality. When used in combination with different types of biomass materials, each additive and binder leads to improved thermal characteristics.

An exhaustive study covering all the links of the chain of production, testing and quality assessment of briquette/pellet batches has not been carried out.

CHAPTER 2. OBJECTIVES OF THE THESIS

This study investigates the possibilities of production, testing and qualitative evaluation of woody and plant biomass briquettes and pellets, fallowing 3 general objectives:

O1.Producing briquettes and pellets using woody and vegetable biomass, in various combinations, on different installations and presses in terms of operation mode and capacity, in order to observe the differentiation of properties according to these aspects. The main activities of this objective were:

- the use of at least 2 briquetting presses and 2 pelletizing presses;
- the use of forest and agricultural species less researched to date;
- the use of combinations that are beneficial for obtaining briquettes and pellets;
- observation of the influence of the granulometry of sawdust from briquetting and the compaction coefficient on the resistance of the briquettes;
- determining the abrasion of lignocellulosic briquettes.

O2. Testing of the main physical, chemical, calorific and mechanical properties of briquettes and pellets obtained in a laboratory or industrial setting. Derived activities from this objective can include:

- for briquettes, those activities such as testing humidity, effective density, calorific value, ash content, volatile matter content, resistance to compression or splitting and abrasion resistance;
- additionally, for lignocellulosic pellets, bulk density and shear strength will specificaly measured.

O3. Finding a cumulative method for determining the quality of briquettes and pellets. Derived activities are:

- defining the quality of briquettes and pellets;
- finding the elements that contribute to define quality;
- providing practical examples of determining the quality of these types of products.

In order to achieve the proposed objectives, it was necessary to go through several research stages specific to a doctoral thesis:

- biographical research - documentation in order to identify the possibilities of solving the doctoral thesis, taking into account the current stage of research;
- experimental research – establishing experimentation methodology and its application;
- applied research – the experimental study of the proposed formulas for qualitative testing and evaluation/ in order to test and evaluate the quality, considering the novelty of the topic;
- Interdisciplinary approach: wood structure and composition, graphics, statistical processing of experimental data, computer science, etc.

CHAPTER 3. THEORETICAL RESEARCH (METHODOLOGY, FACILITIES AND MATERIALS)

The purpose of this chapter is to establish the working methodology, equipment, materials and facilities used, as well as the proposed experimental plan. For each property of briquettes and pellets, an appropriate method of determination and testing was set, most of the time using the standardized test according to the European standards. In cases of no European standard for determination, a method known or frequently used by national and international laboratories was used.

3.1. Moisture content of chopped material, briquettes and lignocellulosic pellets

The moisture content of the chopped material for briquettes and pellets was determined using the gravimetric method. A Kern electronic balance was used for this purpose. Drying the samples to the anhydrous state was performed with a Memmert laboratory oven (Ulm, Germany).

3.2. Granulometry of chopped material

The granulometry of the chopped material used for briquettes and pellets production denotes the manufacturer certain characteristics of future products. Usually, specifying certain size ranges or average value of the chopped material is not enough and does not provide an accurate portrayal of the material, as granulometry does. For the tests of the study we used a vibrating device with different sorting sieves.

3.3. Density of fine material. Bulk pellet density

For the small raw material in the form of chips, sawdust or dust, as well as for pellets, due to their small dimensions, their bulk density was determined using the same methodology. Bulk density was determined using frustoconical vessels and mass measurement was made using a Kern analytical balance (Germany). To obtain the accurate height of the material in the cylinder - the volume of the graduated cylinder, the test vessel was held on a horizontal vibration device for 3 minutes.

3.4. The degree of loosening of fine material

The degree of looseness of the chopped material is a dimensionless coefficient and quantifies the existence of voids in the mass of the chopped material, compared to the raw material of lumber. For the determination, the density method was used, specifically comparing the density of the solid material to the density of the chopped material.

3.5. The production of lignocellulosic briquettes

Lignocellulosic briquettes were produced from each type of remains using a Gold Star hydraulic briquetting machine (Brașov, Romania), equipped with two pistons with perpendicular action and a silo of about 1.5 m^3. The circular motion of the fine material in the silo of the briquetting machine was also pneumatic, derived from the same movement of the main piston, through a lever.
Lignocellulosic briquettes were also made using the hydraulic briquetting machine, equipped with a safety switch for oil temperature.

3.6. The production of lignocellulosic pellets

The production of lignocellulosic pellets was executed using a Saras type laboratory installation, operated by the company with the same name (Saras SRL, Brașov, Romania), with a capacity of 50 kg/h. The actual pellets were obtained after about 2 minutes of operation, during which the fine material was heated by the friction between the rollers and the mold to temperatures of 80-950 ^{0}C, in order to activate the lignin in the used lignocellulosic material.

The high-capacity industrial pellet press is constructed in a robust construction, of U-shaped metal profiles. The material was compressed by the two pressing rollers on the mold. The continuous compacted material emerging from each hole is cut with a knife and the pellets are discharged through a chute. The rotational movement is transmitted from the electric motor to the mold via a bevel conical gearbox.

3.7. Compressive strength of lignocellulosic briquettes

The compressive strength indicates the consistency and compaction of lignocellulosic briquettes (Roser et al.,2006). There is no similar strength in this area, but there are some similarities with of solid wood, other wood-based panels and for concrete. This test helps us understand how well the briquettes withstand stacking during transport or in storage.

Figura 3.10: The machine for determining compressive strength
(made in China/of Chinese production)

Figura 3.11: Sequences during the determination of compressive resistance/strength of lignocellulosic briquettes
a - for lignocellulosic briquettes without voids;
b - for hollow lignocellulosic briquettes/without voids.

The compressive strength of lignocellulosic briquettes corresponds to stress distribution during fracture as well as the shape of the briquette after fracture.

Figure 3.13: Lignocellulosic briquettes obtained during the determination of compressive strength

3.8. Splitting resistance of lignocellulosic briquettes

Due to the fact that lignocellulosic briquettes are products made of fine, almost granular material, it was necessary to introduce a new property of briquettes, namely resistance to splitting. This property measures the strength of the briquettes when we want to halve those which are longer than the size of the feed mouth of a thermal plant. Such stresses can also occur during transport, alongside those of

Figure 3.14.The machine used for determining splitting strength (made in Germany)

shear or compression. A blunt knife was designed for this test.

3.9. Shear strength of lignocellulosic pellets

The shear strength is the primary mechanical property of the lignocellulosic pellets. Pellet shearing can occur during storage on a pallet with several bags of pellets stacked at a certain height, resulting in their breaking or splitting.

The values of shearing and crushing resistance of pellets have practical importance to pellet users. If the pellets do not have good shear strength, they will easily break during transportation and handling. The shear force was determined using a universal testing machine (Ulm, Germany) with specific devices and a reduced speed of action force application of approximately 10 mm/min.

In order to determine the shear resistance of the pellets, two parallel contact plates (with a distance of 0.4 mm between them) were used, with the lower plate fixed and the upper plate displaceable, which represents the blade with a sharpening angle of about 65^0, creating the shear plane. Since the shearing force of a pellet is very low, five 6 mm holes were created on the fixed plate, in order to simultaneously shear the 5 pellets, thereby obtaining a force 5 times higher, which can be highlighted with a higher accuracy on the dial of the testing machine.

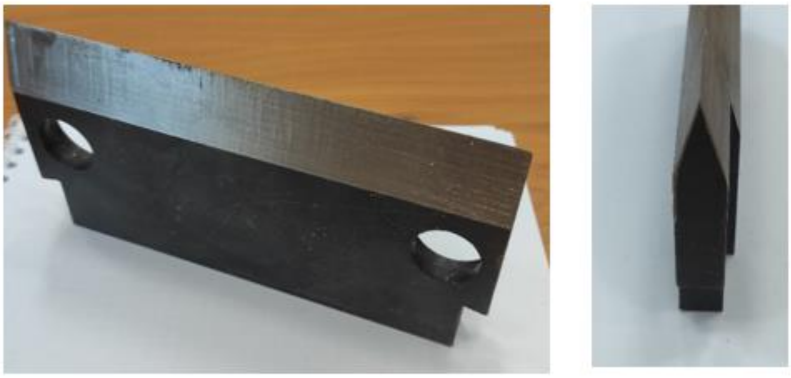


Figure 3.16. The upper plate shaped with an angle of 65^0

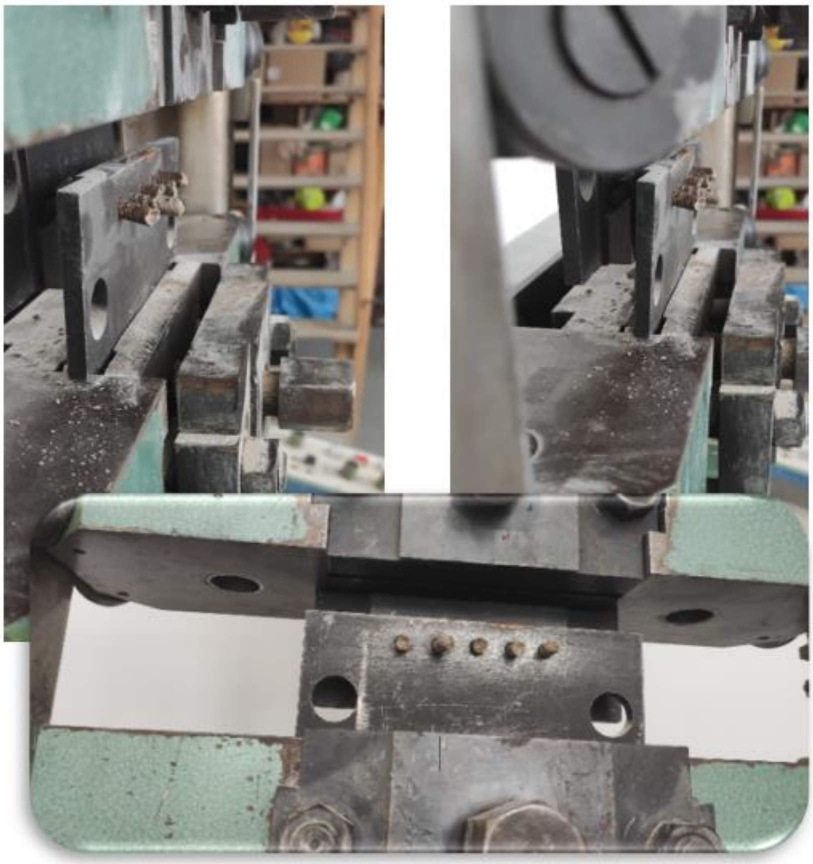


Figure 3.17. Sequences during the determination of shear resistance of lignocellulosic pellets

3.10. Calorific value of briquettes and pellets

The calorific value is the primary thermal property of briquettes and pellets, providing information about the amount of heat obtained from the combustion of wood and plant biomass.

The calorific value of the briquettes and pellets was determined experimentally using an installation equipped with a bomb calorimeter, type XRY-1C, produced by Shanghai Changji Geological Instrument Co. Pellets with the same mass were used to determine calorific value, given that the calorific value does not depend on the shape of the lignocellulosic material, but the solely on the type of material and the lignocellulosic species used.

3.11. Other calorific properties of lignocellulosic materials

Based on the calorific value of the lignocellulosic material and other properties, other calorific characteristics such as calorific density, burning rate and calorific efficiency have been determined.

The energy release rate was determined by relating the calorific value to the burning time per kg, which is equal to the time of the main period in the calorific value per kg test.

The energy efficiency of burning briquettes and pellets was determined based on the maximum calorific value of the briquettes at 0% moisture content. The actual calorific value at 8-12% moisture of the briquettes and pellets represented a certain percentage of the maximum possible calorific value.

Furthermore, based on the obtained results, it was possible to determine the influence of humidity on the calorific value, the limiting value of the moisture value, the calorific value per hectare in the case for plant resources, etc.

3.12. Ash content

After combustion, the inorganic elements in the composition of biofuels, are transformed into a solid residual content in the form of ash.

The quality characteristic known as ash content is particularly important in the case of biofuels. The presence of ash reduces the quality of biofuels, especially the calorific value, negatively affects the combustion process, increases corrosion in boilers, and increases the costs of transportation, storage and disposal.

The ash content of the briquettes helps determine the mass quantity of ash produced, necessary for its periodic removal from the boiler and its processing installation.

Drying was made using a Memmert oven (Ulm, Germany), at a temperature of 105 0C, for at least 1 h, until there no differences could be observed between 2 successive weighings. The actual determination test utilized an STS-type calcination furnace, heated to a temperature of 750 0C for complete calcination, using high-temperature resistant metal crucibles. The calcination operation was considered complete when the material had a light gray color and no sparks were observed above the crucible. After calcination for 2 h, the ash crucibles were cooled in a desiccator and re-weighed with the precision balance.

For the protection of the calcination oven, the fine material was initially burned over a butane gas flame until no further smoke was produced. Since this form of superficially carbonized material was dark black, it was called as black ash, and its ash content was determined accordingly.

3.13. Volatile matter content

Thermal instability is inherent to all organic substances, in varying degrees. When heating solid biofuels by burning them without air supply, the moisture content first evaporates, followed by an destructive action on the molecules accompanied by the volatilization of some gaseous substances. These emitted gaseous substances and vapors constitute the volatile matter.

3.14. Briquettes abrasion

Abrasion of solid briquette materials aimed to determine the amount of dust and small chips generated during their movement on a sieve. Short briquettes were used for this test. An electric vibrating device was used for abrasion. After vibrating for 5 minutes, the mass of dust and fine material that passed through the 4x4 mm sieve was determined.

3.15. Pellets and fine material torrefaction

The torrefaction treatment of fine material, briquettes or pellets had a dual purpose, namely to slightly increase their calorific value and to reduce their hygroscopicity. Torrefaction is a slow incomplete pyrolysis, in which the pellets/briquettes are enriched in carbon at high temperatures. This torrefaction treatment was carried out in a laboratory oven, at temperatures of 200 and 220^{0}C, for a period of 1, 2 and 3 hours. The procedure aimed to measure the decrease in the mass of the dry and heat-treated material by weighing before and after the treatment.

3.16. Conclusions regarding methodology, equipment and materials

The woody and plant species were selected based on their quality characteristics for use as raw materials in the production of solid biofuels.

The research was conducted using a methodology specific to the field, including the development of a suitable experimental plan for the analysis of briquettes and pellets obtained from woody and vegetable biomass. The physical briquettes and pellets samples were analyzed in the laboratory of "*Transilvania*" University Brașov.

Based on the results obtained from the experiments, series of data were collected, allowing for the analysis of the physical and compositional characteristics of the briquettes and pellets according to the intended purpose.

The raw materials used for the research studies were selected taking into account the following:

- beech is a native species, and most widely used - accounting for 31% of the total round wood harvested in 2022. The resulting waste from processing is significant;
- sunflower is the agricultural crop grown on an area of 1082 thousand ha. of cultivated agricultural land in Romania;
- larch is one of the resinous species of great importance for the national economy, due to its superior qualities, rapid growth and high productivity;
- the combination of larch and sunflower husks aimed to analyze the fact that larch contains 20-22% resin and I wanted to study if this can help a better compaction
- oak is a native species with high economic and financial value, it being considered important to use all the waste generated during processing;
- Paulownia is one of the fastest-growing energy trees, whose cultivation has gained significant momentum in recent years, including in Romania. Its characteristics and properties, as well as interest in its inclusion in the economic circuit, made it important to include it in the research;
- because cardboard is made of cellulose, a woody fiber, and considering that approximately 30% cardboard remains unrecycled annually in the European Union, its inclusion in the composition of pellets was considered beneficial.

CHAPTER 4. EXPERIMENTAL RESEARCH

4.1. Results concerning the use of sunflower seed husks and larch sawdust in briquettes

Comparison between larch and sunflower seed shells was made to identify the differences in density, so that later, their influence on the properties of the briquettes could be identified. Also, the use of larch remains was to analyze the influence of resin content on briquette properties.

The intent of the research was to produce good quality briquettes from sunflower (*Helianthus annuus*) seed husks. Larch sawdust was used for briquettes, to compare their quality. Bulk density of larch sawdust was 118.5 kg/m^3.

The average dimensions of the 30 briquette samples made from larch sawdust were: diameter 40.8±0.1 mm and length 30.3±5.3 mm. The degree of expansion of the diameter of the briquette after pressing of only 2%, shows the very good compaction of larch sawdust. The average density of these briquettes was 102.4±147.3 kg/m^3 .

The breaking strength of larch briquettes was 2.04 ±0.2 N/mm^2, comparable to that found by other researches in the field (Spîrchez et al.2019). The calorific value of larch sawdust briquettes was 19375 kJ/kg and 18906 kJ/kg. The ash content of larch sawdust briquettes was 0.4%.

Bulk density sunflower seed hulls was 207±19 kg/m^3 for raw state and 301±26 kg/m^3 for ground state. Briquettes dimensions: average diameter 40.8±0.5mm and length 44±5mm. Not corresponding briquettes after press was 2%.

The effective density of briquettes obtained directly from unground shells was 680±7.3 kg/m3, and that of briquettes obtained from ground shells was 854±73.7 kg/m^3. The breaking strength of briquettes was 0.51 N/mm^2 for those obtained from large seed husks and 0.09 N/mm^2 for those obtained from ground husks.

Since a big difference was observed between the strengths of the two types of briquettes, a mixture of sawdust and sunflower seed husks was experimentally made, in different percentages, respectively 25, 50, and 75%. The results were inconclusive. From this it can be concluded that the mixture of sunflower seed husks and larch sawdust is incompatible and that other solutions should be found to improve the resistance of the briquettes.

In order to observe the influence of the briquetting press on the physical-mechanical properties of briquettes obtained from sunflower seed shells, briquettes made on an industrial screw press were checked, having an average density of 921.8±27.6 kg/m^3 and an average strength of 0.46±0.07 N/mm^2.

It is noted that although briquette density increased greatly from 680 kg/m^3 on the hydraulic press to 921.8 kg/m^3 on the screw press, the compressive strength decreased from 0.51 N/mm^2 to 0.46 N/mm^2.

The calorific value of briquettes made from sunflower seed husks recorded the value of 19265 kJ/kg, while for the lower calorific value the average value was 18655 kJ/kg.
The ash content of sunflower seed husks was 30.72% for black ash and 2.34% for calcined ash, while the content of volatile substances was 73.2% and the active carbon of 24, 4%.

As a general conclusion of the research, it can be stated that larch sawdust is well compressible and briquettes with good mechanical properties are obtained from it, and sunflower seed shells have weaker strengths, even if the densities are acceptable.

4.2. Results concerning briquettes and pellets obtained from two types of paulownia sawdust

The main aim of the research was to find the optimal conditions for obtaining good quality briquettes and pellets from large sawdust from sawing paulownia timber. Although not widely distributed worldwide, paulownia crops are becoming more and more attractive due to reaching wood maturity of up to 10 years, that is, about 10 times faster than the usual softwood species. Two types of sawdust were used, namely Paulownia tomentosa and Paulownia elongata sawdust.

The average values of the bulk density of the two types of sawdust, respectively 146.5 kg/m^3 in the case of Paulownia Tomentosa and 126.2 kg/m^3 in the case of Paulownia elongata.

The pellet density of 1268 kg/m^3 was higher than that of briquettes of 790 kg/m^3 for Paulownia elongata and 1266 kg/m^3 for pellets and 934 kg/m^3 for briquettes respectively for Paulownia tomentosa, this being determined by the principle of obtaining them, respectively hydraulic in the case of briquettes and mechanical extrusion by rolling to pellets.

The resulting calorific value did not depend on the type of compressed product or the type of species analyzed, the values obtained being within the confidence interval. In this sense, Icka et al. (2016) found a calorific value of 15-18 MJ/kg.
The content of black and calcined ash was significantly different for the two paulownia species considered, being higher for Paulownia elongata than Paulownia tomentosa. A 14.5% increase in the amount of black ash and a 72.6% increase in the amount of calcined ash were identified.

The compressive breaking strength of Paulownia elongata briquettes averaged 0.5 N/mm^2, and Paulownia tomentosa briquettes averaged 1.7 N/mm^2. The differences in the resistance to breaking are related in particular to the density of these briquettes, respectively the Paulownia tomentosa briquettes had a density (934 kg/m^3), 18.2% higher than the Paulownia elongata briquettes, but also considering the density differences bulk sawdust.

The shear strength of the pellets, although not standardized, is present in the specialized literature as a methodology and results (Plištil et al. 2005, Spîrchez and Lunguleasa, 2016). The values obtained in the research are higher for Paulownia tomentosa (2.14N/mm^2) compared to Paulownia elongata, the increase being 43%.
A first conclusion of this research shows that the two types of woody species (*Paulownia tomentosa* and *Paulownia Elongata*) in the study are soft species, with very fast growth, whose woody biomass should be taken into consideration for the production of briquettes or pellets.

4.3. Results concerning the use of wheat straw for the manufacture of briquettes

The comparison between T1 wheat straw briquettes (59 mm diameter hollow) and beech briquettes was considered necessary because, although different in species, having the same moisture content and close effective density, there were differences in mechanical, calorific properties and content resulted of ash.
The huge amount of plant biomass left over from wheat harvesting made it possible for new technologies to make better use of this lignocellulosic resource. The present research aimed to evaluate the physical-mechanical and calorific properties of two types of briquettes made from wheat straw.

Three types of wheat straw briquettes, one with hollows and two without hollows, were taken from a manufacturing company using crank-rod briquetting facilities.

All three types of briquettes were foiled, with a length of 350 mm, the hollow ones having two different average diameters of 59 and 73 mm, and the hollow ones with an outer diameter of 71 mm and an inner diameter of 26 mm.
The modeling done wanted to find the maximum height of the stack on a pallet, such that the briquettes at the base of the stack would not break, based on the average effective strength obtained in the research.

The maximum number of rows in a stack depended to the greatest extent on the average compressive strength, from this point of view the specimens without voids T1 having the maximum number of rows of 23 pieces and the group of specimens T3 presented the lowest number of rows of 17 pieces.
The moisture content of the briquettes at the time of the density tests was 10%. The densities of the briquettes with void were differentiated, respectively 1237 kg/m^3 for briquettes with a diameter of 59 mm and 1169 kg/m^3 for those with a diameter of 73 mm.
The splitting strength had low values of 0.17-0.39 N/mm^2, 81-85% lower than the compressive strength, for all three types of briquettes analyzed.

Based on the data obtained with the calorimeter, the average values of the higher calorific value was 17260 kJ/kg and the lower was 17010 kJ/kg, for an average briquette moisture of 4%. Using some elements of linear geometry, the two relations of the upper and lower calorific power were found (PCI=17690-170·Mc; PCS=17690-107.5·Mc).
It is observed that, with the increase in the humidity of the briquettes, the calorific power will decrease.

As shown by other authors, Petrovici and Popa (1997), wheat straw has a lower carbon content than wood, respectively 47.9%. This translates into a lower calorific value.

T1 hollow briquettes had a calorific density of 21.882 MJ/m^3, T2 hollow briquettes had a calorific density of 20.679 MJ/m^3, and T3 hollow briquettes had a calorific density of 20.856 MJ/m^3.

The calcined ash content of straw briquettes average was 9.1%.

The variation range of calcined ash content values of wheat straw was from 8.89% to 9.33%.

In this way, the linear variation equation of those two parameters (PC=19.164 – 0.49·Ac) was found, based on which the calorific value of various plant species can be approximated, depending on the ash content.

A first conclusion refers to the general values of the properties of wheat straw briquettes. Experimental briquettes obtained from wheat straw are made with powerful installations, obtaining a high density.

A second conclusion regarding the 3 types of wheat straw briquettes is that they have almost similar characteristics such as high ash content (8.8-9.3%) and good calorific value (17.4-17.8 MJ/kg). The high ash content is due to the large amounts of secondary oxalates and carbonates, respectively 6-8% compared to 1-3% for solid wood or 2.4% similar to European wood species (Spîrchez et al.,2019).

4.4. Results relating to the torrefaction of beech pellets

Chosing beech wood for the comparison of torrefied and non-torrefied pellets was closely related to the fact that beech wood remains are abundant, being the most widespread wood species in Romania. The purpose of this research was to make a comparison between the torrefied pellets and the classic ones, in order to find the optimal treatment solution, as well as their judicious and efficient use. The beech pellets taken from the market were roasted at temperatures of 160, 180,200 and 220^{0}C, with periods of 1, 2 and 3 hours. After heat treatment, the pellets were investigated for mass loss, calorific value, ash content and shear strength compared to non-torrefied pellets.

It can be seen from Figure 4.28 that, regardless of the type of pellets and the heat treatment temperature, with the increase in the treatment time, the mass loss will increase, usually following a linear variation.

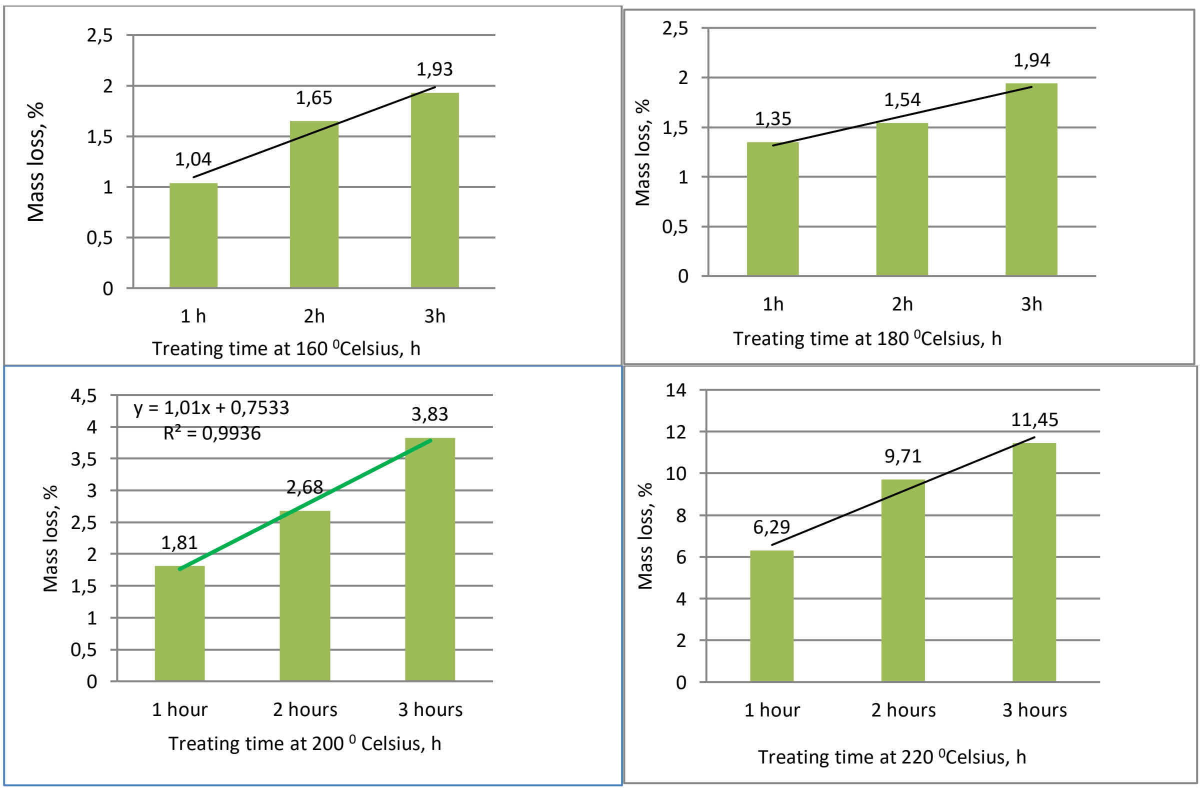


Figure 4.28. Influence of treatment time on mass loss during heat treatment

The shear strength of the torrefified pellets decreased with increasing torrefaction (increasing temperature and/or torrefaction time), as shown in Figure 4.29.

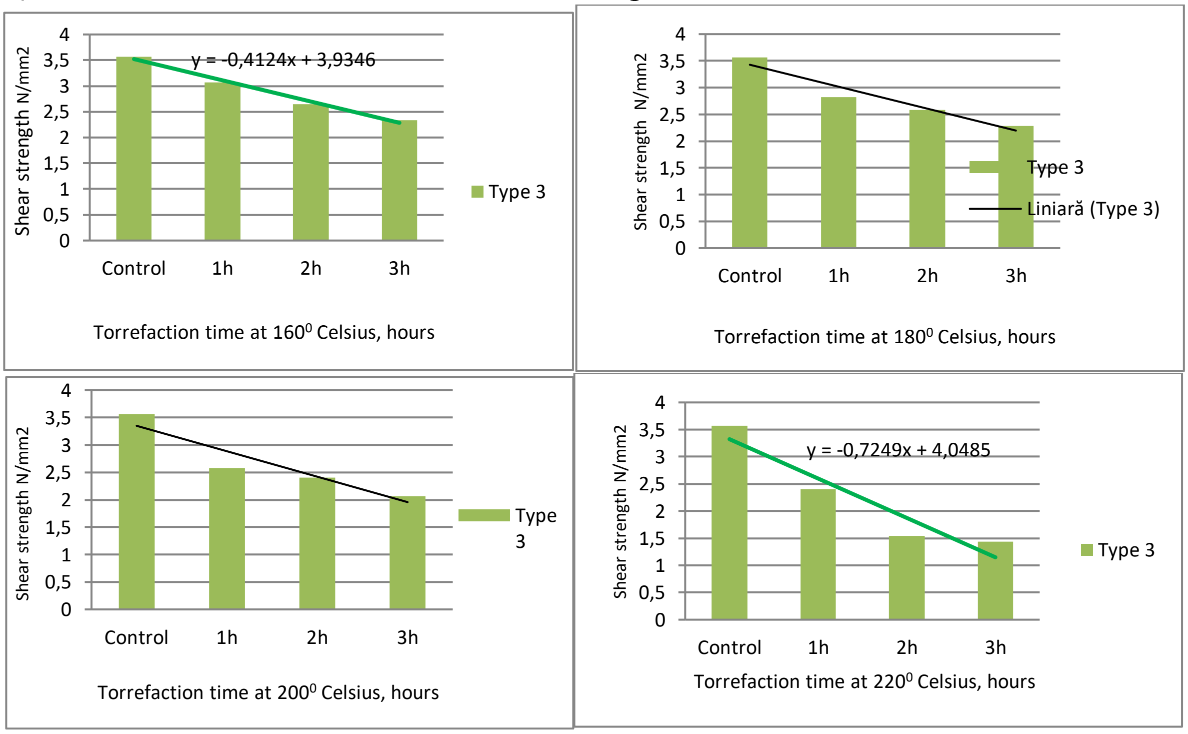
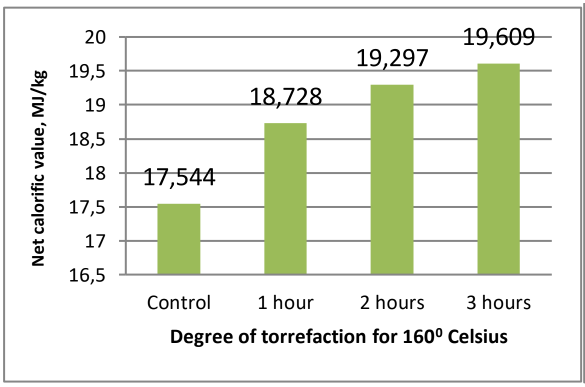
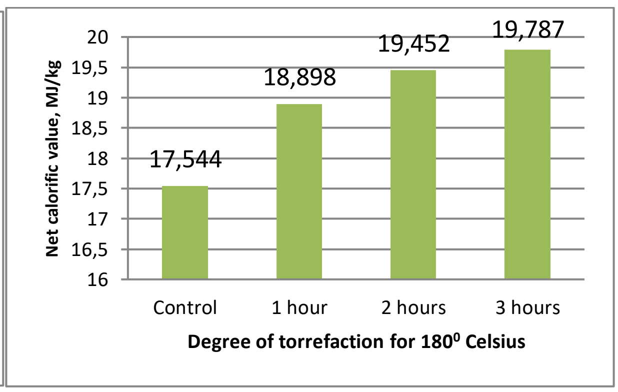


Figure 4.29. Shear strength of torrefied pellets

The lower calorific value of the pellets increased slightly from control samples to those treated with maximum regime.





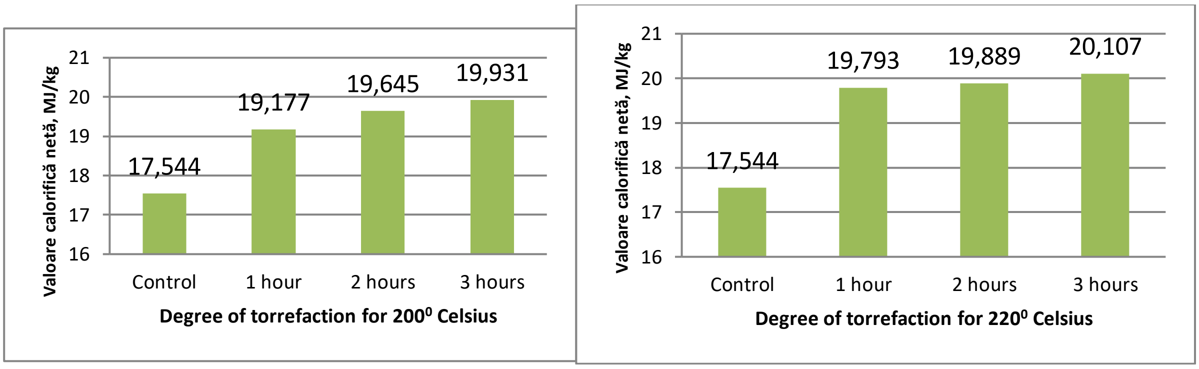


Figure 4.30. The influence of the degree of torrefaction on the lower calorific value of the pellets

The calorific density will increase in the first part up to the torrefaction point 180/3, after which it decreases towards the end according to the Gaussian curve.

The present research comes to clarify some aspects regarding some superior characteristics of torrefied pellets compared to non-torrefied pellets, especially the calorific power, for which an increase of up to 22% is achieved in the case of the type of pellets analyzed. One of the negative influences of the torrefaction process is also highlighted, namely the reduction of the shear strength of the torrefied pellets, obtaining values of up to 48%.

4.5. Results concerning the production of pellets from eucalyptus sawdust (Eucalyptus cinerea)

Eucalyptus (Eualyptus cinerea) is a plant from Myrtaceae family, as tree or shrubs with green leaves all year round. It grows in the African area, being considered an exotic species for Romania, having a reddish color and a very resistant wood.

The comparison between eucalyptus pellets and pellets from native species was made to observe the changes in the properties of the pellets obtained between exotic species and those from native species, Also, the lack of consistent research on the production of compact combustion products of this species was an important reason for the study.

All tested physico-calorific characteristics of pellets made from eucalyptus sawdust met the limiting conditions of the standard, both in terms of density, lower calorific value and ash content.

If we compare eucalyptus pellets with other combustible materials, we can see that eucalyptus pellets have a very good calorific value, its value of 20,863 kJ/kg being among the highest known along with that of willow (Grîu, T., Lunguleasa A.,2013).

4.6. Results related to the production of pellets from cardboard and mixture 1:1 cardboard with oak

Pellets of cardboard and cardboard-oak mixture with a diameter of 6 mm were obtained on a pelletizing machine. The purpose of the research was to observe what are the characteristics of pellets made from cardboard and if they improve significantly after adding sawdust and oak shavings to the composition.

All the physical-calorific characteristics of the pellets are superior to the limits imposed by the Austrian standard, regardless of whether the cardboard scraps were used separately to obtain the pellets or in combination with oak sawdust.
Also, the introduction of oak sawdust into the composition of the pellets was beneficial due to exceeding by more than 55% the density provided by the standard taken into consideration. The main conclusion of this research is that cardboard remains can be used separately or in combination with the biomass of other woody species to produce pellets with superior physical-calorific properties. A good compatibility between cardboard waste and oak sawdust was also observed.

4.7. Results concerning the production of briquettes and pellets from energy willow (Salix viminalis)

The energetic willow (Salix viminalis) is a plant from Salicaceae family, with fast growth (approx. 3 - 3.5 cm/day).

The biomass obtained when harvesting the energy willow can be stored in the form of briquettes and pellets (Grîu T., ş.a2014). Going further, other authors such as Chen et al.2012, improved the energy performance of biomass through torrefaction.

The bulk densities of 480.2 kg/m^3 for oak and 209.9 kg/m^3 for energy willow was obtained and a material loosening coefficient of 1.41 for oak and 1.97 for energy willow.

The density of briquettes differed between those obtained from energy willow (766.7 kg/m^3) and oak (877.8 kg/m^3). These differences were due to the differences in the density of the two wood species.

Regarding the bulk density of the sawdust, values of 480.2 kg/m^3 were obtained for fine oak material and 289.9 kg/m^3 for energy willow.

The main parameter of pellet torrefaction was mass loss, which increased significantly with increasing temperature. Therefore, maximum mass loss values were obtained for temperatures of 220 ^{0}C, between 16.78-23.47% for energetic willow and between 20.77-27.47% for oak.

It is observed that the oak roasts much better than the energetic willow, the losses being somewhat higher, this depending on the chemical composition of the two analyzed species.

It is observed that the energy willow has a higher calorific value than the oak, regardless of the treatment applied, the increase of the energy willow compared to the power of the oak for the maximum treatment applied being 141 kJ/kg or only 0.6%. After the torrefaction process, the calorific value of the energetic willow increased by 3.5%, and that of the oak by 3.4%.

The compressive strength of the two types of briquettes was very different, willow briquettes having a compressive strength of 1.02 N/mm^2 and the oak briquettes having a compressive strength of only 0.33 N/mm^2. This difference highlights the fact that oak briquettes will break much faster during storage in bunk bags.
The splitting perpendicular to the length of the briquette had small values, about 0.08 N/mm^2 in the case of oak and 0.05 N/mm^2 in the case of energy willow.

Regarding the splitting of the briquettes in the direction parallel to the length of the specimen, low average values were obtained, respectively of 1 N/mm^2 in the case of oak briquettes and about 0.85 N/mm^2 in the case of energy willow.

The average pellet shear values were 0.74 N/mm2 for willow pellets and 0.86 N/mm^2 for oak pellets.
Willow briquettes had 1.92% abrasion and oak briquettes had 4.22% abrasion.

Calcined ash kept the values of woody species, respectively they fell below 1%. Indeed, the calcined ash content of energy willow was 12% lower than that of oak.
As a first conclusion, the energetic willow has a good calorific behavior, its calorific properties being identical to those of the white willow (Salix alba), i.e. a calorific power among the highest of the woody species. Also, compared to oak, energy willow has superior combustion properties.

4.8. Conclusions on research results

The briquettes obtained from the husks of sunflower seeds are energy efficient, having calorific values close to briquettes made of larch sawdust, becoming a viable alternative to wood briquettes.

Paulownia tomentosa and Paulownia elongata can be considered for obtaining briquettes and pellets, thanks to the characteristics that allow them to produce finished products that fall within the limits of the specialty standards in terms of compressibility and compactability.

The property values of wheat straw briquettes are almost similar to those of wood briquettes.

The torrefaction of beech pellets led to the clarification of the positive aspects related to the superior characteristics of these products, more precisely the proof of the increase in calorific value from 17.544 to 20.107 MJ/kg.

The negative influence of the torrefaction process results in the reduction of shear strength by up to 48%, at a temperature of 220^{0}C for 3 hours.

By comparing the pellets obtained from eucalyptus with those obtained from other combustible materials, it was observed that eucalyptus pellets also have a very good calorific value, of 20863 kJ/kg.

To obtain pellets with superior physical-calorific properties, cardboard scraps can be used in recipes with a unique composition, only cardboard.

The energetic willow has a higher calorific value than oak by about 6%, regardless of the applied treatment. Following the torrefaction process, the calorific value of energy willow increased by 3.5%, and in the case of oak, the increase was 3.4%. A certain loss of mass occurs through torrefaction. Oak briquettes will break more quickly when stored in overlapping bags. The compressive strength of the two types of briquettes was consistently different, 1.02N/mm^2 for willow briquettes is recorded, and 0.33N/mm^2 for oak briquettes. Regarding the splitting strength perpendicular to the length of the briquette, the values obtained in the case of willow briquettes were slightly lower than in the case of oak briquettes (0.05 N/mm^2 versus 0.08 N/mm^2), and in the case of for splitting parallel to the length, the recorded values are 0.85 N/mm^2 for willow compared to 1 N/mm^2 for oak. The difference between the densities of the 2 species also leaves its mark on the abrasion of the briquettes obtained 1.92% for willow and 4.22% for oak.

The energetic willow has a good calorific behavior, the calorific properties being identical to those of the white willow (Salix alba).

The original contributions of the work resulting from the experimental study relate to briquette abrasion, compressive and splitting strength, as well as pellet shear tests.

Other original contributions refer to the modeling of the influence of carbon content, ash content and lignin content on the calorific value of briquettes and pellets, but also the use of atypical biomasses, such as eucalyptus or paulownia species.

CHAPTER 5. QUALITY OF LIGNOCELLULOSIC BRIQUETTES AND PELLETS

5.1. Theoretical research

The quality of briquettes and pellets is very difficult to define, and quality experts around the world have had different, often contradictory, opinions. The oldest definition is that of the Romanian calitologist Juran, who says that "a product is of quality if it is suitable for use".

5.1.1. Assessment of the quality of briquettes and pellets on the basis of their relative position in relation to the limiting value of the main characteristics

This method measures the quality of briquettes and pellets by relating the actual value of a characteristic to the limiting value expressed by the standard in the field. The foundation of this method is based on extracting the main characteristics of briquettes and pellets, finding the limiting values of each of them and making a percentage comparison between the two elements.

5.1.2. Statistical assessment of the quality of batches of briquettes and pellets on the basis of measurable characteristics [1]

The statistical verification of the batches of briquettes and pellets is a sampling verification, when a small amount randomly extracted from the batch is checked, and the result obtained on the sample is extrapolated to the level of the entire batch. The main advantages of statistical control are given by the reduced checking of products, but especially by the drastic nature of the rejection of a batch of products, when the top management will have to take urgent measures to remedy the production that generated these defective products.

A measurable characteristic is a characteristic that has a unit of measurement. The quality check of the batch of briquettes and pellets is done from the point of view of each measurable characteristic analyzed, which is why an analysis must be performed to identify the one or the most important quality characteristics of the analysis batch.

[1] A. Lunguleasa (2013), Sequential technologies for making and testing wood composites, Lecture notes

The product quality control plan for measurable characteristics (n and k) is determined according to a methodology that includes, as follows:
- choosing the elements on the basis of which the entry into the quality standard is made: the lot, the level of verification, the level of acceptable quality, sampling as a typology and the type of verification method;
- the extraction from the quality standard of the quality control plan, respectively the choice of sample n and k, which represents the acceptance constant.

The establishment of a certain quality control method (s-method, R-method, and σ-method) is based on several considerations, such as economic ones, and the consistency of the information obtained.

The determination of the level of verification is carried out according to the value of the level of consistency of the information, in this sense for the measurable quality characteristics there are three usual values noted as I, II, III and two other values of the levels, respectively the special ones noted as S_3 and S_4.

5.1.3. Method s for statistical evaluation of batch quality of products

Finding and operating a product quality control plan using the *s* method involves several essential operations, among which we mention:
- with the help of Nv and N, a certain code letter can be determined, denoted by LC;
- by means of the code letter and the value of the acceptable quality level, n and k can be chosen, that means the level of the sample and that of the acceptance constant;
- the number of products of the sample is randomly chosen from the entire batch of products;
- the value of k from the standard is determined, respectively the quality characteristic is chosen;
- the two statistical parameters of the sample are calculated, namely the arithmetic mean and the standard deviation of the measured values of the sample;
- based on the values determined above, namely the average, the standard deviation and the quality parameter denoted by the letter q, the quality of the product batches is determined, respectively if the checked batches are accepted or rejected.

The decision to accept or reject the batches of verified products is the comparison between the product quality parameter q and the acceptance constant k. The so-called double separated tolerance limits are defined by the simultaneous existence of two limits, namely the upper and the lower, both having a separate acceptable quality level for each limit. Separate tolerance limits refer to individualized tolerance limits, either upper or lower.

5.1.4. R method for assessing the quality of batches of products

This product batch quality method allows a decision to accept or reject a batch of checked products based on the Rm estimator, which is called the mean range of values. For this, the sample extracted from the batch is divided into sub-samples of 5 elements each, for each of these sub-samples the own amplitude is determined, as the difference between the maximum and the minimum value.

The range of values of a subsample denoted by R is a difference between the highest value and the lowest value of the subsample. The average amplitude is determined as the arithmetic mean of the amplitudes calculated for each sub-sample, and represents the main parameter that is taken into account in assessing the acceptance or rejection of lots within all subgroups of the analyzed value string.

5.1.5. Checking the quality of batches of briquettes and pellets with gauges within tight limits by the Pre-control method

This method drives an operational process within specification and identifies changes that could disrupt the operational process and make out-of-spec parts possible.

The Pre-Control method is based on the assumption of a worse condition, on the basis of which a batch of products of a manufacturing flow that would produce quality products can be accepted or rejected. In this way, the conditions of the change in the manufacturing flow and the change in the quality of the products can be identified.

Based on the theoretical considerations set out above, a coherent set of rules was established, which summarizes the technique of applying the Pre-Control method:

- the statistical distribution diagram is made, highlighting the extreme limits of tolerance;
- the tolerance band is divided, by drawing the two PC lines at ¼ and ¾ of the tolerance interval (PC1 and PC2);
- the production activity starts within the operational process;
- the first piece is checked, and if the characteristic value is outside the specification limits, the operational process is adjusted;
- if one piece out of three is within specification but outside a PC line, the next part is checked;
- if the second piece is outside the same PC line, the technological process is corrected by centering the tolerances, adjusting the machines, etc.;

- if the second piece is inside the PC limits, the process continues, intervening for corrections only if two out of three pieces are outside a determined PC line;
- if of two successive pieces, one is outside a PC line, and the next part is outside the other PC line, then the tolerance interval must be reduced immediately by technological methods.

The Pre-Control method highlights the changes that occur in the trend and variation of an operational process based on the verification of only three parts. This process is able to guarantee a lower percentage of defects if corrections are made whenever necessary.

5.2. Experimental research

5.2.1. Assessment of the quality of Paulownia elongata and Paulownia tomentosa sawdust pellets, based on the relative position in relation to the limiting value of the main characteristics

Two batches of pellets are chosen, from Paulownia tomentosa sawdust and Paulownia elongata, which will be analyzed from the point of view of the quality of the batches of products. A sample of 30 pellets denoted T1 and T2 was drawn at random from each batch.

Pellet quality analysis can be done in the first instance according to each individual characteristic. Taking all of this into account, we can say that type 2 pellets are just as good as type 1 pellets, and a comparison decision cannot be made.

Another method of appreciation is the method of cumulating the points obtained for each characteristic by each type of pellets. By creating the score for each characteristic separately and adding these points together, a superior value of 110.19 points will be found for type 2 pellets.

Table 5.2. Appreciation of pellet quality against the limiting values of the analyzed characteristics

No.	Quality characteristic	Limit value ÖNORM M 7135 și EN 14961-2	Actual value		Relative position %,		Value points out of 100	
			T1*	T2**	Type 1	Type 2	Type 1	Type 2
1.	Medium pellet length, mm	Max 5*D = 30	22	28	-26,6	-6,6	-26,6	-6,6
2.	Bulk density, kg/m^3	Minimum 300	326	325	+8,6	+8,3	+8,6	+8,3
3.	Density, kg/m^3	Minimum 11200	1130	1330	+0,008	+18,5	+0,008	+18,5
4.	Calorific value, MJ/kg	Minimum 18	18,26	18,53	+1,44	+2,94	+1,44	+2,94

5.	Shear strength, N/mm²	Minimum 1,2	1,30	2,23	+8,3	+85,8	+8,3	+85,8
6.	Ash content, %	Maximum 0.8	0,82	0,81	+2,5	+1,25	+2,5	+1,25
Total	-	-	-	-	-	-	-5,75	+110,19

* T1: Pellets paulownia elongata; **T2: Paulownia tomentosa pellets

5.2.2. Assessment of the quality of lignocellulosic wheat straw briquettes on the basis of their relative position in relation to the limiting value of the main characteristics

Two batches of wheat straw briquettes are taken in the form of polyethylene bags filled with briquettes, some of them without gaps (T1) and the others with internal voids (T2).

Table 5.3. Appreciation of the quality of wheat straw briquettes with and without gaps against the values of the limiting characteristics of the standard

No.	Quality characteristic	Limit value DIN 51731	Actual value		Relative position %,		Value points out of 100	
			Type T1*	Type T2**	Type 1	Type 2	Type 1	Type 2
1.	Humidity, %	Maximum 12	10,4	10,1	-13,3	-15,8	-13,3	-15,8
2.	Unit density, g/cm³	1-1,4	1,23	1,17	23	17	+23	+17
3.	Compressive strength, N/mm²	Minimum 1,4	2,1	1,1	50	-21,4	+50	-21,4
4.	Splitting strength, N/mm²	Minimum 0,25	0,22	0,39	-12	56	-12	+56
Total							+47,7	+35,8

T1*-briquettes of wheat straw, without gaps; T2**-briquettes made of wheat straw, with voids.

A first comparison that is made is the one related to the number of positive and negative characteristics attributed to each type of briquettes, respectively how many characteristics in total correspond to the limiting conditions of the standard.

A second comparison is made regarding the analysis of each characteristic separately for each type of briquettes. On total characteristics and in this analysis it is not possible to distinguish a type of briquettes better in quality.

A third comparison given by this method is the comparison of the algebraic sum of the scores for each type of briquettes. From this point of view, the higher score of T1 type briquettes makes it possible to choose it as the best quality batch.

5.2.3. Method s, case of lower tolerance limit for energy willow pellets

This method refers to the use of the standard deviation *s*, applied to the analysis of the density of energetic willow (Salix viminalis) pellets, by using the two variants, respectively the numerical and graphic method. A batch of 1200 pieces of energy willow pellets will be is subject to statistical verification, in order to accept or reject it. According to the statistical control standards for measurable characteristics according to the level of the lot 501-1200 and the usual verification level II, the verification plan index letter J is found. By adopting an acceptable quality level AQL= 4 and normal verification, the two elements of the verification plan are found on the table: the lot level n=35 pcs and the acceptance constant k= 1.39. So the sample of 35 pellets is taken from the lot and the density is checked.

Table 5.5. The numerical decision procedure for the application corresponding to method s

No.	Date	Valori
1	Sample size (from statistical standards)	35
2.	Sample average (calculated)	1148,1 kg/m^3
3.	Mean square deviation of the sample (from calculation), s	18,2 kg/m^3
4.	Lower tolerance limit (from pellet standards)	1120 kg/m^3
5.	Acceptance constant (from the statistical standard), k	1,39
6.	Quality parameter, q	1,54
7.	Decision: q=1.54 > k=1.39. The lot is accepted.	

5.2.4. Method R, lower tolerance limit case, energy willow pellet density

For the use of the R method (range of values), as in the previous case, it is considered the case of the lower limit of tolerance, at a density value of 1120 kg/m^3. A batch of 1200 pieces of energetic willow (Salix viminalis) pellets from the case of the previous application, will be analyzed with the average amplitude R method.

At code letter I, a sample of 30 pellets is found, and at an acceptable quality level AQL=2.5 the acceptance constant k=0.654 is found.

We divide the 30 sample values into 6 subgroups of 5 pellets. The amplitude of each subgroup is then determined, as a difference between the maximum and minimum value, after which these amplitudes are averaged.

Table 5.7. Data of the numerical procedure of the R method

No.	Specifications	Values
1.	Sample size	30
2.	The number of subgroups	7
3.	The sample mean	1148
4.	Average sample amplitude	37,1
5.	Lower tolerance limit	1120
6.	The quality parameter, q	$q = \frac{(\bar{x}-L_i)}{\bar{R}}$= 28/37,1=0,75
7.	Acceptance constant, k	0,654
Decision	q= 0,75 > k=0,654. Batch is accepted	

5.2.5. Upper tolerance case, method s, ash content of briquettes obtained from larch sawdust

A batch of 600 larch briquettes will be analyzed from the point of view of measurable characteristic, namely ash content.

Table 5.9. Numerical decision process for method s and ash content of larch briquettes

No.	General data	Values
1	Sample size (from statistical standards)	35
2	Arithmetic sample mean (from calculation)	0,63
3	Sample mean square deviation (from calculations)	0,04
4	Upper tolerance limit (from pellet standards), Ls	0,70
5	Acceptance constant (from the statistical standard), k	1,39
6	Quality parameter (from calculation, relation 5.1) $q = \frac{(\bar{Ls}-m)}{s}$	1,75
7	Decision: q=1.75 > k=1.39. The lot is accepted.	

5.2.6. Case of double separate tolerance limits, method R, calorific value of eucalyptus pellets (analysed in Chapter 4.5)

For a batch of 250 pieces of eucalyptus pellets we choose a usual check level II, at the lot level of 151-280 and the code letter G is found. Going further in the statistical quality standard method R, normal check, the acceptable quality level AQL=1 is chosen for the lower limit, where the duplicate of the verification plan is found (sample n=15; lower acceptance constant Ki=0.738), and for the upper limit AQL=4 is chosen, with an acceptance constant of Ks=0.536. At this point it is possible to proceed to the actual verification of the sample of 15 pellets extracted from the batch, by simple sampling.

Average amplitude, Rm=0.51
The arithmetic mean of the values, m=19.48
Lower limit Li=17.5; Upper limit Ls=19.9
Acceptance constants from the standard: ki=0.738; ks=0.536
The quality parameter for the lower limit, Qi=(m-Li)/Rm ; Qi=3.88
The quality parameter for the upper limit, Qs=(Ls-m)/Rm ; Qs=0.82
Decision: Qi >ki, Qs>ks. The lot is accepted.

5.2.7. Case of double separate tolerance limits, method s, moisture of oak briquettes

At the code letter J is found the effective sample of 35 briquettes that can be checked from the point of view of the measurable characteristic – the moisture of the briquettes. The AQL level of 0.1% is adopted for the lower limit value and also 1.5% value for the upper limit, in order to use the method of double and separated tolerance limits. From the SR ISO 3951-5:2009 standard, the two acceptance constants ks=1.57 and ki=2.54 are found. At this moment there is all the data to proceed to the procedure of checking the quality of the batch of briquettes, extracting the sample of 35 pieces and checking the moisture with an electrical device.

Table 5.12. Numerical method data for briquette moisture and method s

No.	Statistical parameters	Values
1.	The sample taken from the lot	35
2.	Average of the sample values taken from the lot	10,2
3.	Standard deviation of the sample taken from the lot	0,8
4.	Upper tolerance limit value	12
5.	Lower tolerance limit value	8
6.	The value of the upper acceptance constant, Ks	1,57
7.	Lower acceptance constant, Ki	2,54
8.	The quality parameter for the upper limit, qs	2,0
9.	The quality parameter for the lower bound, qi	2,75
Analysis: qi =2.75 > ki=2.54. Qs =2.0 > ks=1.57 Decision: The lot is accepted.		

5.2.8. Case of dual combined tolerance limits, method R, bulk density of wheat straw lignocellulosic briquettes

We analyze a batch of briquettes made from wheat straw, 350 pieces, from the point of view of bulk density. The technical elements of the product batch delivery are: AQL= 4 %, both for the upper and lower tolerance limit and the usual verification level Nv=II. Checking in the standard STAS 3160/3-84 it is determined that the actual sample requests 25 pieces and the number of groups of this sample of 5 briquettes, which means that a group consists of 5 briquettes. We proceed to the actual verification of the sample divided into 5 groups and obtain the values. The amplitude of each group was determined as the difference between the group's maximum and minimum value.

- lot value 350 pieces;
- the effective value of the sample 25 pieces;
- the average of the sample extracted from the lot 674 kg/m3;
- average standard amplitude of the sample 83;
- upper tolerance limit 720 kg/m3;
- lower tolerance limit 620 kg/m3;
- normalized average: (m-Li)/(Ls-Li)= 0.54;
- average normalized amplitude: R/(Ls-Li)= 0.83;
- the value of the coefficient F=0.707, extracted from the quality standard;
- the value of the parameter MAR=F(Ls-Li)=70.7, extracted from the quality standard.

The curve corresponding to the initial data is chosen from the standards, but also the immediately following lower curve, because if the current point, P(Rm, Xm), is between the two curves, the acceptability of the batch is doubtful and the procedure is repeated for another sample from the batch of briquettes . The current point K (Rm/(Ls-Li); (m-Li)/(Ls-Li)) is placed on this curve, means the real point K(0.83;0.54). The value of the normalized mean and the value of the normalized mean amplitude generate the current point that is located on the characteristic curve chosen accordingly from the standard, by the intersection of the value of the abscissa and the corresponding ordinate.

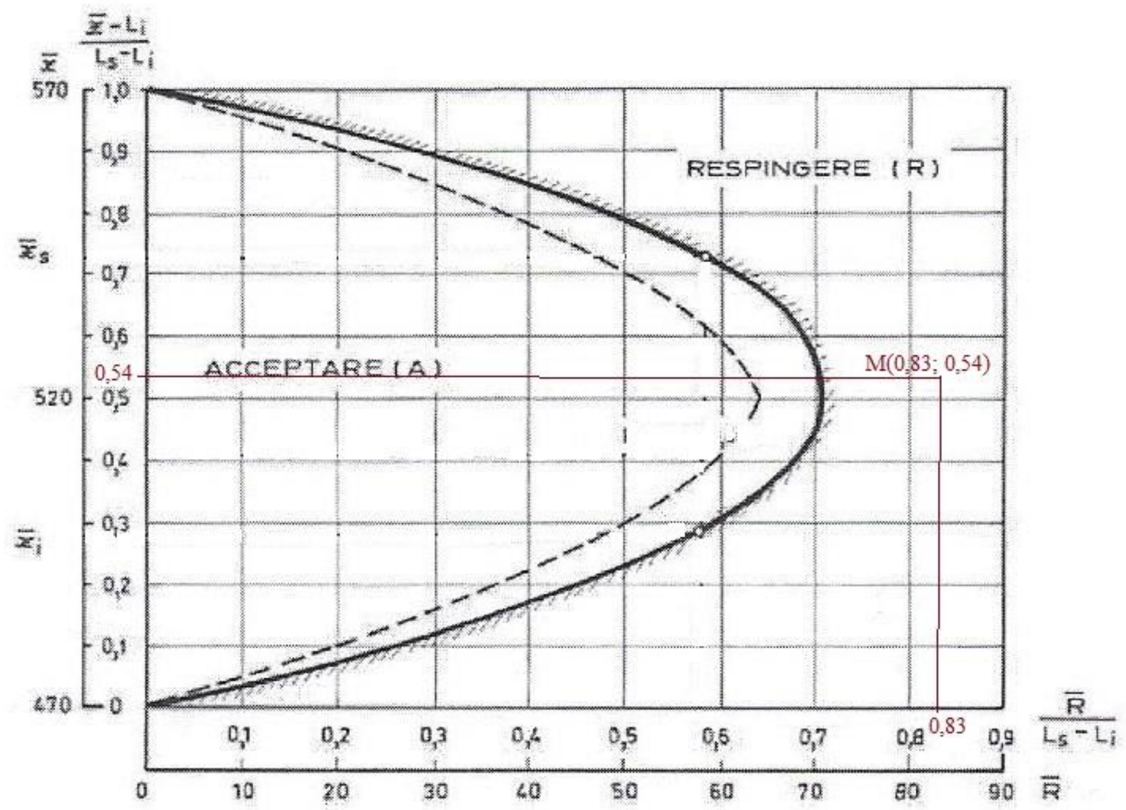


Figure 5.7. Graphical procedure for application 5.2.8

The previously determined current point K(0.83;0.54) is located outside the characteristic curve, respectively in the rejection zone, so the batch is rejected. Even if initially it seemed that the batch would be accepted due to the values falling within the tolerance limits, it is observed that the statistical analysis disproves this impression. This fact could have been observed if the comparison between the mean amplitude of the values of 83 and the value of the parameter MAR=70.7 had been initially made, the rejection of the lot being once again confirmed.

5.2.9. Pre-Control method for manufacturer's quality check for oak pellets

The tight gauge method is applied to the production of briquettes and pellets, to check their quality, respectivly if the briquettes or pellets are within the standardized limits. For example, we chose SC Saras SRL from Prejmer, Brașov, Romania, which produced oak pellets on a 400 kg/h press. The first stage is that of creating the statistical diagram based on a set of 100 values of the pellet densities obtained on the respective installation which were divided into 14 classes.

Based on the classes and the absolute (relative) frequencies, the frequency graph was created.

The 100 values of the measured densities were statistically processed obtaining an average of m=1.251 g/cm^3 and a standard deviation sd=0.047 g/cm^3. For a confidence interval of 99.73%, that is, for an interval of m±3s=1.251±3·0.047, the tolerance limits of the interval Li=1.11 g/cm3 and Ls=1.392 g/cm^3 are obtained. The range tolerance is thus found as the difference between the two limit values T=1.392-1.11=0.282 g/cm^3. This tolerance is divided by 4 and this value is added to the lower limit, which results in the value of the first Pre-control line PC1=Li+1/4T= 1.1805 g/cm^3, and by subtracting this value from the upper limit, the second is obtained line Pre-control PC2=Ls-1/4T=1.2687 g/cm^3. At this point we have all the data to draw the Pre-control chart.

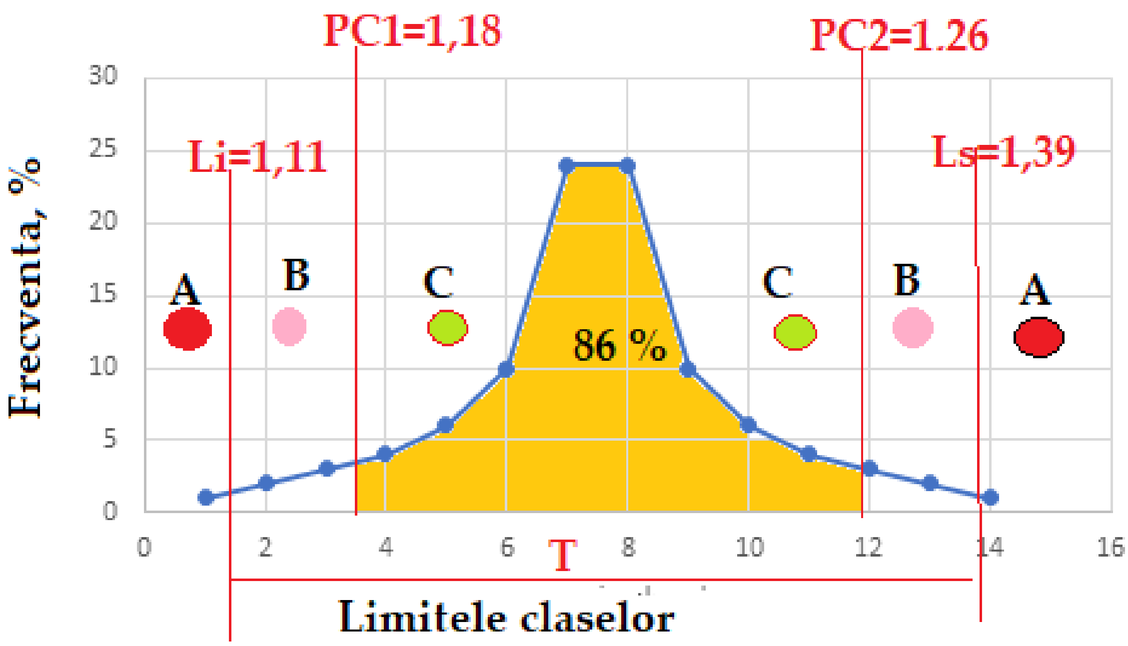


Figure 5.9. Pre-control diagram of quality check of pellets with tight gauges

The operation of the pelletizing press is started and the pellet quality analysis will be done in terms of unit density of the pellets. The main principle of using the Pre-control chart is to find at least 2 consecutive values out of the 3 measured values within the Pre-control limits.

The following 3 values were obtained at SC SARAS SRL: 1.31 g/cm^3, 1.23 g/cm^3 and 1.24 g/cm^3. The technological process could continue because the last two values were found within the two Pre-control limits.

5.3. Conclusions on the quality of lignocellulosic briquettes and pellets

The quality of products obtained from vegetable and woody biomass is difficult to define, but it is very important from the point of view of both the producer and the consumer of finished products of this origin.

Through quality control based on statistical-mathematical methods, quality products can be obtained that meet the needs of use.

Within the method of assessing the quality of briquettes and pellets based on the relative position to the limiting value of the main characteristics, the comparison of Paulownia elongata and tomentosa sawdust pellets was made, as well as the comparison of wheat straw briquettes with and without voids.

For the quality analysis of energy willow (*Salix vimimalis*) pellets, the S method and the R method were applied for the density analysis, imposing the case of the lower tolerance limit for verification, from which it was concluded that the whole batch is accepted due to the fact that the conditions of acceptance.

For the analysis of larch (*Larix decidua*) briquettes, the ash content was analyzed, the case of the upper limit of tolerance, by applying method S, from which it resulted that the lot is accepted.

For the analysis of eucalyptus pellets (*Eucalyptus cinerea*), calorific value analysis, the case of double limits separated by tolerance, the R method was applied, from which it resulted that the lot is accepted.

For the analysis of oak (*Quercus robur L*) briquettes, method S was applied, the case of double separated tolerance limits, from which it resulted that the lot is accepted because the quality parameters calculated for the 2 tolerance limits are higher than the 2 corresponding constants.

For the analysis of the bulk density of wheat straw briquettes, the K method was applied, the case of the double combined tolerance limits, and following the measurements and the application of the formulas, it was found that the result does not fall within the accepted limits and the batch is rejected.

To analyze the quality of oak pellets, the Pre-control method was used, to check the quality at the manufacturer.

6. CONCLUDING REMARKS. OWN CONTRIBUTIONS. CAPITALIZATION OF RESULTS. FUTURE RESEARCH DIRECTIONS

Technological progress in the field is advanced, but with an impediment: high efficiency and productivity technologies are limited due to the production costs involved and the necessary investments.

Combining several raw materials from different woody and plant species, as long as some verified recipes are used, is cumbersome and caused by the incompatibility of some of them.

Corroborating the conclusions of each chapter, as well as by intercorrelating the various solved problems, the main conclusions of the research will be presented.

6.1. Concluding remarks

Through the method of making samples from woody and vegetable biomass, it was aimed to obtain samples through simple cutting methods, which would require a minimum consumption of energy and resources.

For each material used, the granulometry was determined, by using the vibrating device with several sorting screens.

In the case of lignocellulosic briquettes, the resistance to breaking by compression, respectively the resistance to splitting, was determined.

Shear strength was determined for lignocellulosic pellets, as it is considered as the main mechanical property.

Various results validation methods were used to quantify quality.

6.2. Economic and valorisation aspects

The example of capitalizing on experimental research is done for a house with a useful surface of 200 m^2. Considering the calorific value of native pellets of 19.1 MJ/kg and 19.9 MJ/kg for torrefied pellets, an annual demand of 34.2 kg/m^2 for native pellets and 32.8 kg/m^2 in the case of torrefied pellets. If we take into account the initial prices of the pellets, the initial investment, the maintenance of the equipment, the following costs for heating the house will be obtained: 19,700 Euro/year in the case of native pellets and 19,650 Euro/year in the case of torrefied pellets. A net advantage is observed in the case of torrefied pellets due to the increase in calorific value, by outranking the additional expenses of torrefaction.

6.3. Own contributions

Regarding the purpose and objectives of the thesis, to research and propose solutions for the realization, testing and qualitative evaluation of briquettes and pellets, the fulfillment of each proposed objective was pursued, as follows.

In the case of the first objective, briquettes and pellets were made and analyzed using today's important forest and agricultural species, such as larch, paulownia, eucalyptus, energetic willow, husks of the village flower, wheat straw, cardboard scraps, oak.

Regarding the second objective, regarding the testing of the main physical, chemical, calorific and mechanical properties of the briquettes and pellets, a series of experimental activities was performed, testing the moisture content, determining the effective density of the briquettes and pellets and to determine the calorific.The ash content, which is an important quality characteristic, has been determined, with different values depending on the raw material used.

Regarding the strengths of lignocellulosic briquettes and pellets, the values recorded for lignocellulosic briquettes denote consistency and compaction, which had different values depending on the raw material and wood species.

Regarding the abrasion of the briquettes, the value of 1.92% was recorded for the energetic willow, and an abrasion resistance of 4.22% in the case of the oak briquettes. The lower value from the energy willow was due to the lower density, but also the composition and structure of the wood material.

With regard to objective 3, to find cumulative methods for checking the quality of briquettes and pellets, the elements that compete in the definition of quality were identified and a series of concrete examples of determining the quality of batches of briquettes and pellets were made.

The research performed within the doctoral program contains original contributions.

The original contributions of the work resulting from the experimental study relate to briquette abrasion, compressive and splitting strength, as well as pellet shear tests. Other original contributions refer to the modeling of the influence of carbon content, ash content and lignin content on the calorific value of briquettes and pellets, but also the use of atypical biomasses, such as eucalyptus or paulownia species.

The documentary research performed highlights physical, chemical and mechanical properties of biomass belonging to indigenous and exotic species.

The materials used are mainly secondary products and residues to which added value is added and are introduced into the economic circuit, especially in the energy field.

The assessment of the quality of briquettes and pellets was carried out using cumulative verification methods.

Data processing, evaluation of results and conclusions are carried out and presented in an original manner.

6.4. Valorization of the results

The results of the research made by the author during the preparation of the doctoral thesis materialized in the elaboration of 5 papers published in specialized journals

1. ***"The briquettes properties from seed sunflower husk and wood larch shavings"***

VERONICA DRĂGUȘANU (JAPALELA), AUREL LUNGULEASA, COSMIN SPÎRCHEZ (RECEIVED AUGUST 2020), WOOD RESEARCH, vol. 66, nr. 4, 2021, https://doi.org/10.37763/wr.1336-4561/66.4.689699, e-ISSN 2729-8906 66(4): 2021 689-699 pp.

2. **"*Analisys* of Briquettes and Pellets Obtained from two Types of Paulownia (*Paulownia Tomentosa* and *Paulownia Elongata*) Sawdust"**

Spirchez, C., Japalela, V., Lunguleasa, A., și Buduroi, D. (2021), *BioResources vol.* 16, nr. 3, 5083-5096, https://bioresources.cnr.ncsu.edu/issues/vol16-issue3/page/4/

3. **"*Evaluation* of the Physical, Mechanical, and Calorific Properties of Briquettes with or without a Hollow Made of Wheat (Triticum aestivum L.) Straw Waste"**,

Veronica Dragusanu, Aurel Lunguleasa and Cosmin Spirchez, 2022 **https://doi.org/10.3390/app122311936**

4. "*Statistical Appreciation of Pellets Quality for Measured Characteristics*"
Veronica DRAGUSANU (JAPALELA), Aurel LUNGULEASA, Cosmin SPIRCHEZ
RECENT J. (2023), 69:051-059 https://doi.org/10.31926/RECENT.2023.69.051 Pag 51-59
https://www.recentonline.ro/2023/069/Dragusanu-R69.pdf

5. *"Some Properties of Briquettes and Pellets Obtained from the Biomass of Energetic Willow (Salix viminalis L.) in Comparison with Those from Oak (Quercus robur) "*
Veronica Dragusanu (Japalela), Aurel Lunguleasa, Cosmin Spirchez and Cezar Scriba, Wood Processing and Design Wooden Product Department, Transilvania University Brașov
Journal *Forests* **2023**, *14 nr.* 6, art nr. 1134; https://doi.org/10.3390/f14061134, Mai 2023
https://www.mdpi.com/1999-4907/14/6/1134

6.5. Future research directions

Considering the fact that the analyzed topic has not been completely exhausted, but also because the dynamics of works in the field register an upward trend, it is proposed to continue the theoretical and experimental studies.

Further research may focus on the use of natural additives from sugar manufacture, in different proportions, to increase the degree of compaction and greater resistance to shear and compression.

Considering the results obtained from the research on hollow briquettes, I believe that it is necessary to follow this direction in the future, by expanding it to more species.

Regarding woody species, research can continue by looking at combining them in different proportions with each other, or with other plant biomass raw materials.

Another direction of interest can be the use of the remains from agricultural processing, which are most often burned uncontrolled on the plains or at the edge of the waters, but also of the remains resulting from the grooming of the trees.

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SHORT SUMMARY

CONTRIBUTIONS TO THE REALISATION, TESTING AND QUALITATIVE EVALUATION OF BRIQUETTES AND PELLETS FROM WOODY AND VEGETABLE BIOMASS

The thesis "*Contributions to the realization, testing and qualitative evaluation of briquettes and pellets from wood and plant biomass*" has as its main objective the investigation of the possibilities of realization, testing and qualitative evaluation of briquettes and pellets from wood and plant biomass.

The problem proposed in this work had 3 general objectives: making briquettes and pellets using woody and vegetable biomass, in different combinations, on different installations and presses in terms of operation mode and capacity, in order to observe the differentiation of properties according to these aspects; testing the main physical, chemical, calorific and mechanical properties of briquettes and pellets obtained in a laboratory or industrial setting; finding a cumulative method for determining the quality of briquettes and pellets, the elements that compete in defining quality have been identified, and a series of concrete examples of determining the quality of batches of briquettes and pellets have been made.

The experimental study refers to briquette abrasion, compressive and splitting strength, as well as pellet shear resistance. In the framework of the study, modeling was performed fallowing the influence of carbon content, ash content and lignin content on the calorific power of briquettes and pellets, as well as the use of atypical biomasses, such as eucalyptus or paulownia species.