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Use of spruce cones for energy purposes

Diploma Thesis

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DIPLOMA THESIS ASSIGNMENT

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Sustainable Rural Development in the Tropics and Subtropics

Thesis title

Use of spruce cones for energy purposes

Objectives of thesis

Analysis of suitability of using spruce cones in the form of densified solid biofuels.

Methodology

- Gathering and analysing of reference data from science databases for literature review writing.
- Grinding, briquetting and pelleting of spruce cones (production of densified biofuels).
- Determination of spruce cones/briquettes, pellets fuel properties (chemical, physical and mechanical) according to EN and ISO standards.
- Combustion of spruce cones, spruce briquettes and pellets and measuring of emissions.
- Comparison and assessment of energy utilization of spruce cones in loose form vs briquettes vs pellets.

The proposed extent of the thesis

60-65 pages

Keywords

spruce cone, waste biomass, briquetting, pelleting, solid biofuel, combustion

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Declaration

I hereby declare that this Diploma Thesis „Use of spruce cones for energy purposes“ was done by myself. It was written under the control of the supervisor and consultant of this work and used references are well cited.

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Abstract

The world's energy consumption is covered mostly by fossil fuels, which are exhausting rapidly. Therefore suitable alternative for fossil fuels is nowadays becoming more urgent.

The thesis contains of two parts. First part is theoretical part, which collects and analyses literature reviews mostly from scientific databases. The theoretical part gives information about renewable energy resources, biomass and solid biofuels.

The second part of the thesis is practical part, which deals with the possible potential of spruce cones as a source for briquetting and pelleting. Briquettes and pellets without additives were produced for evaluation. The research showed that the material which was used is suitable for production of briquettes and also pellets. Physical, mechanical and chemical properties of briquettes and pellets were evaluated and emissions released during combustion process were compared between densified biofuels (cone briquettes, pellets) and cones in their initial form.

Keywords: Norway spruce, cones, briquettes, pellets, combustion, emissions

Table of content

Declaration.....	i
Acknowledgement.....	ii
Abstract.....	iii
1 Introduction.....	1
2 Literature review.....	2
2.1 Part one – Renewable energy sources.....	2
2.1.1 Biomass energy.....	3
2.1.2 Solid biofuels.....	3
2.1.2.1 Densification of biomass.....	4
2.1.2.2 Briquettes.....	4
2.1.2.3 Pellets.....	5
2.1.2.4 Direct combustion.....	5
2.2 Part two – Background of Norway spruce.....	7
2.2.1 Botanical description.....	7
2.2.2 Range of growth.....	8
2.2.3 Cultivation of Norway spruce.....	9
2.2.4 Utilization of Norway spruce.....	9
2.2.5 Pest and diseases.....	10
3 Objectives and hypotheses.....	11
3.1 General objective.....	11
3.2 Specific objectives.....	11
3.3 Hypotheses.....	11
4 Methodology.....	12
4.1 Literature review.....	12
4.2 Practical research.....	12

4.2.1	Material.....	12
4.2.2	Methods	12
4.2.2.1	Classification according to origin and source.....	13
4.2.2.2	Analytic sample preparation	13
4.2.2.3	Ash content determination.....	13
4.2.2.4	Nitrogen, Carbon and Hydrogen content determination	15
4.2.2.5	Gross calorific value determination.....	15
4.2.2.6	Calculation of net calorific value	16
4.2.2.7	Preparation of raw material	16
4.2.2.8	Briquetting process	17
4.2.2.9	Pelleting process	17
4.2.2.10	Dimensions determination.....	18
4.2.2.11	Moisture content determination.....	19
4.2.2.12	Determination of mechanical durability of briquettes.....	20
4.2.2.13	Determination of mechanical durability of pellets	20
4.2.2.14	Thermal emission analysis.....	21
5	Results and discussion.....	24
5.1	Properties of Norway spruce cones	24
5.1.1	Origin and source	24
5.1.2	Ash content	24
5.1.3	CHN content.....	25
5.1.4	Gross and net calorific value	25
5.2	Solid fuel from Norway spruce cones	27
5.2.1	Production of briquettes	27
5.2.1.1	Dimensions and shape of briquettes	27
5.2.1.2	Moisture content of briquettes.....	28

5.2.1.3	Mechanical durability of briquettes	29
5.2.1.4	Summary of briquette results.....	30
5.2.2	Production of pellets	30
5.2.2.1	Dimensions and shape of pellets	31
5.2.2.2	Mechanical durability of pellets	32
5.2.2.3	Summary of pellet results	32
5.2.3	Thermal emissions evaluation	33
6	Conclusion	37
6.1	Recommendation for future research	38
7	References	39
8	Annexes	I

List of tables

Table 1. Ash content of Norway spruce cones	24
Table 2. Content of C,H,N in Norway spruce cones	25
Table 3. Gross and net calorific value	26
Table 4. GCV and NCV of different plants	26
Table 5. Length and diameter of briquettes from Norway spruce cones.....	28
Table 6. Moisture content of Norway spruce cones briquettes	28
Table 7. Mechanical durability of briquettes	29
Table 8. Mechanical durability of different materials	29
Table 9. Parameters of briquettes	30
Table 10. Length and diameter of pellets from Norway spruce cones.	31
Table 11. Mechanical durability of pellets	32
Table 12. Summary of pellet's results	32
Table 13. Emissions of measured materials	33

List of figures

Figure 1. Spruce cones with seeds	8
Figure 2. Norway spruce	8
Figure 3. Distribution in Europe and USA	9
Figure 4. Spruce beetle and infected tree	10
Figure 5. Grinding knife mill Retsch Grindomix GM 100.....	13
Figure 6. Muffle furnace LAC.....	14
Figure 7. LECO CHN628 Determinator with autoloader.....	15
Figure 8. Calorimeter IKA 6000 and prepared pellet in oxygen bomb	16
Figure 9. Hammer mill with sieve	17
Figure 10. Hydraulic piston press Brikstar.....	17
Figure 11. Pelleting line MGL 200.....	18
Figure 12. Digital calliper.....	18
Figure 13. Oven Memmert 100-800 and prepared samples	19
Figure 14. Rotation drum	20
Figure 15. Durability tester for pellets with sieve	21
Figure 16. Automatic pellet boiler.....	22
Figure 17. Emission analyzer Testo 350 XL	22
Figure 18. Produced briquettes	27
Figure 19. Dimension and shape of produced briquettes	27
Figure 20. Produces pellets.....	31
Figure 21. Dimension and shape of produced pellets.....	31

List of graphs

Graph 1. Mechanical durability of briquettes	30
Graph 2. Dependency of CO and NO _x for woody pellets.....	34
Graph 3. Dependency of CO and NO _x for spruce cones.....	35
Graph 4. Dependency of CO and NO _x for cones pellets.....	35
Graph 5. Dependency of CO and NO _x for cones briquettes	36
Graph 6. Dependency of CO ₂ of all measured materials	36

List of abbreviations

C	Carbon
CO	Carbon monoxide
CO ₂	Carbon dioxide
CULS	Czech University of Life Sciences Prague
EC	European Commission
EU	European Union
FE	Faculty of Engineering
FTA	Faculty of Tropical AgriSciences
GCV	Gross calorific value
GHGs	Greenhouse gases
H	Hydrogen
MC	Moisture content
NCV	Net calorific value
N	Nitrogen
NO	Nitrogen oxide
NO ₂	Nitrogen dioxide
O ₂	Oxygen
RES	Renewable energy sources
RIAE	Research Institute of Agricultural Engineering in Prague

List of symbols

°C	Degree Celsius
a.r.	As received
cm	Centimetre
d.b.	Dry basis
g	Gram (unit of weight)
kg.m ⁻³	Kilogram per cubic metre
mg	Milligram
mg.m ⁻³	Milligram per cubic meter
MJ.kg ⁻¹	Megajoule per kilogram
mm	Millimetre
ml	Millilitre
MPa	Megapascal
ppm	Parts per million

1 Introduction

One of the world's serious concerns these days is to find an appropriate energy source as an alternative for fossil fuels. With the increase of population and urbanisation, there is also an enormous demand for energy supply, which is still covered mostly by the fossil fuels. Although they are exhausting in very rapid way, there are alternatives, which can significantly help to alleviate this problem. One of the alternatives is renewable energy source in form of biomass, which can partly substitute the decreasing fossil fuels.

Solid biofuels, made from biomass, are becoming of great interest as an environmental friendly and renewable source of energy. Finding appropriate materials for production of quality solid biofuels has nowadays an important priority.

The Norway spruce (*Picea abies* L.) is one of the sources of biomass, wood residues or cones have a potential for production of briquettes or pellets. Due to its fast growth, the Norway spruce was planted widely in many northern countries and nowadays its area of distribution goes even to many southern countries. The Norway spruce was planted as a source of firewood, which leaves large amount of unused biomass such as wood residues and cones.

Since the Norway spruce is mostly used as firewood, in paper industry or timber, the main objective of this thesis is to assess possible use of cones as a source for solid biofuels, namely production of briquettes and pellets.

2 Literature review

The literature review of this work is divided into two main parts. Part one provides general information about renewable energy sources with focus on biomass energy and specifically on solid biofuels such as briquettes and pellets and also mentions direct combustion.

Second part shortly describes the background of the Norway spruce, its distribution and utilization.

2.1 Part one – Renewable energy sources

Fossil fuels, which are widely used all around the world mostly for energy purposes, are in a danger of depletion. Energy demand rapidly increases with the fast population and urbanisation growth and development of new technologies. So far the fossil fuels cover about four fifth of the energy consumption (Brožek *et al.*, 2012).

Renewable energy is according to Directive 2009/28/EC defined as: “*energy from renewable non-fossil sources, namely wind, solar, aero-thermal, geothermal, hydrothermal and ocean energy, hydropower, biomass, landfill gas, sewage treatment plant gas and biogases*”. Renewable energy sources have many advantages comparing to fossil fuels. One of the main advantages is reduction of greenhouse gases, sustainable production in long-term point of view and independency on the fossil fuels.

The policy for the production of energy from renewable sources in European Union was established by the Renewable energy directive, which states that at least 20 % of EU’s total energy consumption should be covered by renewable sources by the year of 2020 (EC, 2015). In 2013 the consumption of renewable energy was almost achieved with the value of 19.1 % where the renewable energy sources such as hydropower, wind power, solar power, geothermal drills and modern biomass, contributed with 10.1 % and traditional biomass used for cooking and heating in rural areas in developing countries contributed with 9 % (REN21, 2015).

2.1.1 Biomass energy

Use of biomass for energy purposes is nowadays one of great interests. It is sustainable and renewable source of energy and can contribute to the economic development (Pambudi *et al.*, 2010). According to the Directive 2009/28/EC of the European Commission the biomass is defined as “*the biodegradable fraction of products, waste and residues from biological origin from agriculture (including vegetal and animal substances), forestry and related industries including fisheries and aquaculture, as well as the biodegradable fraction of industrial and municipal waste*”. According to McKendry (2002) and Faik (2013), the content of biomass is 40-50 % cellulose, 20-25 % lignin, 15-25 % hemicellulose and 5-10 % other compounds, although the content of the compounds depends on the plant species. Share of these components is important during establishing the plant’s suitability as a source of energy (McKendry, 2002).

There are several ways how to transform the biomass into energy. According to McKendry (2002), Celjak (2008) and Perďochová (2010) the ways are: **thermochemical** (pyrolysis, gasification, combustion), **biochemical** (anaerobic digestion, fermentation), and **physicochemical** (transesterification) or **mechanical** (briquetting, pelleting). According to these methods, the biomass is further processed into **solid biofuel** such as briquettes, pellets, etc., gaseous fuels such as syngas and biogas, liquid fuels (biodiesel, bioalcohol), or heat (Sims *et al.*, 2006).

One of the most efficient and economically suitable methods is direct combustion to produce heat for heating in houses or water, or producing electricity by transforming the water into steam and ran it through a turbine (Ferry, Cabraal, 2006).

2.1.2 Solid biofuels

Due to the fast exhaustion of the reserves of fossil fuels and increasing demand for energy, the biofuels made from biomass are highly demanded. Biomass materials for energy purposes can be classified according to source of origin or conversion process (Tumuluru *et al.*, 2010).

Solid biofuels are mostly made from waste biomass and with different processing. Woodchips, shavings or logs can be produced by grinding, chipping or cutting;

briquettes and pellets are made by compaction of the material; charcoal is produced by the process of slow pyrolysis.

2.1.2.1 Densification of biomass

One of the most important problems of biomass is its transportation and storing in its initial form. It is so due to its high moisture content, which ranges from 10-70 %, low bulk density and irregular particle size distribution and shape. A feasible solution for this issue is to transform raw material to briquettes or pellets by using the process of densification. According to EN ISO 16559 (2014) the densified biofuels is defined as a biomass which was mechanically compressed, or treated thermally, to obtain a specific shape and size such are briquettes, pellets, cubes or pressed logs. Bulk density is one of the most important parameters when comes to biomass densification. According to Mani *et al* (2003) the bulk density can increase from initial values of 40-200 kg.m⁻³ up to 600-800 kg.m⁻³. Pellets are one of the most densified types of biofuel and it can have values of bulk density up to 600-1,200 kg.m⁻³(McKendry, 2002).

2.1.2.2 Briquettes

Briquettes are according to the Standard EN ISO 17225-3 (2014) defined as “*densified biofuel made with or without additives in form of cubiform, prismatic or cylindrical unit with diameter of more than 25 mm produced by compressing milled biomass*”. In practical view, briquettes are compacted cylinders made of different organic materials with a diameter range from 40 to 100 mm and a length of 300 mm and are mainly used for combustion to generate thermal energy for heating and cooking (Shaw, 2008).

Briquettes could be used as suitable, more ecological alternative for fossil fuels such as coal and firewood. Use of briquettes can be time and money saving and also helps to decrease local deforestation. Agricultural residues or other waste biomass is not often suitable as a source of solid biofuels, because of its high volume, which complicates distribution and transportation. The main reason for compressing biomass is to achieve desirable shape, volume and structure of the material (Pambudi *et al.*, 2010). In terms of fuel compaction, briquetting is the most applied technology (Ivanova, 2012). According to Tumuluru *et al.* (2010) factors, which mostly affect the briquettes

properties, are: shape and size of briquette, particle size distribution, surface area, porosity, bulk density, moisture content, and strength. Van Look and Koppejan (2002) claim, that for burning of briquettes are suitable wood boilers, fireplaces and central heating boilers.

2.1.2.3 Pellets

According to the Standard EN ISO17225-2 (2014) woody pellets are defined as “*densified biofuel made from woody biomass with or without additives usually with a cylindrical form, random length typically 5 to 40 mm and diameter up to 25 mm and broken ends*“. The raw material used for making wood pellets is from woody biomass (biomass from trees, shrubs, bushes). Pellets are mostly processed in a die, with moisture content less than 10 % in total.

According to Andert *et al* (2006) the optimal ratio between length and diameter of the pellets is 1:3 and should not be exceeded. When the water content of the material is low, additional wetting could be done before the process of pressing, which helps to bind the particles together (Kott, 2010).

2.1.2.4 Direct combustion

Combustion is a chemical process, in which the fuel reacts rapidly with oxygen and release heat. During this process, carbon dioxide and water vapour are produced (Stupavský and Holý, 2010). According to Pastorek (2004), the combustion process has four stages, first is the drying of the fuel followed by pyrolysis, then combustion of the gaseous compounds and finally combustion of the solid parts.

During the designing of boilers, bulk density, net calorific value, ash melting and volatile content has to be taken in account (Van Look Koppejan, 2002). According to Carrol and Finnan (2012) woody pellets has ash content between 0.5-2.5 % with net calorific value of 17.5-19.5 MJ.kg⁻¹, and biomass pellets vary between 1-9 % of ash content and net calorific value of 15-18 MJ.kg⁻¹.

Emissions are one of the most important parameter in the field of biofuels. According to the regulation 201/2012 Sb. (MZP, 2012) emissions are introduced as one or more pollutants which are present in the air. Air pollutant is described as any substance present in the atmosphere which can have detrimental effects on humans and

environment. Havrland *et al.* (2011) state that CO₂ released during the combustion is similar to the CO₂ fixed in plants. It means that the CO₂ in the atmosphere is not increasing during combustion of the biomass. Among the main pollutants belong Carbon monoxide (CO), Nitrogen oxides (NO_x), Sulphur dioxide (SO₂), Total organic carbon (TOC) and solid polluting matter (MZP, 2012).

2.2 Part two – Background of Norway spruce

This chapter provides general information about origin and botanical classification of Norway spruce and its historical background.

2.2.1 Botanical description

Norway spruce is one of the most widely planted trees. It is an evergreen coniferous tree, which grows fast and reaches height from 35 to 65 m. Its trunk diameter is approximately from 0.8 to 1.8 m. When the tree is younger it can grow at the speed of 1 m per year (Farjon, 1990).

The Norway spruce belongs to the family called *Pinaceae* and its botanical name is *Picea abies* L. According to Farjon (1998) this family contains from 220 to 250 different species, which place them among the one of the most abundant trees. Most of these species can be found on the Northern hemisphere, but they can range from subarctic to tropical locations, and they form mostly boreal, coastal and montane forests. Norway spruce is also known as white deal, common spruce and according to the country of origin also Baltic, Finnish or Russian whitewood. The cones of the Norway spruce are woody, elongated, overhanging and they have pointed scales. They become mature in the first year and can be from 10-16 cm long and 3-4.5 cm in diameter (Figure 1). The cones fall off altogether (Naturbohemica, 2008). The seeds are dark brown up to 0.5 cm (Figure 1).

The Norway spruce (Figure 2) is widely planted because of the use of its wood and it is also widely used as a Christmas tree. It can live from 200-300 years (Naturbohemica, 2008).



Figure 1. Spruce cones with seeds (Source: Author, 2017)



Figure 2. Norway spruce (Source: Wikipedia, 2017)

2.2.2 Range of growth

Norway spruce has a wide range of growth (Figure 3). It is native to Northern, Central and Eastern Europe and it goes south to northern Greece and to the west of the France. It is now naturalized in Europe out of its native range, which includes Great

Britain and the Pyrenees Mountains, and it is also spread in the north-central of the United States and adjacent Canada (Taylor, 1993).

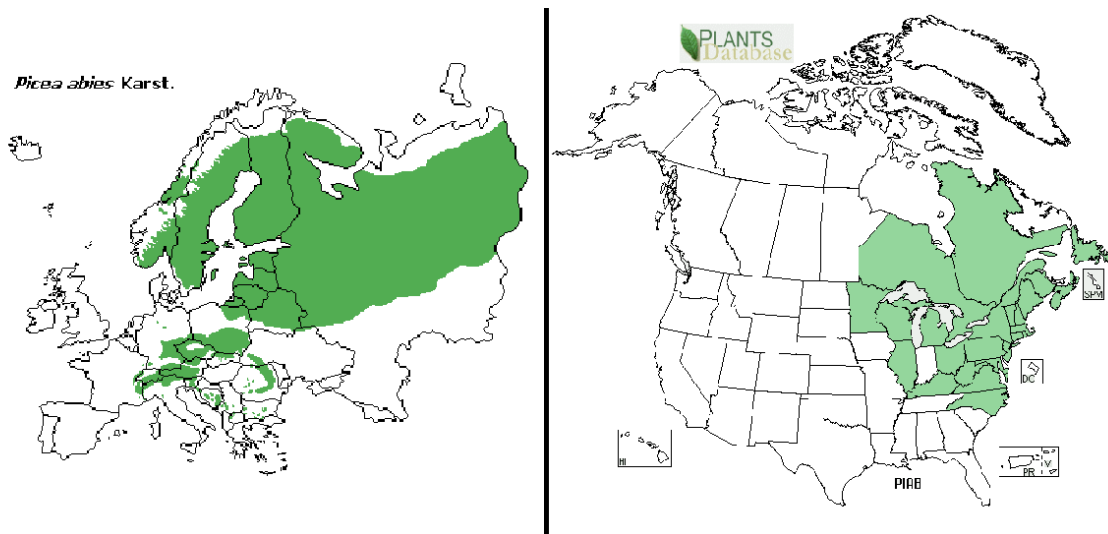


Figure 3. Distribution in Europe and USA (Source: Plants Database, 2016)

2.2.3 Cultivation of Norway spruce

Among all the species of spruces, the Norway spruce is one the most widely planted. Also it is one of the most important coniferous trees in the economically point of view. It is even planted in parks and gardens and big part of it is planted for use as a Christmas tree.

In the northern parts of the United States and Canada, this tree is reported in some locations as an invasive.

The Norway spruce is tolerant to acid soils but not so tolerant with dry or deficient soils. It is often planted on the surface mines. It has a shallow root system which allows it to adapt almost on every surface (Sullivan, 1994).

2.2.4 Utilization of Norway spruce

The wood of the Norway spruce is strong with a straight grain and a fine texture, although it is classified as a soft wood (The Wood-database, 2016). It is mostly used in forestry for timber and in paper industry. It also serves as a tone wood which is used for making some musical instruments such as pianos or violins. It is used to equip interiors, to frames and floor covering or generally for making crates and boxes (Hostetter, 2017).

The Norway spruce can also be used as a source for spruce beer, which is a beverage flavoured with needles, buds and essence of spruce trees. The needles also contain a large amount of vitamin C, so they can be consumed as a tea or eaten directly from the tree (Karellp, 2012).

These trees can serve for windbreak, screen, borders and specimen plants (Norwayspruce, 2016).

2.2.5 Pest and diseases

The Norway spruce belongs to the most resistant spruce species. It is affected by insect but in very small scale. Some moulds and fungi effect and damage the trees. In Europe lives Spruce beetle or engraver beetle (Figure 4), which is one the most destructive pests of spruce in Europe. This beetle attacks damaged and even healthy trees. It is fed on the bark of the trees and can reproduce very quickly (Norwayspruce, 2016).



Figure 4. Spruce beetle and infected tree (Source: Insectfoto, 2017)

3 Objectives and hypothesis

3.1 General objective

The main objective of the thesis was assessment of utilization of spruce cones for energy purposes in form of solid biofuels.

3.2 Specific objectives

Specific objectives are supporting and supplementing the main objective. Definition of the specific objectives is following:

- i. to produce briquettes and pellets from Norway spruce cones;
- ii. to measure chemical, physical and mechanical properties of solid biofuels from spruce cones according to EN and ISO standards;
- iii. to compare utilization of briquettes and pellets vs loose cones based on their properties and combustion characteristics.

3.3 Hypotheses

- i. Norway spruce cones can be used for production of briquettes and pellets without any additives.
- ii. Mechanical durability of Norway spruce cones briquettes is affected by material's fraction.
- iii. Emissions released during combustion of briquettes and pellets are higher compared to combustion of Norway spruce cones in its initial form.

4 Methodology

Methodology of this thesis consists of two main parts. First part is focused on the writing of the literature review and second part describes the practical research.

4.1 Literature review

To establish the theoretical part and literature review, scientific articles from Czech and foreign sources were used. Most articles were obtained from scientific databases such as Science Direct, Scopus, Web of Knowledge and Google Scholar. Articles were identified by combining keywords: biomass, briquettes, pellets, spruce cones utilization, pine cones, Norway spruce, combustion emissions, etc.

4.2 Practical research

Practical research consists of following parts:

4.2.1 Material

Spruce cones were used for research in the practical part. Material was collected in Kostelec nad Černými lesy and was provided by the Faculty of Forestry and Wood Sciences, Czech University of Life Sciences Prague.

Initial material had average moisture content of 10.6 %.

4.2.2 Methods

The research was done partially in Laboratory of biofuels in FTA, CULS, Faculty of Engineering, CULS and also in Research Institute of Agricultural Engineering in Prague.

Practical research started with measurement of physical and chemical properties of spruce cones according to methodology of European Technical Committee for Solid Biofuels. Further, solid biofuel in form of briquettes and pellets from the spruce cones were produced and tested. Mainly mechanical properties (mechanical durability) were measured. At the end, determination of emissions released during the combustion of briquettes, pellets and initial material was done.

4.2.2.1 Classification according to origin and source

Classification of the material is based on origin and source of biofuel. Source of biofuel was determined according to standard ISO 17225-1 (2014): Solid biofuels- Fuel specifications and classes- Part 1: General requirements.

4.2.2.2 Analytic sample preparation

Raw material made from crushed spruce cones was grinded to the particle size lower than one mm by using of grinding knife mill Retsch Grindomix GM 100 (Figure 5) in Laboratory of biofuels, FTA. According to standard EN 14780 (2011): Solid biofuels- Sample preparation, the analytic sample had completely homogenized correct size.



Figure 5. Grinding knife mill Retsch Grindomix GM 100 (Vlachosová, 2016).

4.2.2.3 Ash content determination

According to standard EN 14775 (2009): Solid biofuels- Determination of ash content, the ash content was determined in Muffle furnace LAC (Figure 6). Measurement of ash content was done in Laboratory of solid biofuels, FTA, and was defined by calculating the weight of the inorganic residues after combustion of the sample under controlled temperature. Formula bellow shows the calculation of measuring the ash content. For weighing the material, the digital laboratory scale KERN (model EW 3000-2M) with readout 0.1 mg was used.



Figure 6. Muffle furnace LAC (Source: Author, 2016).

First part of the measurement consists of heating up empty porcelain crucibles up to 550 °C for 60 minutes. After that, the crucibles were cooled down on a heat resistant plate for 10 minutes and then moved to a desiccator without desiccant for cooling down to an ambient temperature. Next step of first part was weighing one gram of analytic sample, which was put into cooled crucibles. In the second part were the crucibles with analytic sample placed into the cold furnace and then continually heated up to 250 °C over a period of 30 minutes. The temperature was kept for 60 minutes to allow the volatiles to leave the sample before ignition. During next 30 minutes temperature was raised up to 550 °C and maintained for 120 minutes to achieve absolute combustion. Ash content was determined for three samples and the final result was calculated as arithmetic mean of two nearest measurements, whereas the difference between two of them did not exceed 0.2 %.

$$Ad = \frac{(m3 - m1)}{(m2 - m1)} \cdot 100 \cdot \frac{100}{100 - Mad} [\%]$$

Ad—ash content [%]

m1—weight of the empty porcelain crucible [g]

m2—weight of porcelain crucible with wet sample [g]

m3—weight of porcelain crucible with ash [g]

Mad—average moisture content of used sample [%]

4.2.2.4 Nitrogen, Carbon and Hydrogen content determination

Content of Nitrogen, Carbon and Hydrogen was determined according to standard EN 15104 (2011): Solid biofuels- Determination of total content of carbon, hydrogen and nitrogen-Instrumental method. The analysis of these elements was done by automatic device LECO CHN628 Series Elemental Determinator (Figure 7) in the Laboratory of RIAE, Prague. For control of quality it is essential to determine CHN elements as they are decisive components of emissions released during combustion process.



Figure 7. LECO CHN628 Determinator with autoloader (Source: Author, 2016)

The method consisted of weighing of 0.1 g of analytic sample and wrapping it into aluminium foil to form a small globule. Then the sample was put into autoloader and afterwards into the combustion chamber due to removal of atmospheric gas. Further the sample moved to the dual- stage furnace system, which operated with temperatures up to 1,050 °C, with pure oxygen to assure complete combustion of all samples. The results were automatically processed and displayed on the laboratory computer.

4.2.2.5 Gross calorific value determination

According to standard EN 14918 (2009): Solid biofuels- Determination of calorific value, gross calorific value was measured in the Laboratory of RIAE, Prague by automatic Calorimeter IKA 6000 (Figure 8). For determination was used manually pressed pellet from analytic sample weighing approximately one gram. During complete

combustion of pellet in the pressure vessel in the calorimeter under compressed oxygen at the temperature of 22 °C, amount of heat per unit of weight was released. The results were automatically calculated and displayed on the calorimeter after entering the weight of the sample and H content.



Figure 8. Calorimeter IKA 6000 and prepared pellet in oxygen bomb (Source: Author, 2016)

4.2.2.6 Calculation of net calorific value

The net calorific value was calculated from the gross calorific value according to equation bellow. Calculation was done according to standard EN 14918 (2009): Solid biofuels- Determination of calorific value.

$$Q_i = Q_{gr} - 24.42 \cdot (M_{ad} + 8.94H) \text{ [J.g}^{-1}\text{]}$$

Q_i —net calorific value [J.g⁻¹]

Q_{gr} —gross calorific value [J.g⁻¹]

24.42—heat of water evaporation

M_{ad} — moisture content [%]

8.94—coefficient for conversion of hydrogen to water

H— hydrogen content [%]

4.2.2.7 Preparation of raw material

Spruce cones were crushed by Hammer mill 9FQ-40C (Figure 9) with sieves of three different diameters of holes, i.e. 4, 12 and 25 mm.

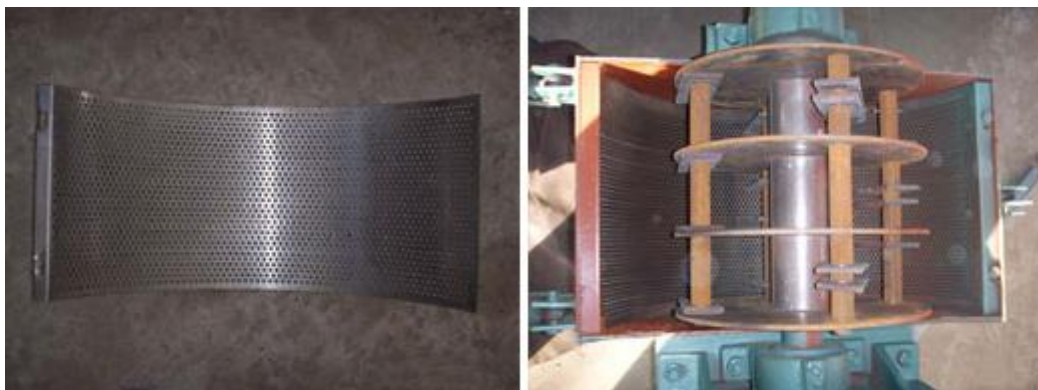


Figure 9. Hammer mill with sieve (Source: Fote Machinery, not dated)

4.2.2.8 Briquetting process

Making of briquettes from three initial fractions was done on the hydraulic piston briquetting press Brikstar model CS 50-12 (Figure 10) produced by Briklis Company (Czech Republic) with the operating pressure of 18 MPa. Process of briquetting was conducted at the FE, CULS. Briquettes were made from crushed spruce cones without any additional binding agents, with initial moisture content and at the room temperature. Shape of the briquettes was cylindrical.



Figure 10. Hydraulic piston press Brikstar (Source: Vlachosová, 2016).

4.2.2.9 Pelleting process

For producing pellets was used line MGL 200 (Figure 11) made by company Kovo Novák (Czech Republic) in the Laboratory of RIAE, Prague. Grinded spruce

cones (fraction 4 mm) were manually added to the pelleting line without any additives. Pelletizing press consists of flat die with 6 mm holes and rollers.



Figure 11. Pelleting line MGL 200 (Source: Kovo Novák, 2016)

4.2.2.10 Dimensions determination

According to standard EN 16127 (2012): Solid biofuels- Determination of length and diameter of pellets, pellets were measured by digital calliper device (Figure 12). First according to the standard, 80 to 100 grams of pellets were weighed. For determination of diameter, 10 pellets were randomly selected and measured. Length of the pellets was established by measuring of each of the selected pellet. Arithmetic mean of all values was used as a result. Similarly the briquette's dimensions were measured using the calliper device. Dimension measurement of briquettes was done according to EN ISO 17225-3 (2014).



Figure 12. Digital calliper (Source: Author, 2017)

4.2.2.11 Moisture content determination

In the Laboratory of biofuels, FTA, the moisture content of the briquettes was determined according to standard EN 14774-2 (2009): Solid biofuels- Determination of moisture content- Oven dry method- Total moisture- Simplified method. A digital laboratory balance Kern (model EW 3000-2M) with readout 0.1 mg was used during the weighting of material and an oven Memmert (model 100-800) was used for drying (Figure 13).



Figure 13. Oven Memmert 100-800 and prepared samples (Source: Author, 2016)

Empty dishes were put into the oven which was heated up to 105 °C. After the constant temperature was achieved, dishes were removed and cooled in the desiccator with desiccant. After 15 minutes, when cooled to room temperature, the dishes were removed and weighed. Samples of briquettes were put onto these dishes, weighed together and placed into the oven for drying at the temperature of 105 °C until the weight was constant in mass. After the drying process, the dishes with briquettes were moved out, cooled in desiccator and weighed again. The moisture content was calculated by using the following formula. This process was done twice and arithmetic mean was considered as a result.

$$Mad = \frac{(m2 - m3)}{(m2 - m1)} \cdot 100 \text{ [%]}$$

Mad—moisture content [%]

m1— mass of the empty dish[g]

m2—the mass of the dish plus sample before drying [g]

m3—the mass of the dish plus sample after drying [g]

4.2.2.12 Determination of mechanical durability of briquettes

Mechanical durability of produced briquettes was measured according to EN 15210-2 (2010) in the FE, CULS. For measuring was used rotation steel drum with cylindrical shape (Figure 14). Inside of the drum is equipped with rectangular steel baffle. Opening of the drum is covered with dustproof lid. A rotation counter is connected to the drum.

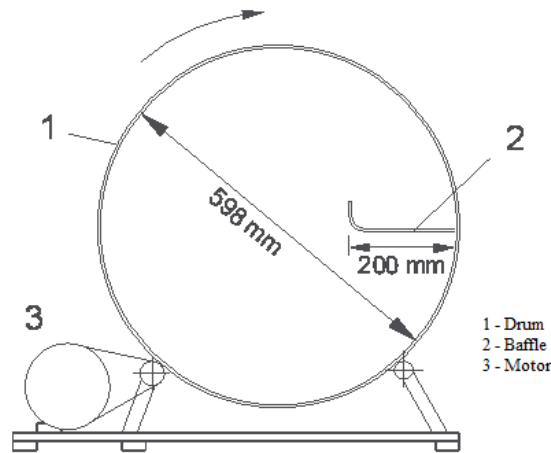


Figure 14. Rotation drum (Source: EN 15210-2, 2010)

Approximately 2 kg of briquettes were weighed and put into the drum. The rotation was set to 5 minutes. After the process was done, the briquettes were removed and weighed again. This procedure was repeated 4 times for each sample. The mechanical durability was calculated according to following formula:

$$DU = \frac{m_A}{m_E} \times 100$$

DU= mechanical durability (%);

m_E= pre-sieved briquettes before the drum treatment (g);

m_A= sieved briquettes after the drum treatment (g).

4.2.2.13 Determination of mechanical durability of pellets

Mechanical durability determination of pellets was done according to standard EN 15210 - 1 (2009): Solid biofuels- Determination of mechanical durability of pellets by Pellet durability tester (Figure 15) in the Laboratory of RIAE, Prague.

Pellet tester with two rotating steel drums and with speed of 50 revolutions per minute was used for determination of mechanical durability. Pellets were weighed four times, each time with the weight of 500 ± 10 g. After that two parts of weighed pellets were put into each drum of the tester for 8 minutes. When finished, pellets were sieved and weighed again. The mechanical durability was then calculated according to the same formula as above.



Figure 15. Durability tester for pellets with sieve (Source: Author, 2017)

4.2.2.14 Thermal emission analysis

Measurement of thermal emissions was based on combustion of three types of fuel (cones' briquettes, cones' pellets and cones in initial form) and was carried on in the Laboratory of RIAE, Prague. First, pellets made from spruce cones were burned in automatic pellet boiler (Figure 16) from Czech company Kovo Novák, which is specifically designed for burning fuel in form of pellets.



Figure 16. Automatic pellet boiler (Source: Author, 2017)

Emission analyser Testo 350 XL (Figure 17) was used for the measuring of gaseous compounds. Flue gas recording was done during continuous combustion. Testo analyser measured the temperature of flue gas and emissions' content of CO, NO and NO₂. Measurement took approximately one hour for one sample, as is stated in the regulation.



Figure 17. Emission analyzer Testo 350 XL (Source: Author, 2017)

Second, briquettes from spruce cones were burned in classical tiled stove and emissions were measured by the same device as with the pellets. Approximately three kilograms of briquettes were put into the preheated stove and after fluent flow of air and steady combustion the emissions were measured for about one hour in total.

At last, spruce cones were burned also in the tiled stove and with the same procedure as the briquettes.

The method of testing of all three samples was the same, a probe, which was a part of Testo measuring device, was placed into the vent (special hole) on the chimneys of pellet boiler and tiled stove and automatic program for emission measurement was launched.

The analyser calculated gas components, which initiate from the values from the direct measurement and from the characteristics of the fuel. The results were calculated according to following formula.

$$CO_2 = CO_{2max} \cdot \left(1 - \frac{O_2}{20.95}\right) [\%]$$

CO_2 = measured carbon dioxide content [%]

CO_{2max} = maximum content of carbon dioxide measured for given fuel, based on elemental analysis [%]

O_2 = measured oxygen content in volume [%]

20.95 = content of oxygen in air [%]

$$NO_x = NO + NO_2$$

NO and NO_x = values directly measured in ppm

5 Results and discussion

This chapter shows results obtained by practical research and compares them with results and observations of other authors. First, physical and chemical properties of Norway spruce cones such as moisture content, ash content, calorific value, contents of C, H, N, etc. are presented. Second, the results from briquetting and pelletizing process are listed. Third, evaluation of emissions during combustion process of briquettes, pellets and initial material is made. Most of the results were calculated as an arithmetic means according to repeatability limit of the current standards for solid biofuels. All measured values are available in the Annex.

5.1 Properties of Norway spruce cones

Chemical and physical properties of the initial material were measured according to EN ISO 17225-2 (2014): Solid biofuels- Fuel specifications and classes, Part 2: Graded woody pellets.

5.1.1 Origin and source

Norway spruce cones were according to standard ISO 17225-1 (2014) categorized as Woody biomass, biomass from trees, bushes and shrubs.

5.1.2 Ash content

Determination of ash content of spruce cones was done according to standard EN 14775 (2009). The result is listed in Table 1.

Table 1. Ash content of Norway spruce cones

Material	Ash content d.b.[%]
Norway spruce cones	2.1

Source: Author, 2017

According to EN ISO 17225-3 (2014) the ash is classified into the category B, where woody briquettes may achieve ash content up to 3 %. Category A values range from 1 to 1.5 %.

Mužik and Souček (2010) measured ash content in woody briquettes made from waste wood during vineyard pruning and the value was 3.46 % but they claim that it is slightly increased. In comparison with briquettes made from spruce cones it is still higher value.

5.1.3 CHN content

Determination of Carbon, Hydrogen and Nitrogen content were done according to standard EN 15104 (2011). The results are listed in Table 2.

Table 2. Content of C,H,N in Norway spruce cones

Material	N d.b.[%]	C d.b.[%]	H d.b.[%]
Norway spruce cones	0.72	50.9	5.72

Source: Author, 2017

Carbon and nitrogen content are important when it comes to emission release during combustion (Cutz, 2017). The Norway spruce cones had high carbon content. ISO 17225-3 (2014) allows nitrogen content from 0.3 up to 1 %, which nitrogen content of briquettes from cones fulfilled, and it is placed in category B. Category A values range from 0.3 to 0.5 %. Chandrasekaran *et al.* (2012) stated that nitrogen content of wood briquettes reaches value under 1 %.

5.1.4 Gross and net calorific value

Calorific value is a crucial parameter in case of biofuels (Özyuğuran, 2017).

Determination of the gross and net calorific value was done according to EN 14918 (2009). The results are presented in Table 3.

Table 3. Gross and net calorific value

Material	GCV [MJ.kg ⁻¹]		NGV [MJ.kg ⁻¹]	
	a.r	d.b	a.r	d.b
Spruce cones	19.4	20.4	17.8	19.04

Source: Author, 2017

Table 4 shows GCV and NCV of selected plants for comparison.

Table 4. GCV and NCV of different plants

Plant	GCV d.b. [MJ.kg ⁻¹]	NCV d.b. [MJ.kg ⁻¹]	Source
Straw	18.8	14.0	Gabrielová (2007)
Cotton plant	19.0	-	Stavjarská (2013)
Sweet sorghum	19.1	17.1	Havrland <i>et al.</i> (2013)
<i>Miscanthus x giganteus.</i>	19.3	-	
Birch wood	19.5	-	Celjak (2008)
Hemp	19.6	18.6	
Woody biomass	20.1-22.0	19.4-20.8	Jevič <i>et al.</i> (2008)

Source: Author, 2016

From the results is obvious that briquettes made from spruce cones has relatively high gross and net calorific value, especially comparing to herbaceous biomass.

According to Krajnc (2015) the coniferous wood has typical value of gross calorific value from 20.2-20.8 MJ.kg⁻¹ and net calorific value from 18.8-19.8 MJ.kg⁻¹. According to EN ISO 17225-3 (2014) briquettes fulfilled the NCV limitations, which should be higher than 15.5 MJ.kg⁻¹. In case of spruce cone's briquettes, category A achieves values from 15.3 to 15.5 MJ.kg⁻¹ and in category B the value is higher or equal to 14.9 MJ.kg⁻¹. For pellets the categories differ. Category A and B both the value is higher or equal to 16.5 MJ.kg⁻¹.

5.2 Solid fuel from Norway spruce cones

Use of pellets or briquettes as a form of solid biofuel is one of very convenient method. Above all it improves the transportation and storage conditions (Chin, 2000).

5.2.1 Production of briquettes

The process of briquetting of the spruce cones has shown that briquettes (Figure 18) can be made without any additives with standard pressure of 18 MPa.



Figure 18. Produced briquettes (Source: Author, 2017)

5.2.1.1 Dimensions and shape of briquettes

The dimension and shape of produced briquettes shown on Figure 19 was done according to EN ISO 17225-3 (2014). Results are listed in Table 3.

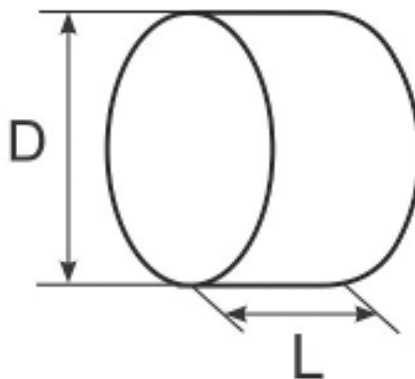


Figure 19. Dimension and shape of produced briquettes (Source: EN ISO 17225-3, 2014)

Table 5. Length and diameter of briquettes from Norway spruce cones.

Material	Length [mm]	Diameter [mm]
Norway spruce cones briquettes	70.1	52.4

Source: Author, 2017

The average value of briquettes' diameter was slightly higher than the diameter of the piston press, which was 50 mm, which is the reason that produced briquettes vary. This could be the result of adhesive properties of the material and storing of the briquettes. It is affected by particle size distribution. Brožek *et al* (2012) claim that material which is crushed finer bounds better in the pressing chamber.

5.2.1.2 Moisture content of briquettes

Moisture content of produced briquettes is listed in Table 5. According to standard EN ISO 17225-3 (2014) the moisture content of briquettes produced in piston press is lower than 15 % of the mass. Moisture content of solid biofuels affects calorific value and transport or storing, therefore it is one of the major parameter.

Table 6. Moisture content of Norway spruce cones briquettes

Material	Moisture content a.r. [%]
Norway spruce cones briquettes	11.4

Source: Author, 2017

According to EN ISO 17225-3 (2014) required moisture content for woody briquettes is bellow 12-15 % for different categories. Moisture content of briquettes from spruce cones fulfilled these limitations. This value can be compared with moisture content of coal with value of 11 % (McKendry, 2002)

5.2.1.3 Mechanical durability of briquettes

According to EN 15210-2 (2010) the mechanical durability was done. The results are listed in Table 6. With regard to the transportation and storing, this parameter is one of the important in the field of solid biofuels.

Table 7. Mechanical durability of briquettes

Briquettes fraction	Mechanical durability [%]
4 mm	80.6
12 mm	79.4
25 mm	86.7

Source: Author, 2017

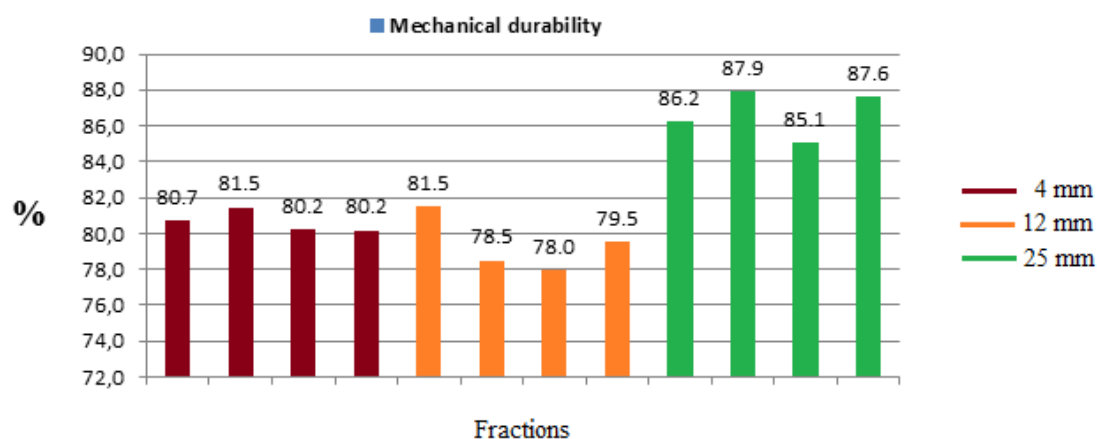
According to EN 14961-1 (2010) mechanical durability requirement is 95 % which briquettes with its value did not fulfil. Brožek *et al.* (2012) state that mechanical durability of shavings made 90 % of spruce have mechanical durability 92.2 % which is 10 % higher than mechanical durability of briquettes from spruce cones. Table 8 shows mechanical durability of different materials, which in general showed better mechanical quality comparing to spruce briquettes.

Table 8. Mechanical durability of different materials

Material	Mechanical durability [%]	Source
Hemp	97.7	Ivanova <i>et al.</i> 2014
Miscanthus giganteus	91.9	
Giant reed	90.8	

Source: Author, 2017

Graph 1 shows process of measuring of mechanical durability for four different fractions.



Graph 1. Mechanical durability of briquettes (Source: Author, 2017)

5.2.1.4 Summary of briquette results

Table 8 shows summary of results for briquettes made from spruce cones according to EN ISO 17225-3 (2013).

Table 9. Parameters of briquettes

Parameter	Unit	Result	Limitation	Evaluation
Ash content	w-%	2.1	$A \leq 3.0$ (B)	achieved
Moisture content	w-%	11.44	$M \leq 15$	achieved
Mechanical durability	w-%	82.2	$DU \leq 95$	not achieved
Net calorific value	MJ.kg ⁻¹ d.b.	19.04	$Q \geq 15.5$	achieved
Nitrogen content	w-%	0.72	$N \leq 1.0$	achieved

Category B according to EN ISO 17225-3 (2013)

The weakest point of produced briquettes is their mechanical durability, which can be improved by using higher pressure in compressing chamber, using of screw press or by using additives or biding agents (Kers *et al.*, 2010)

5.2.2 Production of pellets

Pellets (Figure 20) were produced on the Kovo Novák pelleting line MGL 200. The initial material was additionally wetted with water.



Figure 20. Produces pellets (Source: Author, 2017)

5.2.2.1 Dimensions and shape of pellets

The dimension and shape of produced pellets shown on Figure 21 was done according to EN ISO 17225-2 (2014). Results are listed in Table 7.

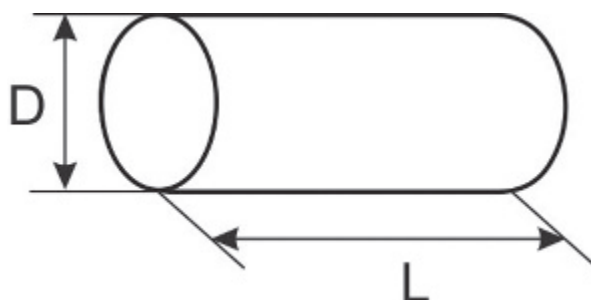


Figure 21. Dimension and shape of produced pellets (Source: ISO 17225-2, 2014)

Table 10. Length and diameter of pellets from Norway spruce cones.

Material	Length [mm]	Diameter [mm]
Spruce cones pellets	26.54	6.1

Source: Author, 2017

The diameter of the produced pellets is given due to the flat die which has the diameter of 6 mm. According to EN ISO 17225-2 (2014), diameter of woody pellets can vary from 6-8 ±1 mm and values of length can reach from 3.15-50 mm.

5.2.2.2 Mechanical durability of pellets

Determination of mechanical durability of pellets was done according to EN 15210-1 (2009). The results are shown in Table 8.

Table 11. Mechanical durability of pellets

Material	Mechanical durability [%]
Pellets	97.5

Source: Author, 2017

According to ISO 17225-1 (2014), the limitations of mechanical durability in category A is higher or equal to 97.5 % and for category B 96.5 %. In this case the requirements were fulfilled. Oveisi-Fordiie (2003) measured mechanical durability of pine wood with value of 97.7 % which is very similar value to cone's pellets.

5.2.2.3 Summary of pellet results

Table 9 displays summary of results for briquettes made from spruce cones according to EN ISO 17225-2 (2014).

Table 12. Summary of pellet's results

Parameter	Unit	Result	Limitation	Evaluation
Diameter (D),	mm	26. 54 (L)	D06-D08	achieved
Length (L)		6.1 (D)	3.15<L>50	
Mechanical durability	w-%	97.5	DU≥97,5	achieved
Net calorific value	MJ.kg ⁻¹ d.b.	19.04	Q≥16.5	achieved
Nitrogen content	w-%	0.72	N≤1.0	achieved

Source: Author, 2017

From table 9 is obvious that pellets from spruce cones achieved high quality and therefore fulfil the requirements for woody pellets.

5.2.3 Thermal emissions evaluation

Determination of emissions was done with 4 types of material. Spruce cones in its initial form, briquettes and pellets made from spruce cones and finally woody pellets, to comparison. Woody pellets and spruce cones pellets were combusted in automatic pellet boiler from Czech company Kovo Novák. Briquettes and cones were burned in classical tiled stove.

The measurements were done under controlled conditions. Oxygen content was 10 % and average temperature 120 °C.

Emissions of combusted materials are listed in Table 9.

Table 13. Emissions of measured materials

Material	CO [mg.m⁻³]	CO₂ [%]	NO_x [mg.m⁻³]
Woody pellets	7,283	10.2	117.7
Cone pellets	7,481	10	104
Cone briquettes	3,714	9.9	233
Cones	401.3	10	266.5

Source: Author, 2017

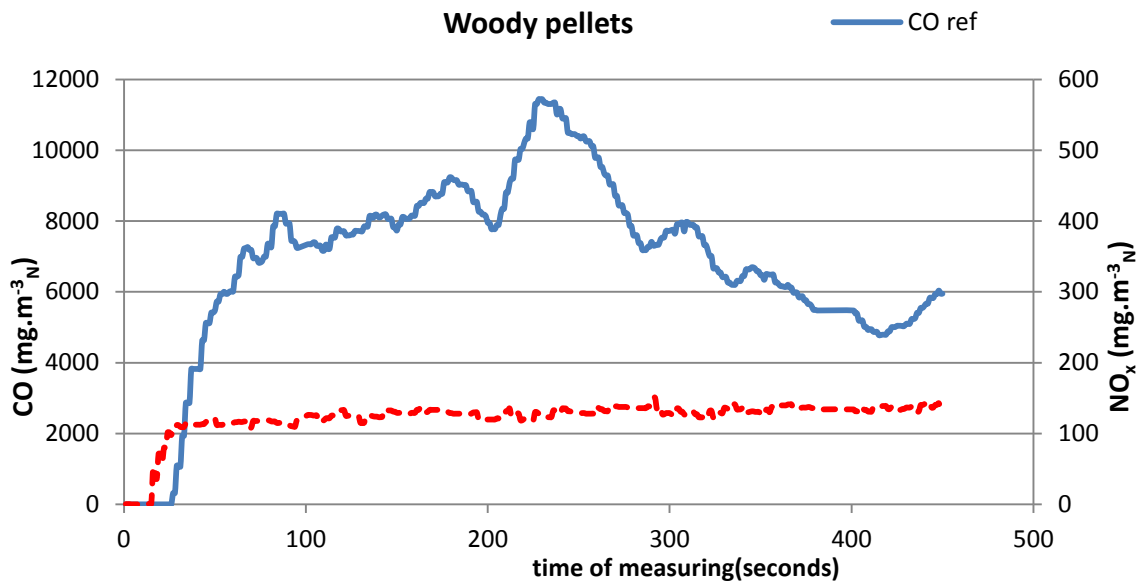
The pellets, briquettes and cones were burning fluently in controlled environment with steady access of oxygen. Intake of fuel in automatic pellet boiler did not suffocate the flame. Classical tilted stove were loaded with measured amount of briquettes, then cones, and during the combustion no additional fuel was added to preserve more or less same access of oxygen.

The CO emission limitation is according to EN 303-5 (2012) the only parameter defined for combustion of biomass in automatic boilers. Limitations value is 3,000 mg.m⁻³. Malat'ák *et al* (2010) claim that carbon oxide is not included among the main greenhouse gases (GHGs).

On the other way, CO₂ is placed among the greenhouse gases, which is specific with long lifetime in atmosphere. It has no specific determination because this gas cycles between atmosphere and biosphere (Alakoski *et al.*, 2016).

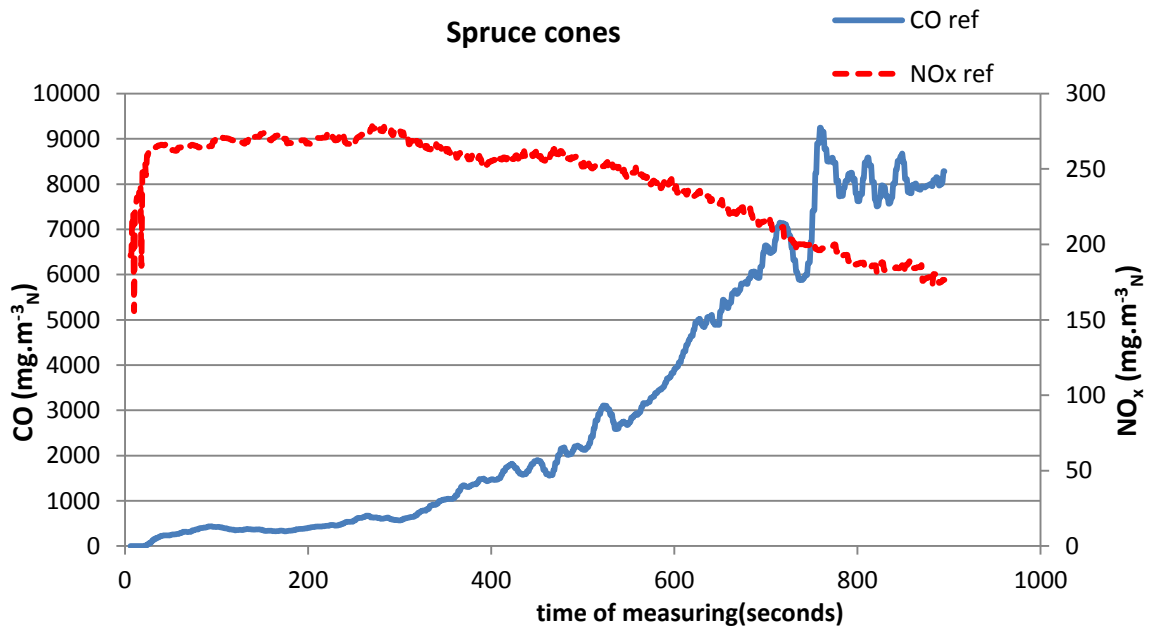
Following graphs show the process of combustion with measured values.

Graph 1 shows the values of measured CO and NO_x of woody pellets; CO₂ has to be displayed separately due to different use of units.



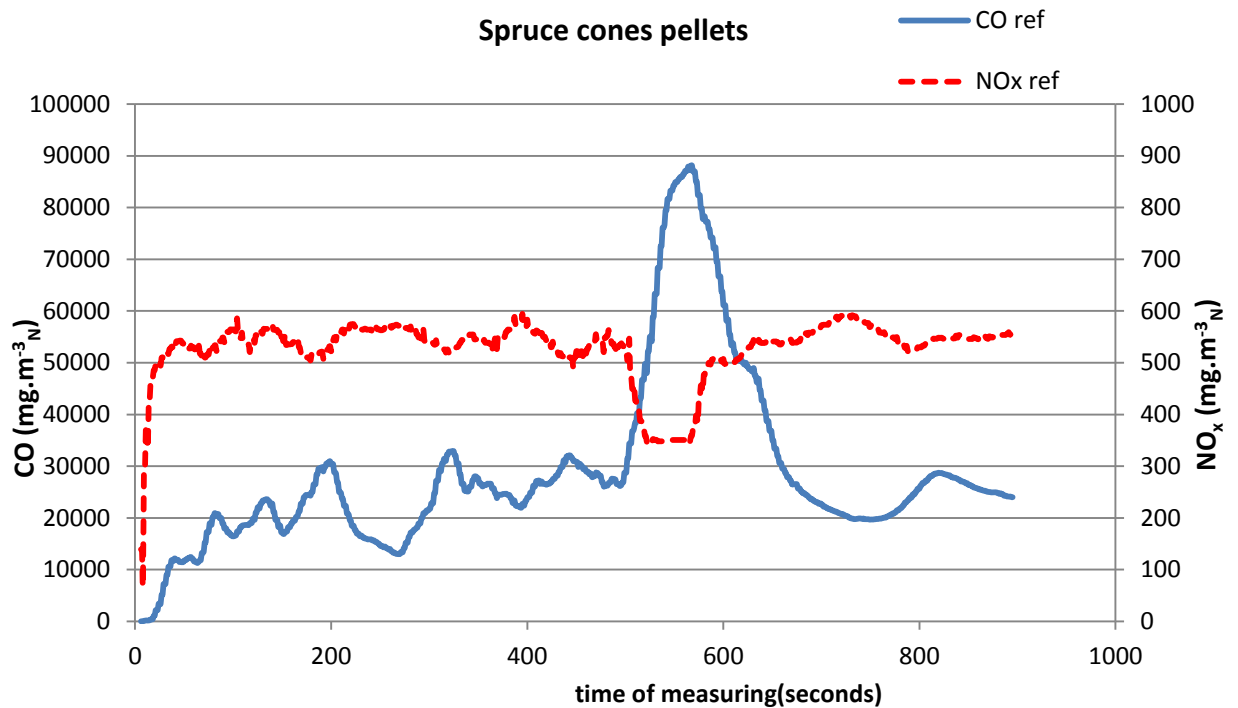
Graph 2. Dependency of CO and NO_x for woody pellets (Source: Author, 2017)

Graph 2 displays the relations of CO and NO_x of Spruce cones in its initial form.



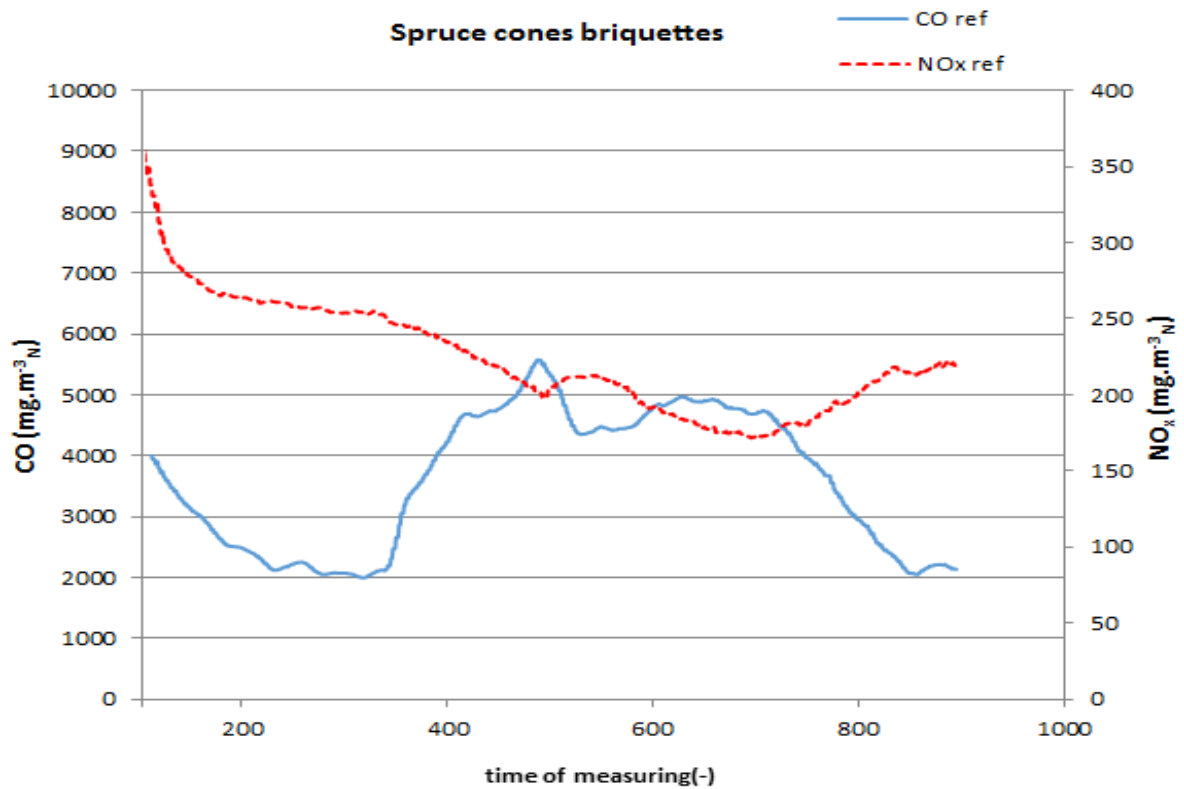
Graph 3. Dependency of CO and NO_x for spruce cones (Source: Author, 2017)

Graph 3 shows measured CO and NO_x of pellets made from Spruce cones.



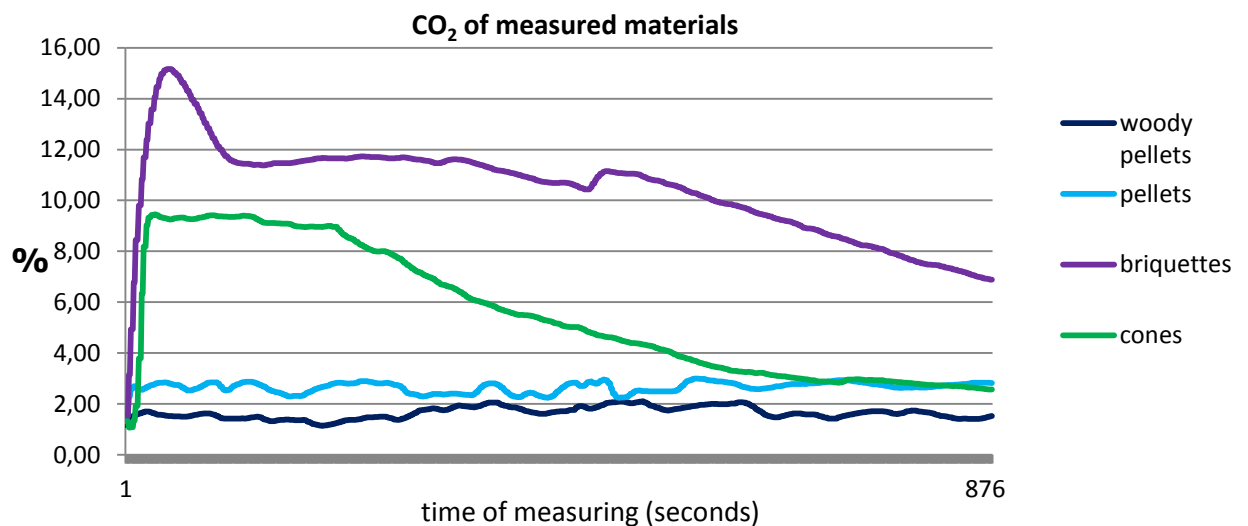
Graph 4. Dependency of CO and NO_x for cones pellets (Source: Author, 2017)

Graph 3 shows results of CO and NO_x of briquettes made from spruce cones.



Graph 5. Dependency of CO and NO_x for cones briquettes (Source: Author, 2017)

Graph 4 shows the relations of CO₂ among all measured materials.



Graph 6. Dependency of CO₂ of all measured materials (Source: Author, 2017)

Progress of measuring of CO₂ for woody pellets, cone pellets, briquettes and cones.

6 Conclusion

Compared to fossil energy resources, waste biomass transformed into solid biofuels is a suitable alternative as a source of energy.

This diploma thesis was focused on using cones from the Norway spruce as a biofuel in form of briquettes and pellets. All research results and other author's findings were presented. Finally the recommendations for further research are given. Due to its physical, mechanical and chemical parameters, the results of the research demonstrated feasibility of the Norway spruce cones for energy purposes. The material had low moisture content and relatively high net calorific value.

The research confirmed first hypothesis. It was possible to produce briquettes and pellets from Norway spruce cones without any additives. Almost every limitations of ISO 17225-3 were achieved. One weak point of the research was the result of mechanical durability of briquettes, which did not fulfil the standard's limitations. This fact could negatively affect manipulation of the material. Other components were fulfilled with positive results such as nitrogen content.

Second hypothesis was partly confirmed. The briquettes made from the Norway spruce cones with different fractions had different effect on mechanical durability. The low fractions had low mechanical durability. The highest fraction proved to be better option but it still did not fulfil the limitations. Therefore it is not recommended due to the difficulties during transportation and manipulating with the material. Pellets made from Norway spruce cones fulfilled limitations of EN ISO 17225-2.

The third hypothesis was partly confirmed. Emissions released during combustion of all three material were measured and Norway spruce cones in its initial form showed the lowest values except for CO₂, because briquettes has the CO₂ content 0.1 % lower than cones. Otherwise values of CO and NO_x were significantly lower. These values were comparable with values measured on wooden pellets. Although the emissions are better with the cones, it is still difficult to use the cones in its initial form due to its large volume, which can complicate transportation and storage.

6.1 Recommendation for future research

Locally it is possible to use cones in its initial form in classical stoves, which can decrease the costs related to process of densification, compared to solid biofuels such as briquettes or pellets.

Research showed that briquettes have positive parameters except of mechanical durability, which can be improved by adjusting the moisture content, use of some binders or possibly briquetting technology with higher pressure or the screw press.

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8 Annexes

Annex 1. Values of measurements of physical, mechanical and chemical properties of briquettes made of Norway spruce cones (Source: Author, 2017).....II

Annex 1. Values of measurements of physical, mechanical and chemical properties of briquettes made of Norway spruce cones (Source: Author, 2017).

Ash content

Number of sample	Weight of empty crucible (g)	Weight of crucible with wet sample (g)	Weight of crucible with ash (g)	Final content of ash (%)
1	21.5148	22.53	21.5355	2.2158
2	26.8695	27.883	26.8893	2.1230
3	26.4795	27.4921	26.498	1.9854

Moisture content

Number of sample	Weight of the empty dish with lid (g)	Mass of the dish with lid and sample before drying (g)	Mass of the dish with lid and sample after drying (g)	Moisture content (%)
1	45,81	238,94	216,49	11,62
2	45,47	191,89	175,17	11,41
3	47,00	183,04	167,45	11,45
4	48,43	250,47	227,52	11,35
5	46,13	205,21	187,08	11,39

CHN content

Number of sample	Measured element (%)		
	N	C	H
1	0.71510	51.166	5.7197
2	0.76512	50.781	5.6708
3	0.75727	51.128	5.7388
4	0.66056	50.580	5.7688

Mechanical durability

Briquettes with different diameters	Weight of briquette before (g)	Weight of briquette after (g)
4 mm	2168.16	1749.82
	2096.07	1707.58
	2179.4	1748.49
	2028.58	1626.2
12mm	2162.98	1762.73
	2179.91	1711.1
	2018.08	1573.55
	2148.19	1708.17
25mm	2026.97	1748.06
	2129.16	1872.16
	2054.23	1747.2
	2058.39	1803.29

Determination of dimensions of pellets

Number of sample	Length (mm)	Diameter (mm)
1	81.24	52.393
2	75.7	52.615
3	81.51	52.183
4	65.45	52.315
5	61.3	52.303
6	51.97	52.433
7	75.73	52.558
8	82.06	51.773
9	83.95	52.628
10	55.43	52.31
11	83.78	52.128
12	74.69	52.793
13	59.78	52.483
14	81.99	52.47
15	81.63	52.52