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Use of cotton plant waste biomass for solid biofuel production

Master's thesis

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DECLARATION

I declare that this presented diploma thesis entitled “Use of cotton plant waste biomass for solid biofuel production” I wrote separately under the supervision of Ing. Bc. Tatiana Ivanova, Ph. D. and used only referred sources of literature which I quote and mention in the list of references. As the author of this thesis I declare that in association with its creation did not infringe the rights of third persons.

Prague, 22nd April 2016

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Bc. Magdaléna Stavjarská

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ABSTRACT

In order to environmental conservation and covering energy demands of today's society there is an increasing effort to use biological waste materials from different production sectors (mostly from agriculture) as a feedstock for the solid biofuel production. One of the promissing waste materials, which is yearly generated in big quantities are cotton residues that could be perspective alternative source of energy in developing countries - the main cotton producers.

The Thesis is devided into two main parts. The teoretical part of present Thesis is primarily focused on analysis of literature information about bioenergy and solid biofuels as well as about cotton plant and its utilization. The practical part presents experimental research dedicated to quality assessment of pellets obtained from cotton resudues through determination of physical, chemical and mechanical properties.

Based on calculation of cotton residues yield and measured gross calorific value the maximum energy potential for cotton biomass was calculated as well. Additionaly, from the data of FAO statistical database the cotton lint yield curve for next 10 years with was estimated.

KEY WORDS

Cotton residues, biofuel standards, pellets, quality tests, calorific value, energy potential

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LIST OF SYMBOLS AND ABBREVIATIONS

CULS	Czech University of Life Sciences Prague
FTA	Faculty of Tropical AgriSciences
FAFNR	Faculty of Agrobiology, Food and Natural Resources
BCRIAE	Bioenergy Center of Research Institute of Agricultural Engineering
ISO	International Organization for Standardization
DC	Developing Country
%	Percent
cm	Centimeter
g	Gramm
h	Hour
kg	Kilogram
mm	Millimeter
t	Ton
J	Joul
MJ	Megajoul
C	Carbon
H	Hydrogen
N	Nitrogen
Cl	Chlorine
S	Sulphur
O	Oxygen
Atm	atmosphere
GCV	Gross Calorific Value
NCV	Net Calorific Value
DU	Mechanical durability
MC	Moisture Content
VM	Volatile Matter
RPM	Revolutions per minute

1 INTRODUCTION

Due to the impending depletion of non-renewable resources and the population growth it is increasingly difficult to obtain energy by environmental friendly way. This fact causes expansion of biofuels production which is considered as a sustainable solution (Malaťák et al., 2010). One of today's favorite biofuels are pellets - solid biofuels produced from biomass in the form of cylinder. The raw material used for pellets' production affect their chemical and physical-mechanical properties, hence the resulting quality (Ivanova, 2012). Because of this it is necessary to test the biomass materials and final pellets according to the applicable standards.

Cotton is one of the most commercial plants grown intentionally for the production of cotton fibers which are fundamental raw material in the textile industry (Hobhouse, 2004). It is grown on large plantations in tropical and subtropical zones (Wendel et al., 2010). Cotton is grown as an annual plant i.e. the harves is once a year. After harvesting of cotton fibres, large amount of cotton restudies remains in the fields, which include cotton stalks, leaves, roots, capsules and a very small portion of fibers. These residues do not almost have any usage and therefore became a waste. Generated large amount of waste within world cotton production, gives a question "What to do with this waste?" (Gemtos and Tsiricoglou, 1999).

The largest proportion of plant biomass waste comes from primary agriculture production, landscape maintenance, wood residues from forestry and industrial production (Coates, 2000). These residues are further processed for the energy purposes into solid biofuel, nowadays especially pellets are popular of the market. Therefore, the focus of this study is to evaluate suitability of cotton waste biomass for pellets production.

2 LITERATURE REVIEW

2.1 BIOMASS

Biomass is defined by the solid fuels standard EN 14588 (2010):

"*An organic material that is plant or animal based, including but not limited to dedicated energy crops, agricultural crops and trees, food, feed and fibre crop residues, aquatic plants, algae, forestry and wood residues, agricultural wastes, processing by-products and other non-fossil organic matters*".

Biomass is defined by the Directive 2001/77/EC as:

"*Biodegradable fraction of products, waste and residues from agriculture (including vegetable and animal substances), forestry and related industries and as well as the biodegradable fraction of industrial and municipal waste*."

Biomass is biodegradable and can be used for combustion or other transformations followed by energy recovery (Weiss et al, 2014).

As transformation may be a mechanical treatment (splitting, crushing) and chemical, thermo-chemical, bio-chemical and mechanical-chemical treatment (pyrolysis, fermentation and pressing) (Stupavský, 2008).

The biomass can be divided into two main groups:

- Waste biomass: agriculture waste, livestock waste, waste from forest management, wood and municipal solid waste
- Biomass from energy crops: energy crops of 1st generation, energy crops of 2nd generation as lignin-cellulosic crops (Weger, 2009)

2.1.1 General characteristics

Specifications of biomass by origin and source:

- wood biomass: biomass from trees, bushes and shrubs
- herbaceous biomass: biomass from plants that have woody stem and which dies by the end of growing season (cereals, grasses, oilseed rape, legumes, flowers)
- fruit biomass: biomass of plant parts, which they are from seeds or contain seeds, berries, pulp

- aquatic biomass: biomass from forestry, arboriculture, agriculture, horticulture and aquaculture
- mixtures and admixtures: mixtures and admixtures thus a combination of the previous groups that mixtures are intentionally mixed biofuels, while admixtures are unintentionally mixed biofuels (Lyčka, 2011; Stelte et al, 2011).

2.1.2 Energy Utilization

Utilization of biomass for energy and compare the biomass and other forms of renewable energy, the biomass has the greatest potential, not only in DCs but in all over the world. Phytoenergetics program implementation is very perspective (Petříková, 2001). The suitable biomass kinds can provide higher energy output. According to Mc Kendry (2002) is utilization of biomass of the word´s energy production in the world about 10 – 14% and covers about 35% of primary energy for cooking and producing heat in DCs (Jakubes et al., 2006). The interim evaluation of the situation the biomass could contribute around 75% of total renewable energy sources (such as solar, wind and hydroelectric power) (Petříková, 2001). Biofuels made from biomass are considered as an environmentally friendly alternative to fossil fuels. Because the price of fossil biofuels grows, an interest about biofuels increases in media and households. It must be produce high quality biofuels competitive with fossil fuels (Celjak, 2008). Biofuel production has advantages and disadvanteges. The great advantage is small amount of ash after burning. Carbon dioxide emissions are not increased during combustion. Disadvantage is a high moisture content, low bulk density, irregular shape and difficulty with transport. Therefore it is important to use the densification process (Váňa, 2001).

2.2 **BIOFUELS**

Biofuels are products which are obtained by adjusting the biomass (Celjak, 2008). Depending on the consistency biofuels are divided into solid, gaseous and liquid. The energy which is contained in biofuels is released during combustion, especially in the form of thermal energy which can be further exploited (Stupavský, 2008).

2.2.1 Liquid Biofuels

Liquid biofuels are biofuels which are transported, stored and prepared for energy use in a liquid state. According to Nigam et al. (2011) there are known three main categories of liquid biofuels:

- alcohol – Bioalcohol, Bioethanol, Biobutanol
- Bio-oil, Biodiesel – transesterification oils and fats
- liquefied gaseous biofuels – Fisher-Trops synthesis

According to Stupavský (2008) there are three main examples of liquid biofuels:

Unrefined oil

Pressed oil is obtained from oilseeds. Vegetable oil has higher density and higher viscosity if compare with diesel. The viscosity is needed for using in existing engines. This can be achieved in two ways:

- chemically: RME Production Plant = Rapeseed Methyl Ester
- heat: increase fluidity by heating the vegetable oil

Biodiesel

Biodiesel is used as a petroleum substitute for diesel engines (Subramanian et al., 2005). The biodiesel is known as low-molecular weight higher fatty acid esters with low molecular weight alcohol: FAME (Fatty Acid Methyl Ester). Biodiesel is essentially waste-free technology because all by-products can be further used. Oil crops are a raw material for production (a renewable resource).

Bioethanol

Bioethanol is ethanol which is produced by the alcoholic fermentation of biomass which is used as biofuel. It is made usually from plants containing higher amount of starch and other carbohydrates. The most commonly used raw materials are corn, wheat and potatoes but also sugar cane and sugar beet (Demirbas, 2005). While plants containing sugar are fermented directly, plants containing starch must be starch enzymatically converted to sugar. Produced bioethanol can be used directly in internal combustion engines as fuel.

2.2.2 Gaseous Biofuels

Biogas is a gaseous mixture of methane and carbon dioxide (CH4 + CO2). Biogas is used as the gaseous products of anaerobic methane fermentation of organic substances. It is produced through decomposition of biomass without air – anaerobic digestion, biomethanization and gasification. It contains energetically valuable methane and therefore the calorific value is between 20 and 25 MJ/m^3. Biogas is mostly used to produce electricity and heat (sewage, biogas stations) as well as a propellant (Herrmann et al., 2016).

Wood gas is synthesis gaseous fuel based on carbon monoxide (CO + H2). It is produced by gasification, it means by incomplete combustion of plant biomass of other carbon source with limited air access. For gasification can be used wood chips, sawdust, wood or grass pellets, wood or other coal and other similar combustible materials (Weiss, 2014).

The use of biogas for energy is more efficient than direct combustion but it requires greater investments. Therefore combustion is the most widespread. It is easier to buy the biomass boiler and burn it than to build a biogas station (Janíček, 2009).

2.2.3 Solid Biofuels

EN ISO 16559: Solid biofuels – Terminology, definitions and descriptions. This standard specifies terms and definitions for solid biofuels from woody, herbaceous, fruity and aquatic biomass from forestry, arboriculture, agriculture, horticulture and aquaculture. Materials originating from various recycling processes for end of life do not fall into scope of this standard (EN, 2015). Solid biofuels are biofuels which are stored, transported and prepared in solid form. The term of solid biofuels includes firewood, wood chips, straw, hay, sawdust, briquettes, pellets (Stupavský, 2010).

Wood chips is a wood which is crushed to a size of 3 to 250 mm. Wood chips are obtained by processing of waste, mining and wood processing. It is a cheap source of biofuel and can be divided into green, brown and white. Green chips include a green parts it means pine needles and leaves. It contains also part of the small branches because it is obtained from forest residues after harvest. Moisture is high; it means that the material is fresh. Brown chips are obtained from the residual part of the tree trunk and the main component is a bark because the wood was not stripped of bark before

processing. White chips are obtained from debarked wood and are mainly used for the production of particle board (Maker, 2004).

Wood chip is a renewable energy source which is not dried or pressed, it means that it is without added energy and therefore the price is lower. The calorific value is measured in the range from 8 to 15 MJ/kg and moisture is different by type of wood from 15 to 50% (Stupavský, 2010).

The briquettes are manufactured by pressing e.g. dry wood dust, sawdust, bark, shavings of plant residues in the form of cylinders, prisms or hexahedron, a diameter of 40 to 100 mm and 300 mm length. Varieties of shapes are shown in the Figure 1 below.

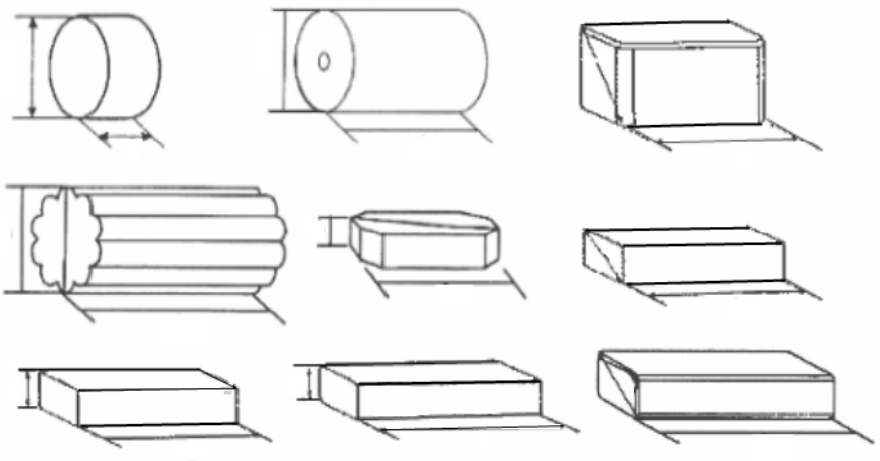


Figure 1: Variety of briquettes shapes

Source: Standard EN 15202-2 (2009)

Briquettes are made from wood, bark, straw and energy crops or briquettes produced from mixture of these materials. They can have different color depending on the type of biomass, the quality of the raw material affected by moisture or bark mixture and the applied technological process of production. Wood briquettes have due to its density, which is about 1,000 to 1,200 kg/m^3, stable and low moisture content (water content is usually 8%) and low ash content (about 1 to 3%). Briquettes are made by strong compression which is called densification or simply briquetting (Plíštil, 2004). The major meaning of densification is the utilization of mechanical pressure to reduce the volume of raw material and its conversion to a solid form which has excellent properties in terms of transportation and handling than the bulky original material (Shaw, 2008).

2.3 **PELLETS**

Pellets are relatively new fuel for home heating and to heat the water and to produce electrical energy. They are highly versatile product based on wood and other material. It is made especially for competition with fossil fuels in operator comfort, performance and price. Technology of pellets was established in 80s of the 20th century in Sweden as practical effective way how to utilize wood waste from wood processing industry. Since that time pellets are cheaper, carbon-free and appropriate alternative fuel for fuels like coal and oil for heating human households. More and more people use the combined heating systems where one part from energy produced is used to heat production and second part will be sold back to the network (Petříková, 2007).

2.3.1 General characteristics

Pellets are granules generally with circular cross-section which is produced in extrusion presses. Extrusion under pressure leads to a high density of fuel which is desirable due to a smallest volume per unit energy content. They have a high calorific value, low ash content and low water content. They are impact resistant; they have low demands on storage space and especially allow automation of the combustion process. The most common material for their production is wood. The actual trend is the development of pellet production from agriculture residues (Bufka, 2007).

2.3.1.1 Basic requirements

Requirements for quality of fuel pellets from plant biomass are determined by standard EN ISO 17225 (2015) which are shown in Table 1.

Table 1: Specific requirements for pellets

Calorific value in d.b.	min 16 MJ/kg
Total water content	max 10%
Ash content in d.b.	max 6%
Pellet density	min 1.12 kg/dm^3
Abrasion from mechanical durability	max 2.3%

Source: Standard EN ISO 17225 (2015)

Fuel quality requirements for stationary sources of air protection are shown in Table 2. Requirements on emission limits for small sources of air pollution by Malaťák et al. (2010) are shown in Table 3.

Table 2: The contents of elements related to in d.b.

Total nitrogen (N)	max 0.9 %
Total sulfur (S)	max 0.15 %
Total chlorine (Cl)	max 0.18 %
Maximum proportion of additives	to 6%

Source: Standard EN ISO 17225 (2015)

Table 3: Emission limits of fuel combustion in small pollution sources

	mg/kWh	**mg/m^3**
CO	4,500	2,000
NOx*	550	250
SO2	130	60
CxHy	130	60
Solid pollutants	420	190

Source: Malaťák et al. (2010)

*Mass concentration NOx is related to NO2. Emission of mg/m^3 is related to dry combustion gases, atmospheric pressure and temperature 0°C and the reference oxygen content 11%.

2.3.2 Pellets Production

Pelleting is a mechanical modification of material by compaction with high pressures (Shaw, 2008).

The material must be dried, crushed and cut short. Required moisture is 8 to 15 % in straw cereals, oilseeds, grasses and energy crops. These crops are compacted into the form of cylinders on average from 6 to 25 mm (exceptionally prisms with the edge to 40 mm), length to 50 mm with a specific bulk density 0.9 to 1.2 (1.4) kg/m^3. The compaction pressure is usually from 80 to 140 MPa and a pressing temperature is about 100°C. Average bulk density is from 550 to 600 kg/m3. Tha calorific value is usually

from 15 to 19 MJ/kg, but can vary. The ash content in the dry matter is 3 to 8 % (Koloničný et al., 2011).

The production process of fuel pellets according to Andert et al. (2006):

1. Collection of raw materials – maximum distance 50 km is recommended.
2. Sorting of raw materials – magnetic separator (metals), disk sorter (stones).
3. Drying – the energy consuming process is required for all kinds of wood raw materials with the moisture content greater than 12 and this process is used especially for drying in the field.
4. Homogenisations of raw material – all culms intended to pelletizing process are processed by crusher.
5. Pelleting – presses with membrane matrix is used for lower outputs, the higher outputs can be achieved by an annual matrix (Vícha and Miroslav, 2004).
6. Cooling and storage of product – pellets reach a temperature 90 to 120°C at the exit from press. Cooling is a process where pellets obtain the strength and resistance against abrasion. For cooling is mostly used counterflow cooler which decreases the temperature of the pellets from 30 to 35°C. Waste heat can be used for pre-drying feedstock.
7. Packing and shipping - pellets are filled into bags (15 to 25 kg), big bags (up to 1000 kg), tanks or in bulk.

2.3.2.1 Densification Process

Densification is a technological process and it is used for improving the conditions. Densification is important for avoiding possible loses and damaging of the densified products. It reduces costs due to more effective and economical friendly production. It reduces problems conected to handling, transportation, utilization and storage. Production of solid biofuels includes processes such as a collection, preparation, drying, grinding, mixing and pressing – compaction (Ivanova et al., 2010).

Other definition of densification is the utilization of some form of mechanical pressure to reduce the volume of raw material and its conversion to a solid form. It is better for manipulation and storage (Shaw, 2008).

Eriksson and Prior (1996) say that technique of densification was used in DCs also all over the world. It is utilization of various species residues as an energy source.

The first mention of densification was in USA in 1880 and it was conected with topic of production of animal feed.

Densification process reduces the bulk density (40 – 200 kg/m^3 to 600 – 800 kg/m^3). When pellets leave the machine they are separated on the end of the pellet production process (Shaw, 2008).

Success of this process is measured by the level of durability – test of force and stress. It imitates the process of manipulation, transportation and storage. Factors exist that influence several properties of raw material, such as moisture content, particle size and content of fat, lignin and fibre. Other factors are related to the steam preheating or addition of the binders. Affect of the final product quality can have the type of equipment (type of roll press and forming pressure). And final group of factors are the post-production conditions like cooling and air humidity. All of these factors have impact on the final product. It is necessary to optimize processes and all of its components to the best result. The process of densification is divided into two different groups. First group is hot and there is high-pressure densification (100 MPa) on the contrary the second is cold and low-pressure densification (0.2 to 5 MPa) (Kaliyan and Morey, 2009).

The main disadvantage of densification is high investment. Other disadvantages are excessive smoke production, poor ignitability and last but not least high energy input into the process (Shaw, 2008).

2.3.2.2 Pellet presses

According to Sladký et al. (2002) the most famous are usually two types of pellet presses:

- with annular matrix (vertical, horizontal)
- with membrane horizontal matrix

Both types use the matrix drive or pusher roller drive.

Press with an annular matrix operates on the principle of a disc, in whose inner part there are two or more rollers that push the material through holes. Performance of these presses is 3 to 5 tons per hour.

Annular matrixes have good pressing characteristics. Pressing pressure is gradually increases and in the narrowest point there is a maximum value. Pressing

process takes place for about 5% of the circumference of the ring (Ochodek et al., 2006). The principle is shown in the Figure 2 below.

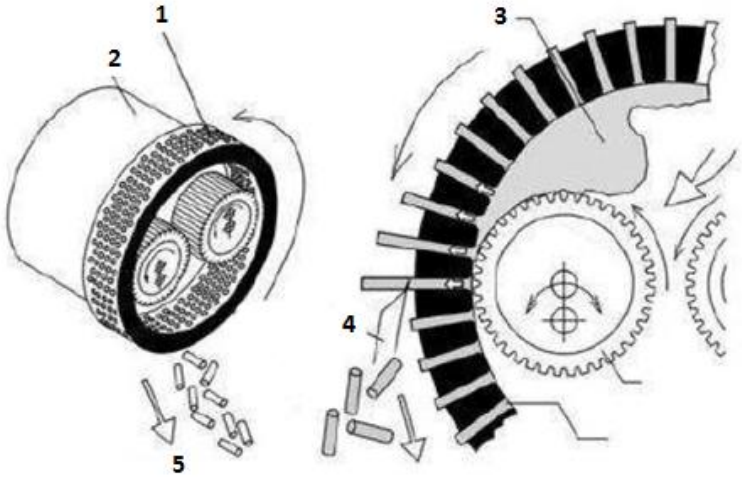


Figure 2: The principle of granulating press with an annular matrix

where:

1 – matrix;

2 – transmission;

3 – crushed material;

4 – cutting knife;

5 – pellets.

Source: Šooš (2000)

The press with membrane horizontal matrix (Figure 3) works on rotating rollers which are rolled over the circular matrix and disc matrix. Rollers extrude the raw material downwardly through holes in the matrix (Ochodek, 2006).

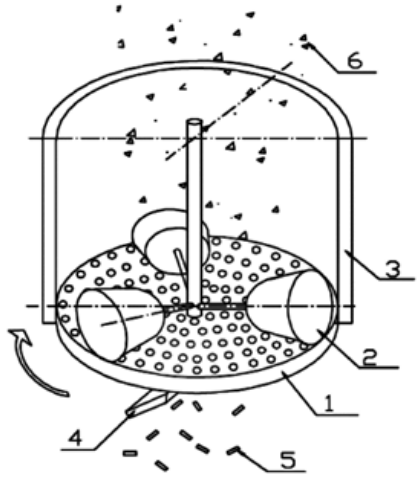


Figure 3: The principle of granulating press with membrane horizontal matrix

Where: 1 – flat matrix; 2 – spherical pulleys; 3 – pressing chamber; 4 – knife; 5 – pellets; 6 – crushed material.

Source: Šooš (2000)

These methods are differed from each other in particular claims which are based on economic and material requirements of manufacturer pellets. While presses with membrane matrix can produce up to 2 tons of pellets per hour, presses with annular matrix can produce up to 5 tons of pellets per hour (Šooš, 2000).

2.3.2.3 Pelet stoves

Pelet stoves are used for heating of individual rooms, small apartments or low-energy houses. Heat transfer is occurs by radiation and in the case of the involvement of the fan also in the air flow. Thermal power of pellet stoves is usually in range from 6 to 10 kW and controlled is manually or automatically via a thermostat. Pellet stoves are simply and easy to operate stoves that are also popular for aesthetics of a burning fire in the room (Stupavský, 2010). The main advantage is automatic transport of fuel between the warehouse and the boiler itself using an automatic conveyor. The boiler can ingnite and off itself. The thermostat controls temperature according to desice of owner of the house (Stupavský, 2012).

2.4 PELLET QUALITY CONTROL

The quality of the produced pellets is depended on the method of production and the quality and appropriate preparation of raw material. There exist a few technical specifications that determine and evaluate the quality of the resulting pellets. The European Committee for Standardization (CEN) determines the standards for defining, observing and testing the densification products.

2.4.1 Factors affecting pellet quality

Important standard for production of solid biofuels is standard: EN ISO 17225-1: Solid biofuels – Fuel specifications and classes – Part 1: General requirements (Ivanova, 2012).

Quality of solid biofuels is determined by a number of characteristics which is ascribed a different meaning. It is necessary divided into three parts: pre-production (chemical composition and physical–mechanical properties), production and post-production properties (Ivanova, 2012). Construction and design of reverse technology for energy recovery must be based on these characteristics (Jevič and Hutla, 2008; Adapa et al., 2013).

Solid biofuels are characterized by their fluctuating quality. The chemical composition is affected in the grow phase of the plant. The physical properties of the produced product are affected in the processing phase. Technical specifications are only

concerned with fuel. For ensuring efficient use of solid biofuels is very important to consider the relationship between fuel and equipment for combustion, gasification or pyrolysis. Therefore and users must supervise the technology compatibility conversion of solid biofuels. It is needed to achieve operational optimized process of applications (Jevič and Hutla, 2008).

2.5.1.1. Pre–production factors: (related to the parameters of used raw material, described overleaf)

Chemical composition – Ash fusibility and content, Content of Cl, N, S, H, C, O, heavy metals, Volatile matter content.

Physical–mechanical properties – Moisture content, Calorific value, Density, Particle size, Dimensions (Ivanova, 2012).

2.5.1.2. Production factors: (related to densification process and equipment)

Pressing temperature - Strength of pellets are related to using of pressing temperature because it influences excretion of the natural binder lignin during the pressing. Optimal pressing temperature is equal to 150 °C.

Compacting pressure - With increasing the compacting pressure mechanical quality of the pellets increases too. Pressure used during densification process enables different binding mechanisms (Chin and Siddiqui, 2000).

2.5.1.3. Post–production factors: (related to the conditions of storage and handling)

Storage conditions - Storage conditions are influenced by air temperature, relative humidity and other weather changes and mode of packaging. With the increasing of air temperature and air humidity, mechanical quality of pellets is decreased. When the temperature and humidity were constant there was no effect on quality (Adapa et al., 2013).

2.4.2 Standards

All products of biomass which are produced for the purpose of combustion must be tested. It must be known their quality and composition. It is important to conduct correct sampling, transport and storage. Samplings are performed according to the EN

14778-1. Important parameters for the fuels quality according to appropriate standards are listed below:

- EN 15210 – 2 Solid biofuels: Determination of mechanical durability of pellets and briquettes - Part 1: Pellets
- EN ISO 16559 Solid biofuels – Terminology, definitions and descriptions
- EN 14780 Solid biofuels – Methods for sample preparation
- EN ISO 18134 – 2 Solid biofuels – Determination of moisture content – Oven dry method – Part 2: Total moisture – Simplified method
- EN ISO 18134 – 3 Solid biofuels – Determination of moisture content – Oven dry method – Part 3: Moisture in general analysis sample
- EN 14775 Solid biofuels – Determination of ash content
- EN ISO 16968 Solid biofuels – Determination of minor elements – As, Cd, Cr, Cu, Pb, Hg , Ni, Zn
- EN 15148 Solid biofuels – Determination of the content of volatile matter
- EN 14918 Solid biofuels – Determination of calorific value
- EN 15103 Solid biofuels – Determination of bulk density
- EN ISO 16994 Solid biofuels – Determination of total content of sulphur and chlorine
- EN ISO 16948 Solid biofuels – Determination of total content of carbon, hydrogen and nitrogen

Moisture content

The moisture content significantly affects the calorific value. Moisture is evaporated during combustion and gasification. Anhydrous biomass is almost not occur in nature. The moisture content influences sustainability storage. When moisture content is over 16% it starts biological transformation processes or degradation. These are linked to looses and changing the composition of the fuel at the same time (lignin content, increasing the ash content, etc.). Especially for herbal biofuels with increasing moisture there is a risk of a fire due to respiration mesophilic and then thermophilic bacteria that are able to heat the material to the temperature about 70°C. The

temperature above 100°C leads to a chemical oxidation which can lead to spontaneous combustion (Kotlánová, 2010).

The ideal moisture content ranges between 5 – 10 %. Values which do not belong within this range can causes decreasing of mechanical quality of the pellets (Bohm and Hartmann, 2005).

Ash content

Ash content is an anhydrous state, the weight of anorganic residues remaining after the combustion of the fuel under specifies conditions expressed as a mass fraction in % of the dry fuel (Kotlánová, 2010). Fuels with low ash content are preferable. Ash contains elements which influence the choice of an appropriate combustion and process control technology. Ash forming elements are divided into two groups, the major and the volatile elements. Ashes from combustion of solid biofuels contain interesting amount of plant nutrients which find its utilization in the soil. Especially for content of phosphorus, ash can be used as a fertilizer in agriculture as an industrial by-product which is often recognized as a solid waste. When the biomass ash is returned to the locations where the biomass was harvested, it is considered as the one of the best sustainable option because it can bring back the nutrients to the original soils and hence closes mineral recycles (Obernberger et al., 2006).

Heavy metals

Large part of heavy metals remains in the ash and reduce its utility as fertilizer. Generaly it determined following eight elements: Arsen (As), Cadmium (Cd), Chromium (Cr), Cooper (Cu), Lead (Pb), Mercury (Hg), Nickel (Ni), Zinc (Zn) (Kotlánová, 2010).

They are located in the particles of ash and in fly ash. Its removal can be minimized the risk of contamination of crops during fertilizing. Contents of heavy metals are important of environmental. Metals and other elements can be input to solid biofuels from preserving chemicals, colors, used mineral oils (during storage, transport and handling), used tools or machines, additives, plastic or chemical ingredients (Obernberger et al., 2006).

Volatile matter content

High proportion of volatile matter content may effect emissions. Sample is annealed in a muffle furnace without air access at high temperature to escape amount of volatile matter after carbonization. It is a part of total combustibles contained in the fuel. It looks like a flame. The content of VM depends on the geological age of the fuel. When the fuel is geological younger, VM content is higher and the ignition is more easily than older fuel. The lowest content has a black coal opposite of turf and wood.
VM significantly assists to fuel ignition and stabilizes combustion processes (Naik et al., 2010).

Calorific value

Net calorific value (NCV) is influenced mainly by water content (more than by type of biomass). The calorific value of dry matter influences the composition of matter. It is a major parameter for biofuels. Net calorific value is defined as amount of energy released by a unit weight produced by the complete combustion of material. Higher calorific value corresponds to higher ability of pellet burning (Obernberger et al., 2006). Net calorific value is calculated from gross calorific value. GCV is the heat released by the complete combustion of 1kg o material that all the water vapor in the flue gases condenses into liquid form. For this reason each value of gross calorific value is higher than NCV (Kotlánová, 2010).

Bulk density

Bulk density is important parameter for storage and transportation of fuel and influences moisture content. With NCV determines the energy density. It is calculated by the quotient of mass of a sample material filled into a measuring container and its known volume (Bohm and Hartmann, 2005).

Sulfur and Chlorine

It is of primary importance for the sulfur oxides. During combustion the sulfur is transferred into gaseous phase, it is created SO_2 and SO_3. SO_2 in gaseous form and the sulfate escape into the surroundings. Sulfur may also increase the risk of corrosion (Obernberger et al., 2006).

The chlorine content is an important parameter with regard to the creation of HCl and its associated corrosion. These effects are used with alkali metal and SO_2 at the surface of the heat exchanger and with other metal parts. Increased content of chlorine can lead to a reduction in the softening temperature of ashes (Kotlánová, 2010).

Nitrogen, carbon and hydrogen

The nitrogen content affects the nitrogen oxide production. Almost all this element is transferred to the gaseous phase during combustion. Oxidation of nitrogen, which is contained in the fuel, is the most important mechanism for the formation of NO_x from biomass. Carbon and hydrogen are oxidized during combustion by exotermic reactions and positively contributes the GCV. Content of Carbon is lower for herbaceous biofuels than wood fuel which explains lower GCV of herbaceous fuels. Content of hydrogen influences NCV due to formation of water (Obernberger et al., 2006).

Mechanical durability

One of the most important investigated mechanical properties of solid fuels. DU is a qualitative indicator that describes the cohesion during handling (Ivanova, 2012). Kotlánová (2010) says that this test is characteristic only for pellets and briquettes. Especially for pellets, because pellets are fed into the combustion devices. Secondly expressed as abrasion i.e. material which is separated from the biofuel and then as mechanical resistance i.e. how large sample will remain after the test. This property describes the ability of pellets to resist to externam environment in some cases several phase distribution process. This process begins from the production line where pellets have to be stored, then put in the bags, move to a pallet and transfer into a warehouse and then move into the tank in the boiler.

Particle size

Particle size is one of the most important factors that influence the final pellet quality. During decreasing of particle size is increased mechanical quality of pellets (MacBain, 1966).

2.5 **COTTON**

The mention of the term “cotton” almost everyone think about textile industry and production all kinds of clothes. The cotton is grown on huge plantation and after collection of white fiber tufts is created a problem which is called “what about waste of cotton”? This is the reason why it is necessary to solve this (Hobhouse, 2004).

2.5.1 General characteristics

Gossypium, cotton in English. This is a genus belonging to the family of the mallow (*Malvaceae*) and includes about 40 species (Hobhouse, 2004). The genus *Gossypium* has a long history of taxonomic study. The genus includes 4 domesticated species: *G. hirsutum*, *G. barbadense*, *G. arboreum*, *G. herbaceum*. *Gossypium hirsutum* is widespread and constitutes 90 % of all cotton production (Vymazal, 2012; Wendel at al, 2010). It is an annual plant mainly growing on plantation of the tropics, subtropics and the warmest temperature region (Figure 4). Flower colors are white, yellow (Figure 5), brownish and pinkish. After flowering there remains a capsule with 3 to 5 chambers. 2 to 11 seeds are in 1 chambre. Each seed being dotted two kinds of fibre: long fiber called lint and shorter fiber called linter. These fibers constitute so called tufts which are prepare for harvesting (Pospíšil and Hráchová, 1989).

Figure 4: Cotton plantation
Source: Baker (2015)

Figure 5: Yellow flower of cotton
Source: Galapagos (2013)

2.5.2 Origin of cotton

Cotton origin is unclear. The authors are fundamentally diverging in their findings. One opinion is that the oldest finding of cotton is older than 7,000 years and came from Mexican caves (Wendel et al., 2010). Brink and Escobin (2003) argue that the first cotton was discovered in 2,000 BC in Pakistan. Pospíšil and Hráchová (1989) refer that a country of origin is China. Hobhouse (2004) states that cotton came from Africa.

In America there was found several different kinds of cotton. In Mexico there was handmade cotton industry. The price of raw cotton was high, processing costs as well, but the traders had positive incomes because the finished price of produced goods had large margin. Interest in cotton products kept increasing and everyone wanted to earn more. Cotton began to grow on American soil and therefore import was decreased. The management of the cotton field required a lot of labor force. Everything had to be done manually because the mechanization was missing. The result was extension of slavery. End of the 18th century the graduate of physics department Eli Whitney constructed a ginning machine, which harvested cotton and replace a lot of labor force. Before he patented it, his idea was stolen and quickly spread among farmers. Therefore the costs of farm were reduced (Hobhouse, 2004).

2.5.3 Harvest

Figure 6: Uneven ripening
Source: Moliver (2013)

Cotton harvest is problematic harvest because tufts do not ripen together in the same time (Figure 6). Hand collection is more advantageous because only ripest capsules are collected. But it must be harvested from 4 to 5 times until all the capsules will be harvested. Although this process is slow, it is better because it has higher yield and better quality cotton (Wendel et al., 2010). For mechanized harvesting is one big disadvantage that the machine does not distinguish ripe from unripe capsules. Therefore it often used chemical sprays. Defoliants after which the leaves fall down and they cause the uniform ripening or dessicants which cause drying tissues, opening capsules and speeding up ripening (Valíček, 1989; Ball and Glover, 1999).

In America there is very popular machine, cotton harvester so called stripper, John Deere brand, type 7460 and working width is 8.02 m (Figure 7). Disadvantages are a high contamination of dry plant parts and not ripen capsules (Deere, 2013).

Figure 7: Stripper John Deere 7460

Source: Deere (2013)

2.5.4 Production and Utilization

It exists several studies which focused on cotton waste utilization as livestock feed (Poore and Rogers, 1995), paper production (Ververis, 2004), composting (Ayers, 1997) and energy production (Coates, 2000; Holt et al., 2004).

Cotton is processed for energy purposes mainly in the form of heat. In solid, liquid and gaseous form (Holt et al., 2004).

In Greece there is a progress that in textile industry fosil fuels could be replace for biomass, because cotton stalks help to reduce greenhouse gas emissions. Local available quality of ginning waste from cotton is considered to be sustainable and viable bioenergy. A power 5 MW could replace 52 % of heavy fuel oil. Invested costs were estimated at € 1,678,746 (Zabaniotou et al, 2010).

Gravalos et al. (2010) indicates that individual parts of cotton are suitable for energy production. With his team he researched net calorific value of all parts of cotton in Greece. He shows that individual parts have differences between them, only the root and main stalk have approximately the same values of NCV, in the range from 17.64 – 17.8 MJ/kg. Seeds have the highest NCV 22.5 MJ/kg to 23 MJ/kg due to higher fat content and hence the higher amount of energy. Comparation with other authors is shown in Table 4.

Table 4: Average yield of cotton stalks according to different authors

Author	Yield of cotton stalks
Gravalos et al., 2010	2 – 3 t/ha
Sumner et al., 1986	3 – 7 t/ha
Coates, 1997	2.3 – 5.7 t/ha
Gemtos and Tsiricoglou, 1999	1.5 – 4.1 t/ha

Source: Gravalos et al. (2010), Sumner et al. (1986), Coates (1997), Gemtos and Tsiricoglou (1999)

Average yield of these stated values is 3.6 t/ha.

The cotton stalk is underused part of plant and it is a waste. One possibility is to drying for hay. The most succesfull method is cutting aboveground plants parts with using traditional machines, in this case moving machine with piston knives and packaging of hay into large round bales. This harvest operation is energy efficient because it is obtained 51.8 GJ/ha of gross energy. After cutting, raking and pressing remain 35.5 GJ/ha of net energy and this among could be produced each year (Gemtos and Tsiricoglou, 1999).

Briquettes and pelles can be made from stalks or at least partially include into production. E.g. into briquettes that are produced from the shells of pecans is added recycled paper. And this paper could be substitute by cotton stalk. Quality should remain relatively the same and despite initial costs would have to invest; it would mean an overall reduction in material costs because paper parts are expensive (Coates, 1997).

3 OBJECTIVES

3.1 MAIN OBJECTIVE

The main objective of the present thesis was to evaluate the quality of pellets made of cotton residues and to analyze the suitability of using cotton for the production of solid biofuels.

Stated main objective was fulfilled by achievement of two stated specific objectives described below.

3.2 SPECIFIC OBJECTIVES

First specific objective

First specific objective was to evaluate chemical, physical and mechanical properties of pellets produced of cotton residues in order to access their final quality.

Second specific objective

Second specific objective was to calculate maximum energy potential based on the yield of the cotton residues and gross calorific value.

4 METHODOLOGY

Methodology of this thesis contains as well as all scientific papers theoretical and practical part. Theoretical research consisted of secondary data extracted mainly from scientific articles. ScienceDirect, Scopus, Web of Science and Biom databases were used for data searching by key words: Cotton residues, biofuel standards, pellets, quality tests, energy potential. The overwhelming majority of impact articles were in English and Czech languages. Second part of this thesis was practical research which created prevalent part. Methodology of used research is described below.

4.1 MATERIAL AND SAMPLES

The material for all types of tests was brought from north part of Tajikistan, Sughd region (Figure 8). Cotton was harvested by manually in November 2015, imported in December 2015 and 10 kg of material was available.

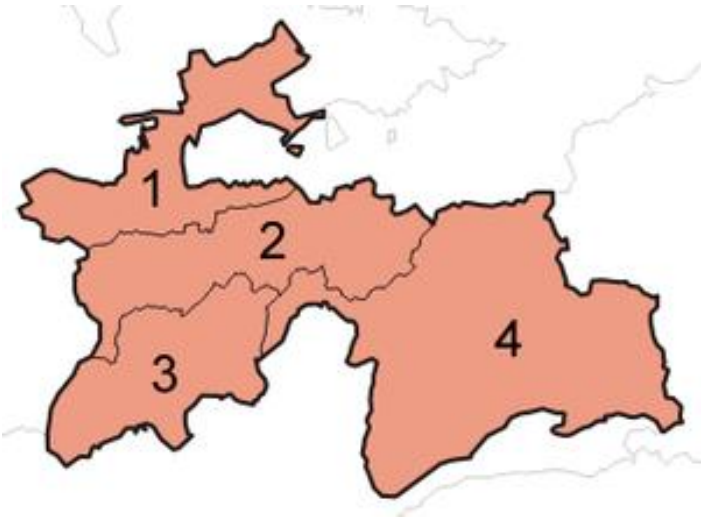


Figure 8: Map of Tajikistan regions

1 – Sughd
2 – Region of republican subordination
3 – Khatlon
4 – Gorno-Badakhshan

Source: BBC (2015)

4.1.1 Material characteristics

For all types of tests it was needed to prepare the relevant sample from cotton plant. Average weight of 1 plant was 60 g and 120 cm length. Material that was available included parts of cotton root, cotton stalk, cotton leaves, capsules and a very small portion of fibers, called together cotton residues.

4.2 PREPARATION OF PELLET SAMPLE

Cotton residues were imported only with a little modification using a simple cut only by reason of easier transportation. For further tests it had to be adjusted.

4.2.1 Material crushing

For the first adjustment of cutting material it was used shredder AL-KO New Tec 2400R (Figure 9). Material was cut to the pieces of 5 to 10 cm. After that it was used Hammer mill 9FQ – 40C (Figure 10). The grinding mill energy input is 5.5 kW. Material was grinded through 8 mm screen. Both of these machines are available on the premises of Technical Faculty laboratory.

Figure 9: AL-KO New Tec 2400R shredder

Source: Author (2016)

Figure 10: Grinding (Hammer mill 9FQ – 40C) in process

Source: Author (2016)

4.2.2 Production of pellets

Production of pellets was carried out on simple Pellet Press which is located at Bioenergy Center of Research Institute at Agricultural Engineering.

Crushed cotton residues were inserted into the container of the pelleting press and through horizontal matrix pellets were produced by compression. For producing it was used matrix with diameter of holes 6 mm.

4.3 **PREPARATION OF ANALYTICAL SAMPLE**

Preparation of analytical sample was carried out at Laboratory of biofuels at FTA. It was used average sample of cotton plant (Figure 11; left) and it was cut into 1-3 cm pieces by scissors (Figure 12; right) and then it was shredded into smaller pieces in the grinder IKA (Figure 13) and afterwards ground to pieces less than 1 mm in the laboratory at FTA by Cutter Mill (Figure 14). The last part was to paste analytic sample into a glass bowl with a lid for protection to water absorption (Figure 15).

Figure 11: The average cotton plant

Source: Author (2016)

Figure 12: The first part of milling process – by scissors

Source: Author (2016)

Figure 13: Grinder IKA MF 10 basic

Source: Author (2016)

Figure 14: Cutter Mill – GRINDOMIX GM 100

Source: Author (2016)

Figure 15: Prepared analytical sample

Source: Author (2016)

4.3.1 Storage conditions

The milled samples (Figure 15) were stored in laboratory at FTA of CULS in desiccators of various sizes (Figure 16).

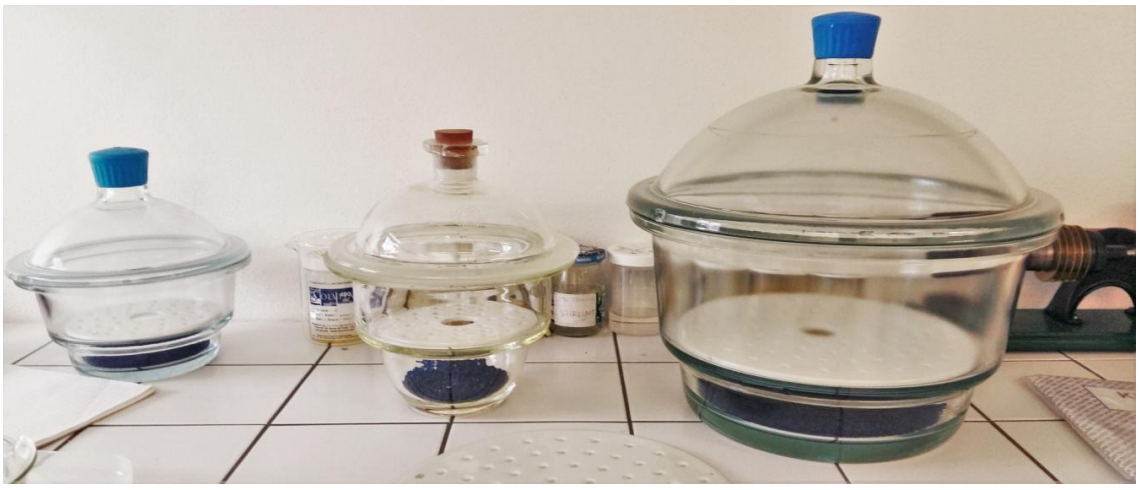


Figure 16: Used desiccators for storage of samples

Source: Author (2016)

4.4 EXPERIMENTAL METHODS

A comprehensive set consisting of the main chemical, physical and mechanical tests was used for experimental research of this paper.

Experimental tests were carried out in the laboratories at Bioenergy Centre of Research Institute at Agricultural Engineering, in Laboratory of biofuels at Faculty of Tropical AgriSciences of CULS and in Laboratory of Environmental Chemistry of CULS. These methods of quality testing are described in following pages.

4.4.1 Moisture Content Test (MC)

Determination of moisture content was carried out according to European Standard EN ISO 18134-2 (2016): Solid biofuels – Determination in moisture content – Oven dry method: Part 2:Total moisture – Simplified method and Part 3: Moisture in general analysis sample.The MC of the biomass was measured by drying of dryer Memmert 100-800.

Determination of MC for analysis sample was conducted according to Part 3: There was prepared 2 analytical samples with an approximate weight 1g and placed in the dryer at 105°C until the weight remained the same two consecutive interim measurements, means that samples are weighed before, during and after drying.

Determination of MC for pellet sample was performed according to Part 3: There was prepared 2 pellet samples with an approximate weight 300 g and placed in the dryer at 105°C for the same process which is described above.

Formula for determination of moisture content:

$$\boldsymbol{w = \frac{m_2 - m_3}{m_2 - m_1} \times 100}$$

where:

w = moisture content (%)

m_1 = mass of empty crucible (g)

m_2 = mass of crucible with sample before drying (g)

m_3 = mass of crucible with sample after drying (g)

The result shall be calculated for two decimal places and rounded to 0.1 %.

4.4.2 Ash Content Test (AC)

Determination of ash content was carried out according to European Standard EN 14775 (2009): Solid biofuels – Determination of ash content.

Empty porcelain crucibles were in a muffle furnace for 60 minutes at 550°C. After removing the crucibles were cooled the heat-resistant plate for 5 – 10 minutes. Than the weighed samples were inserted with an approximate weight 1g into cold muffle furnace and then furnace temperature was increased uniformly to 250°C for 30 minutes. The samples stayed at this temperature for 60 minutes inside to lose the volatile substances before ignition. A steady increase in the furnace temperature continued at 550°C for an additional 30 minutes. At this temperature was persisted for 120 minutes.

Porcelain crucibles were removed from the furnace and it was cooled on heat – resistant plate for 10 minutes. It was transferred to a desiccator without desiccant and cooled to room temperature. After reaching room temperature the crucible with ash was weighed with an accuracy of 0.1 mg and the weigh was recorded.

Formula for determination of ash content:

$$\boldsymbol{A_c = \frac{(m_3 - m_1)}{(m_2 - m_1)} \times 100 \times \frac{100}{100 - M_{ad}}}$$

where:

A_c = Ash content (%)

m_1 = mass of empty crucible (g)

m_2 = mass of crucible with sample (g)

m_3 = mass of crucible with ash (g)

M_{ad} = water content in a sample expressed as a mass fraction (%)

The result has to be recorded as the average of two determinations with an accuracy of 0.1%.

4.4.3 Gross Calorific Value Test (GCV)

Determination of gross calorific value was carried out according to the applicable Standard EN 14918 (2009): Solid biofuels – Determination of gross calorific value and net calorific value. This standard describes a method for determining the calorific value of solid biofuel at constant volume and the reference temperature of 25°C in the calorimeter with a pressure vessel which is calibrated with combusting of certified benzoic acid. The result of this method is gross calorific value of the analytical sample at constant volume with the total water from the combustion gases in the liquid state.

Figure 17: Sample in combustion dish
Source: Author (2013)

Samples of crushed cotton with a weigh of 0.5 g to 0.6 g were inserted into a combustion dish (Figure 17) and then into calorimetric pressure vessel. A wire was used to support combustion. The pressure vessel was closed with matrix and it was filled with oxygen with 28 Atm pressure. After that it was inserted to a calorimeter at the defined position. The combustion process took 8 minutes and then showed dTk value which used for further calculation. For this measurement it was used Calorimeter MS – 10 A (Figure 18).

Figure 18: Used Calorimeter MS–10A
Source: Author (2016)

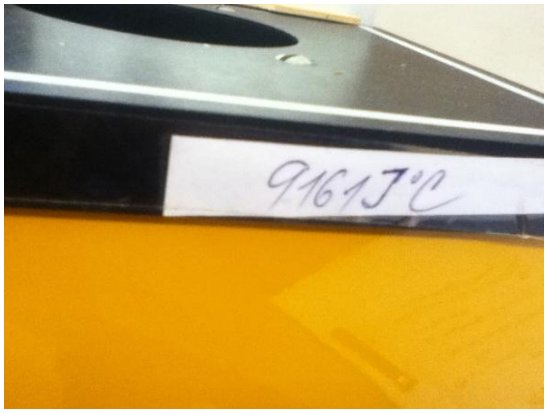


Figure 19: Calorimeter constant
Source: Author (2016)

Formula for determination of gross calorific value:

$$\boldsymbol{GCV = \frac{dTk \times Tk - (c_1 + c_2)}{m}}$$

where:

GCV = Gross Calorific Value (J/g)

dTk = temperature jump (°C)

Tk = heat capacity of calorimeter (J/°C) (Figure 19)

c_1 = paper

c_2 = wire

m = sample weight (g)

4.4.4 Net Calorific Value Test (NCV)

Obtained values of Gross calorific value were used for calculation of NCV. The difference between GCV and NCV is that NCV is not possible laboratory measured and therefore it is calculated from GCV. In the test of GCV the water vapor is condensed but NCV process water contains in the fuel gas remains in the gaseous state i.e as water vapor means the latent heat of water will not release of condensation.

Formula for determination of net calorific value:

$$\boldsymbol{NCV = GCV - 24.42 \times (w + 8.94 \times H_a)}$$

where:

NCV = Net calorific value (J/g)

GCV = Gross calorific value (J/g)

24.42 = coefficient of 1% water in the sample at 25°C (J/g)

w = water content in the sample (%)

8.94 = coefficient for the conversion of hydrogen to water

H_a = hydrogen content in the sample (%)

4.4.5 Heavy Metals Content Test

Determination of heavy metals content was done according to standard EN ISO 16968 (2015): Solid biofuels – Determination of minor elements. The process was carried out at Faculty FAFNR in Laboratory of Environmental Chemistry of CULS. There were used samples with weight 0.3 g in 3 replicates. The mixture with concentrated nitric acid was heated at maximum power 300 W, temperature 180°C and pressure 21 bar for 12 mintues (Šindelářová, 2015). Element contents in the digests were measured by inductively coupled plasma mass spectrometry ICP-MS, Agilent 770x.

4.4.6 Carbon, Nitrogen and Hydrogen Content Test (CHN)

Determination of CHN was carried out at Bioenergy Center of Research Institute of Agricultural Engineering according to the standard EN 15104 (2011) with laboratory device LECO CHN628 Series Elemental Determinator (Figure 20). The sample was burnt in oxygen (sometimes it may be burned in mixture with oxygen and carrier gas) and from this was created ash and gaseous combustion products. The whole process is that dried material rounded to 0.1 g of weight was packed into a small globule from aluminium foil. The sample was loaded into an autoloader and for removing atmospheric gas it inserted into the purge charger. To ensure complete combustion of all samples, the further sample was introduced into the dual-stage furnace system which operating at temperatures up to 1050°C. The computer automatically showed results.

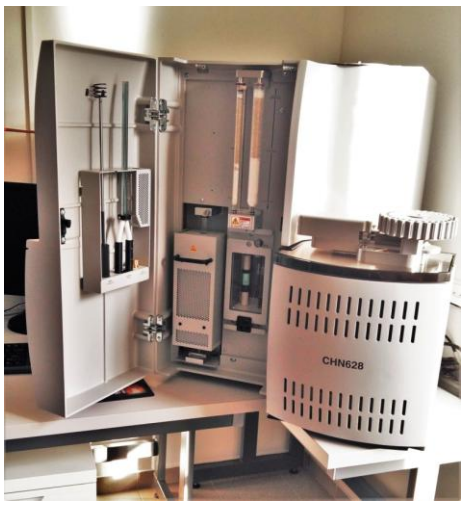


Figure 20: Laboratory device for CHN determination

Source: Author (2016)

4.4.7 Volatile Matter Content Test (VM)

Volatile matter content was determined by burning of material analytical sample without air under standard conditions such as loss of weight after deducting the weight of water contained in the sample according to the Standard EN 15148 (2010). It was used laboratory device ELSKLO brand, type MF5, heat output is 2.3 kW. The test was carried out at BCRIAE. The preparation and drying of sample was took place at FTA at CULS according to standard EN 14774-3 (2010).

For the first part of process were empty crucibles with lid put to the Muffle furnace for 7 minutes for temperature 900 ± 10 °C. After that crucibles were cooled to indoor temperature and were put into the desiccator. The crucibles with samples an average of 1 g were weighted and were put into the Muffle furnace ELSKLO for 7 minutes for the same temperature again. Afterwards crucibles with sample were cooled, put into desiccator and then weighted. The volatile matter was calculated according to formula.

Formula for determination of volatile matter content:

$$\boldsymbol{V_d = \left[\frac{100(m_2 - m_3)}{m_2 - m_1} - M_{ad}\right] \times \left(\frac{100}{100 - M_{ad}}\right)}$$

where:

m_1 = mass of empty crucible and lid (g)

m_2 = mass of crucible with sample and lid before heating (g)

m_3 = mass of crucible with sample and lid after heating (g)

M_{ad} = moisture = percentage by mass in the general analysis sample (%)

4.4.8 Durability Test (DU)

Determination of mechanical durability was carried out according to European Standard EN 15210-1 (2010): Solid biofuels – Determination of mechanical durability for pellets. It consists of 2 parts. The first part is about quantity of particles sieved and the second part about a test of durability.

First it was made a sample weight in range from 1 to 1.5 kg and it had to be 5 to 10 times hand shaken on a sieve of 40 cm diameter and sieves holes diameter of 3.15 mm. It was before the determination of mechanical durability.

The second part was determination of mechanical durability. It was measured by special rotary drum with simple Pellet Tester with prismatic shape.

Pellets were divided onto two parts for about 500 g and weighted with accuracy 0.1 g. Pellets were inserted into pellet tester with 50 RPM, i.e. 500 revolutions per 10 minutes. Furthermore testing pellets were removed and weighed.

Figure 22: Pellet tester used

Source: Author

Figure 21: Cotton pellets after test

Source: Author

Formula for determination of mechanical durability:

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

where:

DU = Mechanical durability (%)

m_A = sample weight after crumbling (g)

m_E = sample weight before crumbling (g)

4.5 CALCULATION OF MAXIMUM THEORETICAL ENERGY POTENTIAL

Calculation was intented for Sughd province in Tajikistan. This was a target area, because the cotton which was available for testing was just from this province (more in chapter 4.1). It was used Sughd area where it is cultivated cotton. For obtaining theoretical energy potential it is necessary to multiply the GCV in unit mega Joule per kilogram and number of tons of cotton residues per hectare. GCV have to be in dry basis because of theoretical energy potential. The result will be in GJ/t.

Number of tons of cotton residues was obtained from calculation average weight of 1 imported plant residues in grams which was multiply number of plants per hectare in Sughd where the cotton was grown.

$$\mathbf{EP = GCV_d \times \ Y_d}$$

where:

EP = Energy potential (GJ/ha)

GCV_d = Gross calorific value in dry basis (J/g)

Y_d = Yield of cotton residues (t/ha)

Cotton residues mean cotton roof, cotton stalk, cotton leaves, capsules and a very small portion of fibers, which was remained after harvest.

4.6 PREDICTION CALCULATION OF COTTON LINT PRODUCTION FUTURE PRODUCTION

First of all it was used statistical database Fao. From this database all available data of the production of cotton fibers were used during the period from 1992 to 2012. Data were processed in MS Excel programme and in this programme there was added a linear trendline in order to get an estimate of cotton lint production for the next 14 years up to 2026. This linear trendline was added to the graph with real date from the mentioned period.

4.7 **DATA PROCESSING**

For data processing of result values obtained during experimental research were used Microsoft Office Excel programme. For primary organization and classification of data obtained from exparimental measurement the Microsoft Office Excel software was used. Mentioned software was also used for tables and graphs creation. The results of laboratory tests were calculated with respect to repetability limits defined by relevant standards.

5 RESULTS AND DISCUSSION

It is essential to mention that referential values of pellet properties produced from other crops used in the discussion of this paper could be produced under different manufacturing conditions, however they are still comparable.

5.1 DIMENSIONS OF PRODUCED PELLETS

The length and diameter of pellets were determined according to standard EN 16127 (2012): Solid biofuels- Determination of length and diameter of pellets. For measurement it was used a digital caliper.Properties of the final samples are described below in the Table 5.

Table 5: Properties of the pellet samples

Shape	Cylindrical
Diameter	6 mm – 8 mm
Length	25 – 70 mm

Source: Author (2016)

5.2 MOISTURE CONTENT

5.2.1 Moisture Content of cotton biomass

According to standard EN ISO 18134–3 (2016): Solid biofuels – Determination of moisture content – Oven dry method – Part 3: Moisture in general analysis sample. Process of moisture content determination is described in detail in the Table 6.

Table 6: Specific measurements of MC determination process – analytical sample

No. of sample	m_1 (g)	m_2 (g)	m_3 (g)	MC (%)
1	26.3527	27.3949	27.3176	7.417002
2	26.5698	27.6507	27.5700	7.466001

where: m_1 = mass of empty crucible; m_2 = mass of crucible with sample before drying; m_3 = mass of crucible with sample after drying

Source: Author (2016)

Values indicated average MC of tested samples from cotton residues equal to **7.44%**.

5.2.2 Moisture Content of cotton pellets

Determination of moisture content is considered as one of the most important test within the pellet quality determination because feedstock moisture content influences final quality of those densified products. Results of two sample measurements which represented investigated cotton pellets exhibited approximately same values with a minimal difference. According to standard EN ISO 18134–2 (2016): Solid biofuels – Determination of moisture content – Oven dry method – Part 2: Total moisture – Simplified method. Process of moisture content determination is described in detail in the Table 7.

Table 7: Specific measurements of MC determination process – pellet sample

No. of sample	m_1 (g)	m_2 (g)	m_3 (g)	MC (%)
1	179.42	488.62	462.28	8,518758
2	219.73	529.65	501.21	9,176562

where: m_1 = mass of empty crucible; m_2 = mass of crucible with sample before drying; m_3 = mass of crucible with sample after drying

Source: Author (2016)

Values indicated average MC of tested samples of pellet from cotton residues equal to **8.85 %.**

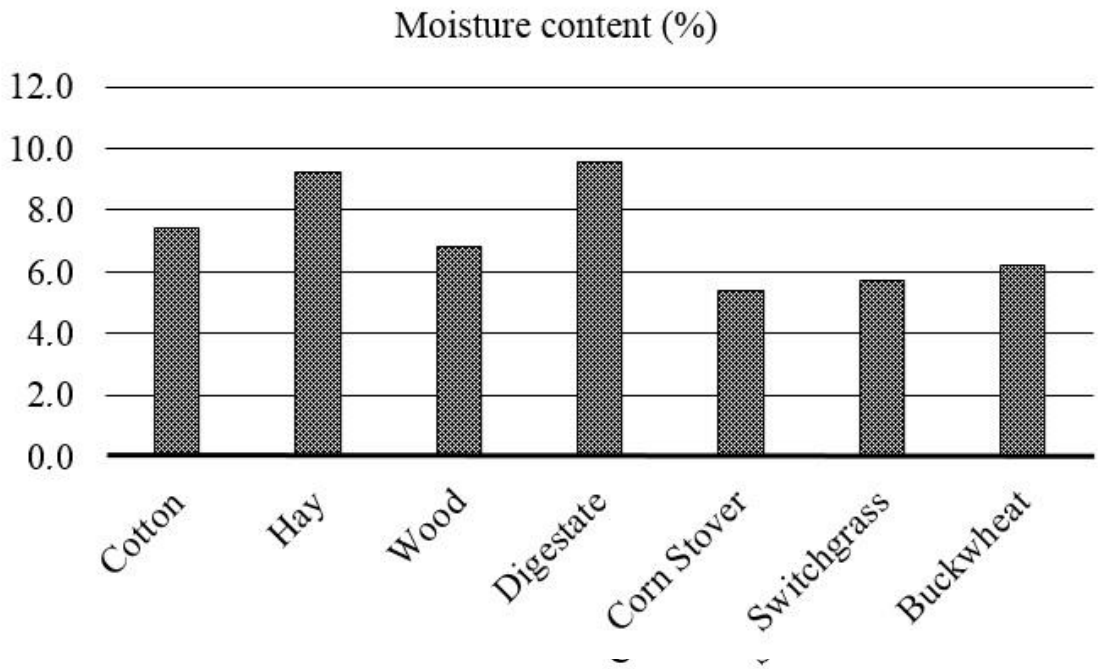
Mentioned technical standard EN ISO 18134–2 (2016) stated that moisture content of all solid biofuels must be maximal 10%. According other author overall evaluation of pellet moisture content should ranges between 8–10% in total (Kofman, 2007).

Accornding to previous researches pellets produced from differently grown and fertilized hay exhibits moisture content equal to 9.24% (Kirtsen et al., 2016), pellets produced from pure wood exhibits moisture content equal to 6.83% (Rhén et al., 2006) and moisture content of pellet from digestate, the waste material from biogas plant station (Roubík et al., 2016) ranges between 9.2% and 9.9% (Kratzeitsen et al., 2010). Research of Kaliyan and Morey (2010) also proved moisture content equal to 5.4% for

pellets from corn stover and equal to 5.7% for switchgrass pellets and Obidzinski et al. (2016) proved moisture content equal to 6.2% for buckwheat hulls pellets.

Graph 1 (see below) expresses all mentioned result value of different feedstock pellet moisture content for better orientation.

Graph 1: Compasiron of moisture content for different feedstock pellet types



Source: Author (2016), Kirtsen et al. (2016), Rhén et al. (2006), Kratzeitsen et al. (2010), Kaliyan and Morey (2010), Obidzinski et al. (2016)

In compare, previous research of Karunanithy et al. (2012) that was focused on briquettes (next common used solid biofuel) produced from cotton stalks exhibits moisture content of briquette samples equal to 8.9%.

Overall evaluation of measured moisture content and secondary data used for the comparison, it can be concluded that obtained level of moisture content of cotton residual pellets corresponds to the mandatory technical standard EN ISO 18134–2 (2016) but in compare with other feedstocks it exhibited ordinary result values.

5.3 **ASH CONTENT**

Results of experimental testing performed by two tested samples and detail values obtained during the process are noted in Table 8 below.

Table 8: Result values of experimental ash determination process

No. of sample	m_1 (g)	m_2 (g)	m_3 (g)	A_c (%)
1	24.5966	25.6848	24.636	**3.8613**
2	25.3667	26.4741	25.406	**3.8431**

where: m_1 = mass of the empty dish; m_2 = mass of dish with sample; m_3 – mass of dish with ash ; A_c – ash content
Source: Author (2016)

Overall average result of ash content was calculated equal to **3.85%**. According to mandatory technical standard EN 14775 (2009): Solid biofules – Determination of ash content which were used for the experimental test performance, the result value should be rounded to one decimal place. For the better expression of result differences between speficic tested samples, result values were noted with two decimal places.

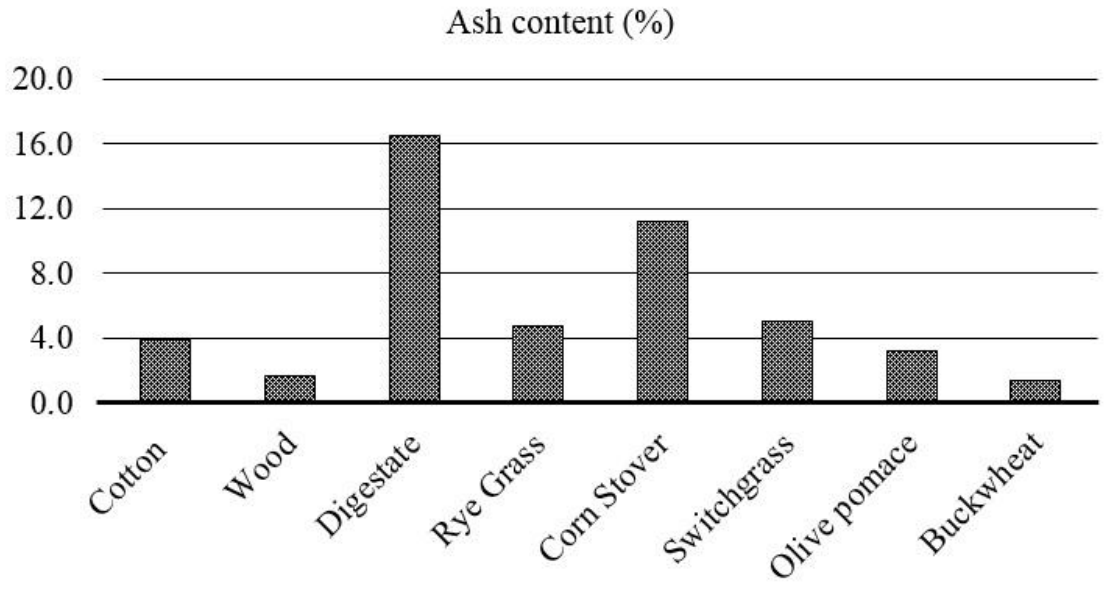
Mentioned standard EN 14775 (2009) do not determines any limits for acceptable range of ash content but standard EN ISO 17225-1 (2015): Solid biofuels – Fuel specifications and classes – Part 1: General requirements stated that ash content of cotton residues commonly ranged between 1.6% and 9.4%. When comparing this range with results of this paper it indicates that used feedstock material had acceptable chemical properties and corresponds to these recommended values. According to Kofman (2007) pellet produced from high quality wood exhibits extremely low ash content equal aproximatelly 0.7%. Different previous research proved the ash content of pellets from different part of tree (stem, branch and bark) ranged from 0.5% to 2.7% (1.6% in average). According to authors opinion the level of ash content depends on wood fiber type (Rhen et al., 2007).

Previous research done by Kratzeisen et al. (2010) exhibits that ash content of digestate pellets is in range between 14.6% and 18.3% and that indicated ash content equal to 16.45%. Other authors who deal with pellet ash content reported ash content equal to 4.7% for rye grass (Lolium perenne L.) pellets (Nathan et al., 2011), to 11.2% for corn stover pellets (Kaliyand and Morey, 2010), to 5.0% for switchgrass pellets

(Mani et al., 2006) to 3.2% for pellets from olive pomace (Barbanera et al., 2016) and equal to 1.39% for buckwheat hulls pellets (Obidzinski et al., 2016). In compare of ash content of different solid biofuels, previous research of Karunanithy et al. (2012) published ash content of briquettes from cotton stalks equal to 14.6%.

For clearer comparison of primary and secondary data noted in this chapter, Graph 2 which contains all those data was created (see below).

Graph 2: Comparison of average ash content of different feedstock pellets



Source: Author (2016), Rhen et al. (2007), Kratzeisen et al. (2010), Nathan et al. (2011), Kaliyand and Morey (2010), Mani et al. (2006), Barbanera et al. (2016), Obidzinski et al. (2016).

In compare to mentioned different feedstock material which expressed wood and herb biomass and waste material can be concluded that result value observed from this paper answers to saticfactory level of this quality indicator, however pellets produced form pure wood can exhibits greater values of ash content.

5.4 CALORIFIC VALUES

5.4.1 Gross calorific value

Results noted in this chaper are divided into two categories according to the properties of calorific value. This quality indicator was measured and expressed in so called wet and dry states. Testing of both different states contained measurements of two samples.

Table 9 (see below) exhibits ongoing measured values during the GCV_wdetermination. Results of two mentioned samples were used for the mean value calculation whereby the final GCV_w was stated equal to **17.36 MJ/kg**.

Table 9: Specific measurements of feedstock material GCV_w determination process

No. of sample	c_1 (g)	m (g)	dTk (J/°C)	GCV_w (%)
1	0.0620	0.5701	1.21231	17.351
2	0.0629	0.5978	1.26857	17.374

where: c_1 = mass of paper; m = mass of sample; dTk = calorimeter constant; GCV_w = Gross calorific value in wet state

Source: Author (2016)

Second tested material was subjected to the GCV in dry state (GCV_d) determination. Resulting values were used for the calculation of average GCV_d value which was stated at **18.63 MJ/kg.** Ongoing result values used for the calculation of final GCVd value are noted in Table 10.

Table 10: Specific measurements of feedstock material GCV_d determination process

No. of sample	c_1 (g)	m (g)	dTk (J/°C)	GCV_d (%)
3	0.0653	0.5453	1.24986	18.663
4	0.0636	0.5251	1.20157	18.602

where: c_1 = mass of paper; m = mass of sample; dTk = calorimeter constant; GCV_d = Gross calorific value in dry state

Source: Author (2016)

As was mentioned in the "Material and samples" chapter, tested feedstock material used for the pellets production contained all different parts of the cotton plant (stalks, leaves, roots, tufts of unprocessed cotton). Standard EN ISO 17225–1 (2015): Solid biofuels – Fuel specifications and classes – Part 1: General requirements describes typical value of $GCV_w$of cotton leaves ranges from 16.4 to 17.5 MJ/kg and $GCV_w$of cotton stalks ranges from 15.8 to 18.3 MJ/kg. In compare, with results obtained from experimental testing of this thesis, it is visible that used feedstock material not only fullfiled expectation but also proved higher level of GCV_w.

Secondary data which originates from previous research done by Gravalos et al. (2010) who described GCV_w of different organs of whole cotton plants. Results of this paper proved different GCV_w level of specific plant parts; highest GCV_w level was observed for cotton seeds. According to the author's opinion was this phenomena caused by higher fat content and thus by higher amount of energy. Result values of mentioned paper with specific plant organs and its GCV are noted in Table 11 (see below).

Table 11: GCV of different organs of cotton plant

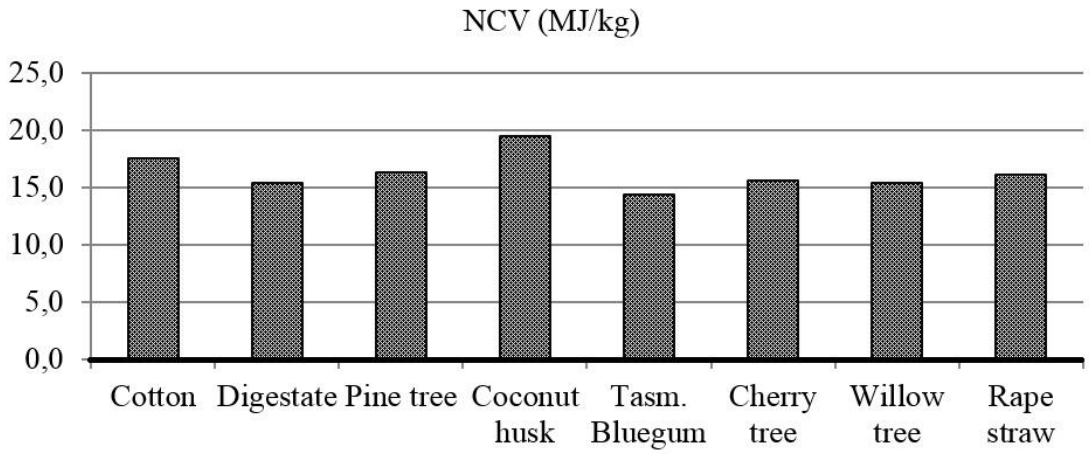
	Plant organs	**GCV (MJ/kg)**
	Seeds	22.933
	Main stem	17.733
	Root	17.707
	Vegetative branches	17.376
	Fruiting branches	17.368
	Bur	17.141
	Locks	16.679
	Terminal bud	16.396
	Leaves	16.059

Source: Gravalos et al. (2010)

5.4.2 Net calorific value

Result values of NCV of tested feedstock material were calculated by using proper formula according to the mandatory technical standard EN 14918 (2008): Solid biofuels – Determination of calorific value. Whole process of calculation and proper

formula are noted in Methodology chapter. Calculated result exhibits NCV equal to **17.58 MJ/kg.** Other technical mandatory standard EN ISO 17225–2 (2015): Solid biofuels – Fuel specifications and classes–Part 2: Graded wood pellets shows that NCV of pellets produced from chemically untreated wood residues should ranges between 16.5 and 19 MJ/kg. Kratzeisen et al. (2010) published research focused on pellets produced from waste materials which proved NCV values equal to 15.4 MJ/kg for digestate and to 16.3 MJ/kg for pinewood (with bark) material. Other authors published NCV equal to 19.45 MJ/kg for coconut husk material (Grover and Mishra, 1996), to 14.41 MJ/kg Tasmanian bluegum (*Eucalyptus globulus*), to 15.55 MJ/kg for wild cherry wood (*Prunusavium*), to 15.37 MJ/kg for Babylon weeping willow (Salix babylonica), to 15.62 MJ/kg for sycamore wood (*Acer pseudoplatanus*) (Telmo and Lousada, 2011) and to 16.13 MJ/kg for rape straw (Stolarski et al., 2013). Comparison of these feedstocks is in the Graph 3.



Graph 3: Comparison of different feedstocks NCV in MJ/kg

Source: Author (2016); Kratzeisen et al. (2010); Grover and Mishra (1996); Telmo and Lousada (2011); Stolarski et al. (2013)

5.5 **HEAVY METALS CONTENT**

In the framework of heavy metals determination several chemical elements of investigated cotton residual pellets was monitored. Namely Chromium (Co), Nickel (Ni), Copper (Cu), Zinc (Zn), Arsenic (As), Cadmium (Cd), Mercury (Hg) and Lead

(Pb). Within heavy metals determination which was performed according to the standard EN ISO 16968 (2015): Solid biofuels- Determination of minor elements following values (see in Table 12) were measured.

Table 12: Minor elements analysis in d.b. in µg/kg

No. of sample	Co	Ni	Cu	Zn	As	Cd	Hg	Pb
1	89.743	1184.756	3010.494	5668.587	81.587	9.245	1.503	168.885
2	91.448	1216.145	3026.232	6256.234	82.944	8.639	1.839	173.349
3	85.907	1103.426	3210.125	5729.017	79.904	9.144	1.556	168.974

Source: Author (2016)

Mentioned data noted in Table 12 were used for the final calculation of result values of specific elements which was performed in accordance to instruction stated by standard EN ISO 16968 (2015). Results of calucation which express average of final values of heavy metals elements in cotton residues are noted in Table 13 below.

Table 13: Content of minor elements in cotton residue pellets in d.b. in mg/kg

Element	Content
Co	0.089
Ni	1.168
Cu	3.082
Zn	5.885
As	0.081
Cd	0.009
Hg	0.002
Pb	0.170

Source: Author (2016)

Pellets produced from different kinds of biomass naturally exhibits different values of heavy metals according to its origin. Thus different requirements must be achieved to keep satisfactory the level of pellet quality. Accpetable values of heavy metals in pellets from different kinds of biomass are stated by mandatory technical standards EN ISO 17225–2 (2015): Solid biofuels – Fuel specifications and classes – Part 2: Graded wood pellets and EN ISO 17225–6 (2014): Solid biofuels – Fuel specifications and classes – Part 6: Graded non–woody pellets and are noted in Table 14 below.

Table 14: Heavy metals content of different biomass pellets in d.b. in mg/kg

Element	Wood material	Cereal straw	Non-woody material
Co	< 10	≤ 50	≤ 50
Ni	< 10	≤ 10	≤ 10
Cu	< 10	≤ 20	≤ 20
Zn	< 100	≤ 100	≤ 100
As	< 1	≤ 1	≤ 1
Cd	< 0.5	≤ 0.5	≤ 0.5
Hg	< 0.1	≤ 0.1	≤ 0.1
Pb	< 10	≤ 10	≤ 10

Source: EN ISO 17225-2 (2015); EN ISO 17225-6 (2014)

The fact that feedstock used for the experimental research of this thesis consisted all plant organs (also the tufts of raw cotton) classification of feedstock is not expressly understood according to mentioned biomass kinds categories. But if the result values of present research would be compare than with the woody materials values. This comparison indicates that all measured heavy metals values corresponded to the required levels accept one. Result values of Lead (Pb) which exhibited value equal to 0.170 mg/kg which did not correspond to the standard reqirement < 10 mg/kg. In conclusion results of heavy metals determination proved high level of chamical quality of pellets produced from cotton residues.

5.6 **CHN CONTENT**

Within the chemical content determination of cotton feedstock a Carbon (C), Nitrogen (N) and Hydrogen (H) content was stated. Measured result values of experimental testing are noted in Table 15 (see below).

Table 15: Chemical composititon of cotton feedstock material in %

Chemical components	Content (%)
Carbon	39.05
Nitrogen	0.73
Hydrogen	4.81

Source: Author (2016)

According to the mandatory technical standards EN ISO 17225–1 (2015): Solid biofuels – Fuel specifications and classes – Part 1: General requirements typical C, N and H value ranges of cotton leaves are following:

- Carbon: 39.6 – 43.7%
- Nitrogen: 0.2 – 2.9%
- Hydrogen: 5.3 – 6.1%

Within the monitoring and evaluation of result values of this chapter, the measured primary data were compared with data of other authors from presious researches published before. Mentioned primary and secondary data are noted in the Table 16 (see below) for easier comparison.

Table 16: Comparison of chemical components of other commonly used feedstocks in %

Material	Carbon (%)	Nitrogen (%)	Hydrogen (%)
Bituminous coal	73.17	1.43	5.54
Pine sawdust	55.34	0.10	5.83
Rape seed	50.39	0.75	5.98
Willow chips	50.28	0.03	5.42

Wheat straw	48.51	0.31	5.52
Miscanthus	48.11	0.53	5.43
Rice straw	41.43	0.75	5.01
Cotton residues	**39.05**	**0.73**	**4.81**

Source: Author (2016); Stolarski et al. (2013); McKendry (2002); Oladeji (2010); Sotannde et al. (2010)

Lowest result values of tested chemical components were highlited by bolt font. After comparing primary data with data of other authors it is clearly visible that cotton residues exhibited in case of Carbon and Hydrogen lowest values which is highly recommended according to the propriate standard EN ISO 16948 (2016): Solid biofuels - Determination of total content of carbon, hydrogen and nitrogen. In case of Nitrogen content cotton measured result value exhibited ordinary level of this chemical component in comparison with othe feedstocks. Thus after overall evaluation all mentioned datas it can be concluded that cotton residues exhibits satisfactory Carbon, Nitrogen and Hydrogen level.

5.7 **VOLATILE MATTER CONTENT**

As well as other experimental tests performed within research of this thesis, even volatile matter content (VM) determination consisted testing of two samples. Results of those two samples exhibited indentical final VM results. Detail measurement values are noted in Table 17 below. According to the standard EN 15148 (2009): Solid biofuels – Determination of the content of volatile matter, a minimal difference between two measurements is highly recommended in accordance to keep the repeatability limit; maximum acceptable difference between measurements is 2.0%. Considering this fact the average result value of VM was stated to **74.9%**.

Table 147: Specific measurements of raw material VM determination process

No. of sample	m_1 (g)	m_2 (g)	m_3 (g)	VM (%)
1	18.3167	19.3169	18.5678	74.8950
2	21.1680	22.1668	21.4191	74.8950

where: m_1 = mass of empty crucible and lid; m_2 = mass of crucible with sample and lid before heating; m_3 = mass of crucible with sample and lid after heating; VM = Volatile matter content

Source: Author (2016)

Final appearance of cotton sample after determination of VM level is displayed at Figure 23 (see below).

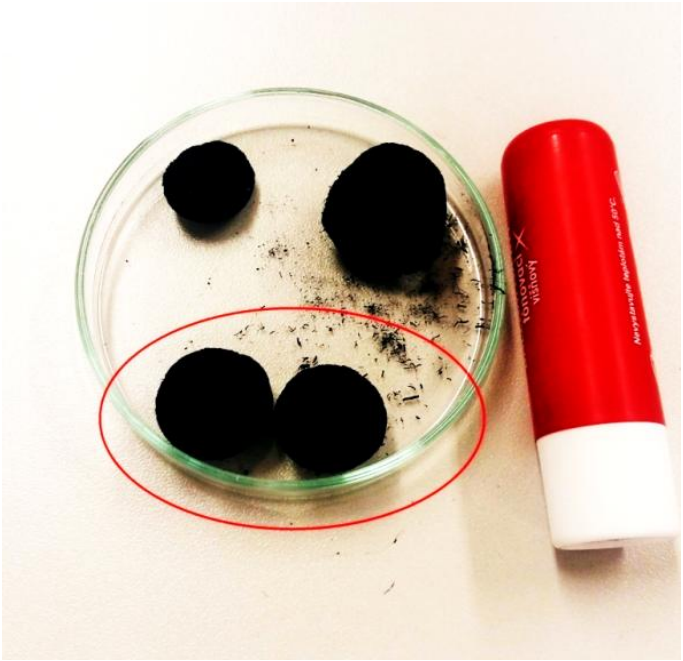


Figure 23: Cotton sample after VM test (compare with Jatropha and Rice straw)

Source: Author (2016)

Determination of VM level contains between basic chemical test of pellet and other solid biofuels quality determination. Thus lagre number of feedstock materials was already tested before and randomly selected diverse material and its values are noted in Table 18 below for clearer evaluation of result value of this thesis.

Table 15: Comparison of VM of different specific raw materials in %

Material	VM (%)
Corn cob	86.53
Pine	78.84
Virginia mallow	77.47
Willow	77.04
Tropical hardwood	75.17
Cotton residues	**74.90**
Rape straw	74.58
Municipal waste composting	69.76
Rice husk	67.98
Wheat straw	59.00
Barley straw	46.00
Bituminous coal	35.00

Source: Author (2016); Emerhi (2010); Prasityousil and Muenjina (2013); Telmo and Lousada (2011); Stolarski et al. (2013); McKendry (2002)

As it is visible in the Table 18, cotton residues investigated in experimental research of this thesis exhibits common result values of VM. Table 18 also reflects fact, that cotton residues exhibits better results of VM than herbaceous materials but worst results of VM than wood materials. According to mandatory technical standard EN 15148 (2009) lower level of VM is required thus it can be concluded that cotton residues reached satisfactory VM level.

5.8 **MECHANICAL DURABILITY**

Two samples of cotton pellets were subjected to experimental testing to determinate mechanical durability. Result values proved mechanical durability at level of 98.1% for first sample and 97.7% for second tested sample. Detail values obtained during the process of experimental testing are noted in Table 19 below.

Table 169: Measurements of DU of tested samples

No. of sample	m_E (g)	m_A (g)	DU (%)
1	0.5052	0.4956	98.1
2	0.5070	0.4952	97.7

where: m_E = mass before testing; m_A = mass after testing; DU = mechanical durability
Source: Author (2016)

Average mechanical durability was stated at **97.9%** which corresponds to the highest level of this quality indicator (>95.0%) according to the technical standard EN 15210-1 (2009). According to Kofman (2007) is required mechanical durability of pellet from all feedstock materials equal to at least 97.5%. Demonstation of other possible different pellet feedstocks and mechanical durability of these final products are noted in Table 20 (see below) for better comparison with results of this paper.

Table 17: DU of different feedstock pellets in %

Feedstock material	DU
White oak	99.0
Yellow poplar	99.0
Sweetgum	99.0
Red maple	98.8
Southern red oak	98.5
Tupelo	98.4
Cotton residues	**97.9**
Loblolly pine	97.2
Corn stover	96.0
Olive pomace	95.6

Switchgrass	87.0

Source: Author (2016); Barbanera et al. (2016); Kaliyan and Morey (2009); Kaliyan and Morey (2010)

If compare values noted in Table 20 it can be conluded that pellets produced from cotton had great level of mechanical durability but there are planty of other feedstock which reached the same or even better level od mechanical durability.

Authors who were dealing with briquettes produced from cotton residues published results of mechanical durability equal to 99.56% (Rajkumar et al., 2013) and 92.30% (Karunanithy et al., 2012). Both of these results are acceptable for the commercial usage which is defined by mechanical durability <90% as well as pellets tested wihtin this paper.

5.9 **THEORETICAL ENERGY POTENTIAL**

Total area of Tajikistan is 14,255,000 ha (Fao, 2013). Total area of Sughd province is 2,540,000 ha (Fao, 2013). From this area, the area occupited cotton cultivation is 57,000 ha according to TAJSTAT (2014).

According to TAJSTAT (2014) in Sughd region 100,000 plants in planted in 1 ha. According to Author´s data the average weight of 1 plant (residual biomass is 60 g). It means that cotton residues yield is 6 t/ha or 342,000 tons per total area where the cotton is grown in the selected region. 6t/ha is the yield in natural state (wet basis), therefore it had to be expressed in dry basis. Moisture was deducted from yield and the result was 5.4 t/ha.

Cotton residual biomass mean cotton roof, cotton stalk, cotton leaves, capsules and a very small portion of fibers, which was remained after harvest.

For obtaining theoretical energy potential it is necessary to multiply the GCV in dry basis (17.36 MJ/kg) and dry basic yield of cotton residues (t/ha). The result is **93.59 GJ/ha**.

This energy potential is average value in comparison of other selected crops. Comparison with other crops is presented in the Table 21 on next page.

Table 18: Characterization of waste biomas from selected crops for energy potential

Crop	Biomass yield (t/ha)	Moisture (%)	Biomass yield in d.b. (t/ha)	GCV (MJ/kg)	Energy potential (GJ/ha)
Topinambur	33.6	70	10.1	15.71	158.36
Hemp	8.4	15	7.1	18.06	128.95
Cotton	**6**	**7**	**5.6**	**17.36**	**93.59**
Oat	4.1	10	3.7	17.64	64.62
Sunflower	3.8	10	3.4	16.97	57.95

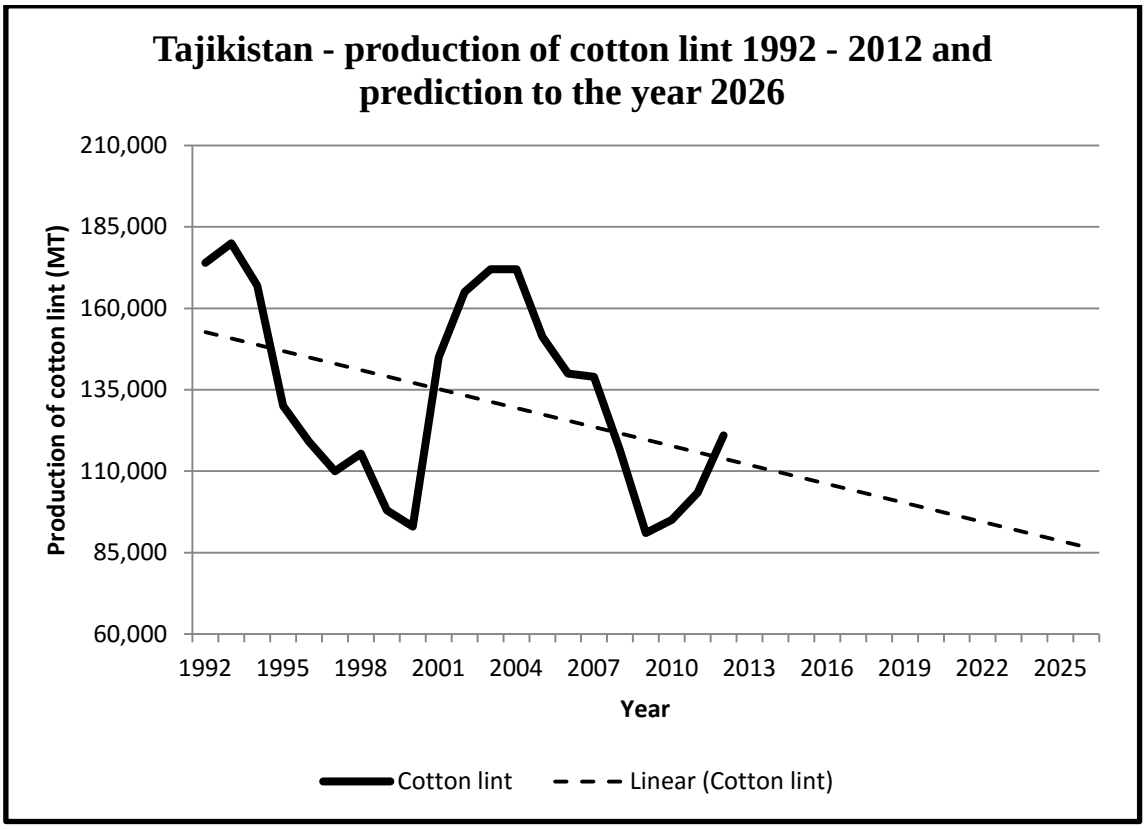
Source: Author (2016), Havlíčková (2010)

Crops which are intentionally grown for energy purposes have the higher value of energy potential than sunflower, oat or cotton that are primary grown for other industries. Tha results of table 18 show that energy potential of cotton residues is high.

5.10 PREDICTION OF COTTON LINT FUTURE PRODUCTION

Based on FAO statistical data, the prediction of cotton lint production was calculated for the future 14 years according to the linear trenline in Graph 4 (see below).

Graph 4: Production of cotton lint from 1992 to 2012 and prediction of production to the year 2026 in Tajikistan



Source: Fao (2012)

This estimation could not be considered for trustworthy because of the small amount of data. Unfortunately larger numbert of data was not available. This is only a rough estimate which could be taken into account as input data for the calculation of the potential cotton residues yield. For more accurate estimation large period of real data would be required. Therefore it is possible that the production line will not decline. It depens on the climat conditions, the world economy, demand for cotton products, the competitive fight with synthetic fibres, etc,.

6 CONCLUSION AND RECOMMENDATION

Results of tests were showed that pellets produced from cotton residues have great test values. Mainly the great strengths were identified as high quality physical and mechanical properties such as high durability fully accepted for commercial usage and high calorific value and percentage of moisture is in accordance standards too.

All the tested of chemical properties such as content of ash, heavy metals, volatile matter and carbon, hydrogen and nitrogen are in line with standards.

Whereas the cotton is not intentionally grown for biomass, energy potential is high with respect to the main use is for textile industry therefore the processing of cotton residues are suitability for the pellet production and it is absolutely according the European Standards.

The final evaluation the quality of pellets made from cotton residues is very positive due to great resulting values and it is recommended to produce these types of pellets. These test results are especially useful for targeted incrasing the share of renewable energy which plays an irreplaceable role in the state energy policies. It was assumed that resulting values obtained in pellet tested of cotton from Tajikistan will approximately corresponded to the values obtained of pellet from cotton from other areas and it is likely to show certain dependence on local conditions and fertilization.

It is recommended to concentrate further research for testing pellets from cotton from other regions and comparision between them and for achieving for complete evaluation applicable for global extension idea about pellets from cotton residues.

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