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**Use of the changes in the volume of digestate briquettes stored in the soil**

**Master Thesis**

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## **Declaration**

I hereby declare that the present Master Thesis entitled “Use of the changes in the volume of digestate briquettes stored in the soil“ is my own work and all the sources have been quoted and acknowledged by means of complete references.

In Prague 22.4.2015

.....  
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## ABSTRACT

This paper focuses on a possibility of a non-energetic use of the material remaining after the anaerobic digestion in biogas stations. It aims to investigate effects of water sorption on changes in selected physical properties (volume and moisture) of briquettes made from separated and partially dehydrated digestate in comparison with briquettes made from *Miscanthus sinensis* when stored in soils and also the effects of briquettes on the soil water regime. The material was subjected to examination in different experimental conditions to achieve the study objectives. The water sorption of briquettes was examined in two different types of soil - loam and loamy sand, in laboratory and outdoor environment. Experiments have demonstrated that the course of water sorption differed when examined in laboratory and outdoor conditions, except the speed of sorption, where the sorption maximum was reached in 12 - 14 days, regardless the soil moisture and indoor/outdoor conditions. Even after reaching the sorption maximum the briquettes were able to store the adsorbed water over a period. It has been proved the water uptake and the change in volume of briquettes depending on soil texture when those properties significantly differed in loamy soil and loamy sand soil used. Regression analyzes have shown the medium negative dependence of the moisture of soil on the moisture of briquettes, meaning the moisture of soil decrease with increasing moisture of briquettes and also no dependence of change in volume on moisture of briquettes.

Key words: water sorption, digestate, volume changes of briquettes, density of digestate  
briquettes, type of soil, loam, loamy sand

## AUTORSKÝ REFERÁT

Tato práce se zaměřuje na možnosti neenergetického využití tuhého zbytku produkovaného anaerobní digestí v bioplynových stanicích. Jejím cílem je prozkoumat účinky vodní sorpce na změny ve vybraných fyzikálních vlastnostech (objem a vlhkost) briket vyrobených z částečně dehydrované digestátu a porovnání s briketami vyrobenými z ozdobnice čínské (*Miscanthus sinensis*), uložených v půdě a také vliv uložení briket v půdě na její vodní režim. K dosažení námi stanovených cílů práce byl materiál podroben zkoumání v různých podmínkách. Schopnost briket sorbovat vodu byla zkoumána ve dvou různých typech půdy - hlinité a hlinitopísčité, v laboratoři a venkovním prostředí. V obecné rovině náš výzkum potvrdil, že průběh sorpce vody se v podmínkách laboratorního a venkovního prostředí lišil, tedy kromě rychlosti sorpce, kde bylo maximální sorpce dosaženo mezi 12-14 dnem od vložení materiálu do půdy, bez ohledu na půdní vlhkost a podmínky prostředí. Dokonce i po dosažení maximální sorpce, briкеты byly po určitou dobu schopny uchovat adsorbovanou vodu. Bylo zjištěno, že příjem vody a změny v objemu briket závisí na půdní textuře, kdy se tyto vlastnosti v námi použité hlinité a hlinitopísčité půdě významně lišily. Regresní analýza ukázala střední negativní závislost vlhkosti půdy na vlhkosti briket, tedy že s rostoucí vlhkostí briket vlhkost půdy klesá a také nezávislost změny objemu briket na vlhkosti briket.

Klíčová slova: sorpce vody, digestát, objemové změny briket, hustota briket z digestátu, typ půdy, hlinitá půda, hlinitopísčité půda

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## LIST OF ABBREVIATIONS

AD	Anaerobic digestion
BGS	Biogas Station
C	Carbon
CULS	Czech University of Life Sciences
Cl	Chlorine
FTA	Faculty of Tropical AgriSciences
FAO	Food and Agriculture Organization of the United Nation
GHG	Green House Gasses
GWP	Global Warming Potencial
K	Potassium
MSW	Municipal Solid Waste
MPa	Megapascal
N	Nitrogen
NFE	Nitrogen-free extract
OM	Organic Matter
P	Phosphorus
pH	Potential of Hydrogen
w	Moisture
WRB	World Reference Base
wt	Weight percentage

## 1 Introduction

Over recent years, an increasing amount of organic materials from renewable energy sources has become available for application to land as digestates, those materials remaining after the anaerobic digestion (Tambone et al., 2009). Many works have been published during the last few decades about the process of anaerobic digestion which offers several environmental, agricultural and socio-economic benefits throughout production of biogas as clean, renewable fuel, for multiple utilizations and as one of the practical alternatives in order to achieve national and international Greenhouse Gas emission reduction targets and digestate as improved fertilizer of the quality of manure, with considerable reduction of odors and inactivation of pathogens (Pezzolla et al., 2012). Since the digestate is mostly applied into the soil in its liquid form, which causes several difficulties with its transportation and storage, nowadays there has been a great focus on possibilities of processing of the digestate solid fraction and its further use in the soil (Rehl and Müller, 2011). Therefore, it is important to understand the soil environment as one of the most important natural resources for the efficient crop production. Linkages between physical properties of digestate processed into the form of briquettes or pellets and physical–chemical soil properties are still poorly understood due to the variety of factors involved in the process (Regelink et al., 2015).

This this paper has been focused on the sorption properties of the digestate briquettes in completely limited environment - soil conditions were simulated in the laboratory wooden containers and also were carry out outside in the soil of the experimental plot, where the physical–chemical soil properties and physical properties of digestate briquettes were influenced by the changing weather conditions. As the comparative material due to digestate briquettes properties were used briquettes from an energy crop *Miscanthus*, which were chosen because of the material qualities similar to digestate. There were measured several characteristics as change in weight, diameter and length of briquettes, moisture of the briquettes and moisture of the surrounding soil and the time period of the experiment. This paper has been is primary formulated as an experimental work, which was undertaken primarily to acquire new knowledge of the fundamental principles of phenomena and observable facts, without a specific particular application or use in practice.

Water sorption, generally speaking, causes the increase in a volume of absorbing objects. Since all the processes connected to water sorption of not only digestate briquettes, but also briquettes from other biomass are not well-known yet, this topic has been chosen due to its a this significant knowledge gap. It brings a great potential in further research in use of the briquettes properties, as for example their use as a water reservoir in soils with irregular rainfall inputs (Pecen et al., 2014), since briquettes after sorption contain an extensive volume of water.

## 2 Literature review

This chapter presents an overview of scientific literature which has been published on the topic of mutual relation between digestate and soil properties and which has been used in our own further research.

### 2.1 Digestate

According to the decree of the Czech Ministry of Agriculture no. 474/2000 Coll., on fertilizer requirements, digestate is defined as organic fertilizer made exclusively from manure and roughage from anaerobic fermentation. It is an organic material which contains a minimum 25% of combustibles and a minimum of 0.6% N in dry matter and also it must meet the limit values of hazardous elements (see Table 1).

**Table 1: Limit values of hazardous elements in organic fertilizers**

mg/kg dry matter								
cadmium	lead	mercury	arsenic	chromium	molybdenum	copper	nickel	zinc
2	100	1	10	100	100	5	50	400

Source: Vyhláška Ministerstva zemědělství č. 474/2000 Sb, 2000.

#### 2.1.1 Anaerobic digestion and digestate origin

Over the last few decades the anaerobic digestion process has been studied for its application in biomass and solid waste digestion and for its wide use in agriculture and waste water treatment (Lindmark et al., 2014). There are numerous important benefits of the anaerobic digestion process such as energy savings through production of biogas which is a renewable energy source, reduction in air and water pollution and preservation of natural resources by using the end-products as soil amendments and fertilisers (Tambone et al., 2009; Albuquerque et al., 2012). Digestate, the digestion co-product, is an improved fertiliser in terms of both its availability to plants and its rheology (Ward et al., 2008). Anaerobic digestion also uses stable wastes and field residues as a tool to fertilise crops selectively within the cropping system (Möller and Stinner, 2009). The technology is also considered as one of the most important mitigation options for the greenhouse gases

emissions (GHG) from farming (Kythreotou, 2014). By means of optimal recycling measures it is possible to prevent emissions of GHG and also to prevent leaching of organic matter and nutrients to the natural environment (Holm-Nielsen et al., 2009). Chen et al. (2008) and Ward et al. (2008) see main advantages of anaerobic digestion in low sludge production in comparison to aerobic treatment technologies and a low energy requirement. In spite of the above mentioned benefits, the operational stability seems to be the weak point of the process, which still prevents anaerobic digestion from being widely commercialized, since the success of the process is highly dependent on mixing for distribution of microorganisms and nutrition, inoculation of fresh feed, homogenization of the material, removal of end products of metabolism and for evening out the temperature inside the digester. Microorganisms are sensitive to mixing intensity and may not survive, so failure to maintain the balance between microorganisms is the primary cause of reactor instability (Chen et al., 2008; Lindmark et al., 2014). Economic efficiency of the whole process is then influenced by investment costs and costs for operating the biogas plant also depends on optimum methane production (Amon et al., 2007).

The anaerobic digestion process involves the degradation and stabilization of organic materials under anaerobic conditions by microbial organisms and leads to the formation of a gas mixture of carbon dioxide and methane known as biogas and microbial biomass (Chen et al., 2008). This process is found in many naturally occurring anoxic environments including sediments, waterlogged soils and watercourses (Ward et al., 2008). Biogas can be produced from nearly all kind of biological feedstock types. These materials mainly come from the primary agricultural sectors such as fruit and vegetable processing wastes and packinghouse wastes and from various organic waste streams from the society as a whole. The largest resource is represented by animal manure and slurries from cattle and pig production units as well as from poultry, fish and fur. Another agricultural substrate suitable for anaerobic digestion is energy crops (Chen et al., 2008; Holm-Nielsen et al., 2009; Amon et al., 2007). The most common ones recognized as valuable energy crops are maize (*Zea mays* L.), herbage (Poaceae), clover grass (*Trifolium*), Sudan grass (*Sorghum sudanense*), fodder beet (*Beta vulgaris*), where maize is the most dominating crop for anaerobic digestion in biogas plants (Amon et al., 2007; Holm-Nielsen et al., 2009). For economic reasons, the selection of an optimal feedstock for running the AD process is adjusted to an achievement of a maximum volume of biogas produced in BGS, which

means an maximum volume of electric current produced (Pecen et al., 2014). The quality and amount of the biogas and of the digestate produced, mainly depends on quality of the biomass added into bioreactors to carry out the fermentation process. Factors influencing the biomass and further end products quality, such as e.g. plant management, harvest, conservation or environmental conditions in the digester, are pictured in the figure below (Amon et al., 2007) (Figure 1).

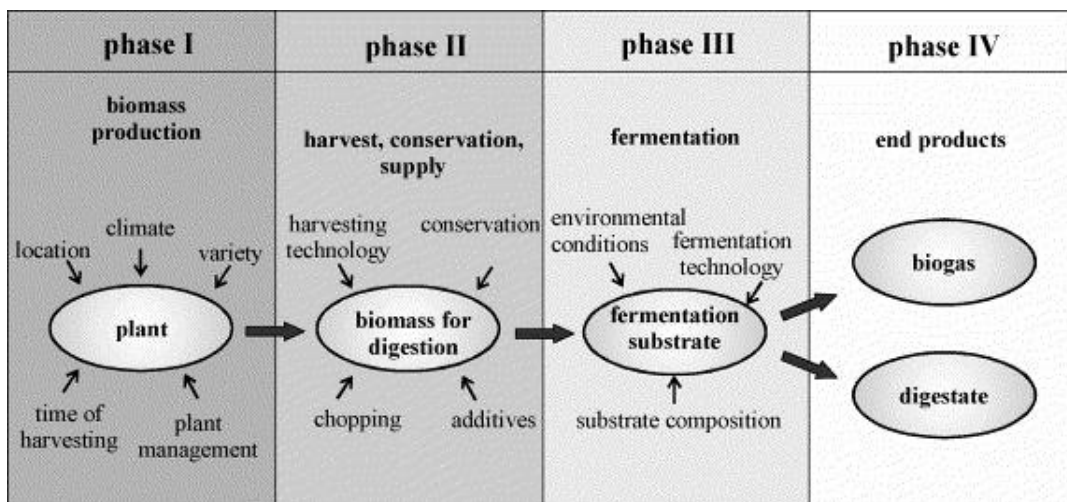


Figure 1: Influences on biomass products production along the production process (Amon et al., 2007)

Anaerobic digestion as a whole process can be divided into four main stages, which are pretreatment, digestion, gas recovery and treatment of the digestate (Verma, 2002). The pre-treatment of waste is usually made to obtain homogeneous feedstock through the solubilisation of organic material, the reduction of particle size and the improvement of biodegradability. However, intensive pretreatment can also lead to losses of organic material and a formation of resistant compounds (Lindmark et al., 2014). The digestion of organic material occurs in four microbial process steps (hydrolysis, acidogenesis, acetogenesis and methanogenesis) and it takes place inside a digester, where the feed is diluted to achieve desired solids content and remains there for a designated retention time (Verma, 2002). Every anaerobic digester is designed to fulfill the basic requirements such as a short hydraulic retention time, to allow for a continuously high and sustainable organic load rate and to produce the maximum volume of methane (Ward et al., 2008). The digestion is dry or wet, which depends on the solid content. The water used for mixing

with solid particles can be either clean water or other liquid wastes such as sewage sludge or re-circulated liquid from the digester effluent. During the digestion stage the biogas is produced (Verma, 2002). Once the biogas is produced, it is generally composed of 48–65% methane, 36–41% carbon dioxide, up to 17% nitrogen, <1% oxygen, 32–169 ppm hydrogen sulphide, and traces of other gases (Rasi et al., 2007; Ward et al., 2008). Hydrogen sulphide needs to be removed for boilers and combined heat and power units. If the gas is to be used as natural gas or vehicle fuel, carbon dioxide has to be removed as well. The biogas has to be then upgraded because of impurities, which it contains and that can damage boilers (Monnet, 2003).

During anaerobic digestion, about 20–95% of the feedstock organic matter is degraded, depending on feedstock composition (Möller and Müller, 2012) and also the decomposition achieved in the digester depends on the active biomass. The more anaerobic microorganisms are present, the faster decomposition occurs (Bauer et al., 2009). The residual product of AD is called digestate and it is usually used as fertilizer and soil amendment. Its application into soil is the most attractive option in terms of environmental issues, because of the nutrients recovery and the attenuation of the loss of organic matter suffered by soils under agricultural exploitation (Gómez et al., 2005).

### **2.1.2 Composition of digestate**

After the anaerobic treatment, according to the technology of digestion used, the produced digestate can be further refined into a solid and a liquid fraction (Holm-Nielsen et al., 2009; Teglia et al., 2011). The liquid part has a high level of nutrients while the solid fraction balances the humic equilibrium of the soil (Pedrazzi et al., 2015). When liquid, the material can consist of up to 95% water on average and a transportation of a bigger amount of digestate can cause several logistical problems. That is why digestate in liquid form is often managed through direct spreadings on agricultural lands (Rehl and Müller, 2011). But if low-quality digestates with excessive loads of pollutants are introduced into soil, soil fertility may be adversely affected, ground-water quality threatened, and the food chain contaminated (Trzcinski and Stuckey, 2011). Because of the risk, that nutrients from liquid digestate could leach into groundwaters and cause pollution, AD sites tend to move towards digestion techniques which produce digestate in solid form (Teglia et al., 2011). As

mentioned in the Rehl and Müller (2011) study, the predominant criterion for choosing a proper digestion technique is the profitability, which arises from higher revenues due to an increase of the product value, a reduction of the transportation costs and an extension of the market by novel fertilizer products (liquid fertilizer and pellets or compost used in landscape and horticulture).

In general, digestate material contains approximately 60–80% of the dry matter and the same amount of phosphorus of the original indigested slurry, but only 20–25% of the nitrogen and 10–15% of the potassium of the indigested material content (Holm-Nielsen et al., 2009). The dry matter content of the solid fraction is typically 25-30% according to Bauer et al. (2009); Möller et al. (2010) and Möller and Müller (2012), Holm-Nielsen et al. (2009) estimate the DM content of digestate to be even higher, from 25 to 35%. The amounts of organic dry matter and the carbon content of digestate are decreased by the decomposition of easily degradable carbon compounds in the digestors (Makádi et al., 2008). Digestate characteristics after solid–liquid separation are presented in Table 2.

**Table 2: Characteristics of liquid and solid digestate fraction**

	Liquid fraction of digesates	Solid fraction of digestates
DM (%)	4.5-6.6	19.3-24.7
Organic DM (%DM)		40-86
Total N (%DM)	7.7-9.2	2.2-3.0
Total C (% DM)	48.0	39.6-40.0
C:N ratio	3.7-4.8	11.2-19.3
Total P (% DM)	0.4-0.7	1.9
Total K (% DM)	3.9	3.6
pH	7.9	8.5

DM= Dry matter

Source: Möller and Müller (2012)

The NH<sub>4</sub> content of the digestate is about 60-80% of its total N content (Makádi et al., 2008). In this form it could be lost due to ammonia volatilisation (Owamah et al., 2014). When it is converted to NH<sub>4</sub>-N, it can be immediately utilized by crops. The pH of digestate is generally alkaline, which is a useful property because of the worldwide problem of soil acidification. It increases under the AD, but its range depends on the



quality of ingestate and the digestion process (Makádi et al., 2008). The alkaline pH could have contributed to the reduction in pathogens in the biofertilizer digestate as most pathogens cannot tolerate high pH levels (Owamah et al., 2014).

Composition of digestate is primarily influenced by the feedstock. But, compared to the feedstock, digestate as a final product of AD has lower amounts of nutrients and organic substances, lower C:N ratio (10:1) and comprises a bigger amount of active ammonium (Borkovec, 2014). Digestate production, according to the feedstock, can be divided into two main categories: digestate derived from animal manure and plant biomass and digestate derived from municipal solid waste and animal by-products (MENDELU, 2008).

#### **2.1.2.1 Digestate derived from animal manure and plant biomass**

This category includes biogas plants that process materials such as corn silage, grass green, silage, potato leaves and other plant biomass, or animal manure. Möller and Müller (2012) in their study point out the main properties, which digestates derived from animal slurries as well as from crops, energy crops especially, have compared to other digestates. They have higher ammonium content, decreased total and organic carbon contents and decreased organic matter contents. In contrast, Poggi-Varaldo et al. (1999) mentioned in their study, that digestates coming from feedstocks provide the lowest total ammonium concentrations, as well as the lowest chemical oxygen demand, biochemical oxygen demand and volatile organic acids. According to Tambone (2010) these kind of digestates also have reduced biological oxygen demands, elevated pH values, smaller carbon to nitrogen ratios, and reduced viscosities. This category also excludes any waste or animal by-products. For digestate coming from co-fermentation of plant biomass and animal manure such as slurry, dung and dung water, sanitary requirements must be established. This includes mainly testing the digestate for harmful bacteria (MENDELU, 2008).

### **2.1.2.2 Digestate derived from organic fraction of MSW and animal by-products**

Digestate in this category, could be made from animal manure and plant biomass as the digestate of a previous category, but may be also formed from an organic fraction of municipal solid waste, which means from solid waste generated by households, commercial establishments, industries and institutions (Farrell and Jones, 2009), such as different kinds of sludge, plant tissue wastes from agriculture, horticulture, forestry, animal faeces, urine and manure (MENDELU, 2008). When, instead of using organic fraction of municipal solid waste, the digestate is processed from animal by-products such as slaughterhouse waste, former foodstuffs, milk, colostrum, and meat and bone meal (Roháček, 2013), the station must meet specific hygienic requirements, such as being equipped with pasteurisation and sanitary units and being equipped with its own control laboratory or use an external laboratory (MENDELU, 2008). Even though the processing difficulties, for example Wu et al. (2011) study results showed, that the co-digesting of dairy manure and milk could increase biogas productivity and was also found to improve substrate solids breakdown.

### **2.1.3 Digestate quality management**

As digestate is sometimes not fully stabilized, it can contain unwanted elements, which were not degraded during digestion and also present a residual biodegradability (Teglia et al., 2011). Digestate quality and consistency can only be ensured by the use of appropriate anaerobic–aerobic process parameters on uniform feedstock comprising adequate proportions of different waste types (Abdullahi et al., 2008). Digestate quality can be assessed based on physical, biological and chemical aspects. The presence of physical impurities such as metal, plastic and rubber, sand and stones, glass and ceramic and cellulosic materials can cause serious damage to the environment (Albuquerque et al., 2012). Digestate can also contain hazardous particles such as pathogens or seeds, which can result in transmission of diseases within the environment. The chemical aspects are related to the presence of nutrients, persistent organic contaminants, heavy metals and other inorganic contaminants (Monnet, 2003). The heavy metal content originates from an anthropogenic source and is not degraded during the digesting process. The main origins of the heavy metals are the food processing industry, animal feed additives, domestic sewage,

flotation sludge and fat residues (Makádi et al., 2008). Digestates are usually rich in nitrogen when a large part of nitrogen is converted into ammonium during AD. This can lead to phytotoxic structure of the material (Teglia et al., 2011). The phytotoxic effects of the resulting soil amendment can be decreased, according to observation by Abdullahi et al. (2008), by increasing the aerobic post-treatment period. This can decrease the amount of easily biodegradable components of the waste. From other characteristics, digestates can also be too wet or too odorous. All these aspects prevent their direct application on agricultural land (Teglia et al., 2011). By combining food waste, which has a wet structure and decompose rapidly and slow degrading green, a suitable digestate which can be composted easily without the need for dewatering can be produced. After all, digestate require final aerobic 'polishing' to enhance fertilizer value and applicability as a soil conditioner. This method has been reported to be a suitable treatment as it is capable of reducing moisture content, odour as well as carbon and pathogens (Abdullahi et al., 2008). Chemical composition of every digestate from each biogas plant is different, depending on the feedstock and the manner of digestion. Prior to the application of digestate on the field it is therefore necessary to carry out its analysis in a laboratory (Lošák et al., 2013).

## **2.2 Digestate briquettes**

As was already mentioned, the digestate coming from the fermentation process has a different composition. Originally, it is a liquid and it is divided into two fractions by mechanical separation then - the liquid part with 5-6% of dry solids, which is supposed to be returned back into the fermentation process of a new material in BGS and a partially dehydrated digestate with a moisture level of between 75-85%, which after further treatment modifications can be used for combustion, fertilization or production of stock bedding (Pecen et al., 2014). One of the ways, to further treat the solid digestate fraction, can be its compression into a form of briquettes. The digestate briquettes characteristics refer in general to characteristics and properties of briquettes and pellets designated for energetic purposes and also the processing of digestate is similar to the processing of any other biomass with energetic value (Černá, 2013). The importance of focusing on processing all the different kinds of biomass comes from the fact, that biomass energy currently plays a major role in meeting the present energy needs of developed and developing countries (Grover and Mishra, 1994).

### **2.2.1 Physical properties of briquettes**

A major disadvantage of biomass is its low bulk density, which makes handling difficult, high costs of transportation and storing, and poor combustion properties. However, these problems can be overcome by compacting the loose biomass to form briquettes (Grover and Mishra, 1994), since briquettes are far better to handle rather than loose biomass. The process of briquetting can be defined as the densification process of loose biomass material in order to produce compact solid composites of different sizes with the pressure application (Saikia and Bikash, 2014). By briquetting, has been met a need to achieve consistent physical properties such as size and shape, bulk and unit density, and durability, which significantly influence storage, transportation and handling characteristics (Tumuluru et al., 2011; Zhang and Guo, 2014). For some uses, physical properties of interest include moisture content and high mechanical strength (Amaya et al., 2007; Grover and Mishra, 1996). Durability (abrasive resistance) tests simulate the mechanical handling of pellets (Zhang and Guo, 2014). The moisture content is determined by mechanical and physical water sorption, when water first fills the bigger spaces and penetrates into of smaller cavities between particles (Pecen et al., 2014). The physical properties are crucial in any description of the binding mechanisms of biomass densification (Grover and Mishra, 1996). Before briquetting, the size of the particles is changed by crushing, cutting and sorting and the material is dried to a final moisture content of about 10% according to Černá (2013), according to Grover and Mishra (1996) should not be more than 10% because it makes the briquettes weak and instable.

### **2.2.2 Briquetting technologies**

The most common type of densification of biomass is direct extrusion type, where the biomass is dried and directly compacted with high heat and pressure (Saikia and Bikash, 2014; Grover and Mishra, 1994) and which was also used for processing of our material. Due to the application of high pressures and temperatures, there are solid bridges developed by diffusion of molecules from one particle to another at the points of contact (Kaliyan and Morey, 2010). In processing, there are two basic types of the briquetting technology: the piston press and screw press. In the piston press, the biomass is extruded through a die by a reciprocating ram at a very high pressure. In the screw press, the

biomass is extruded continuously by a screw through a heated taper die (Grover and Mishra, 1994). For purposes of our own further research, digestate briquettes were made with a piston press. The comparison of both technologies is shown below (Table 3).

**Table 3: Piston press and screw press comparison**

	<b>Piston press</b>	<b>Screw press</b>
Optimum moisture content of raw material	10-15%	8-9%
Density of briquette	1-1.2 gm/cm <sup>3</sup>	1-1.4 gm/cm <sup>3</sup>
Maintenance	high	low
Homogeneity of briquettes	Non-homogeneous	Homogeneous

Source: Grover and Mishra (1994)

The digestate briquettes, because of its high content of nutrients, can be used either as an agricultural fertilizer or, since fermentation residues still contain persistent carbohydrate (cellulose, lignocellulose), they can be used also as a fuel. The utilization of digestate as fuel helps farmers to deal with limits of its application into soils (Pedrazzi et al., 2015). However, high mineral content and sulfur and nitrogen compounds lead to a relatively high quantity of slag and cause corrosion in the boiler as well as relatively high emissions (Kristöfel and Wopienka, 2013). In case of agriculture use, the crumbly and unstable structure of digestate briquettes is not a big problem, since the material is supposed to be decomposed after application of briquettes into the soil (Černá, 2013).

### **2.3 Composition of soil**

FAO (1987) defines soil is a complex body composed of five major components: mineral matter, which is obtained by the decomposition of rocks; organic matter which originates in plant residues, animal remains and microbial tissues; water, which is obtained from the atmosphere and chemical, physical and microbial reactions in soil; gases which are released from atmosphere, reactions of roots, microbes and chemicals in the soil and organisms, who are represented by microbes, worms and insects. These components all together create physical, chemical and biological properties of soil (Stenberg et al., 1998).

For the purpose of our research topic, in the paragraph below, we are going to focus on soil physical properties.

### **2.3.1 Physical properties of soil**

According to FAO (2015), in soil there are discerned following physical properties: colour, consistency, density, water flows, porosity, soil texture, available soil moisture, soil water characteristics such as soil moisture and storage capacity and soil structure.

#### **2.3.1.1 Soil sorption**

Absorption, according to Rouquerol et al. (2013), is defined as a penetration of the fluid (i.e. liquid or gas) into the solid phase. It means it occurs either as water-solid or gas-solid sorption. Both reactions bring changes in the solid structure (Szekely, 2012). The process of absorption is accompanied by adsorption process, understood as enrichment of the material in a space between two bulk phases. Sorption, in term of as a uptake of fluids by a molecular sieve, embrace both phenomena of absorption and adsorption. The solid material carrying out the absorption or adsorption process is called sorbent (Rouquerol et al., 2013). In soil, where solids are made from the mineral and organic fraction, the liquid and gaseous phase are represented by the soil water solution and the soil air. These two fill all the spaces between solid particles. Soil air differs from the atmospheric air, since the composition is significantly affected by metabolic activity of roots of plants, microorganisms and other edaphon (Taufarová et al., 2014). Soil water is one of the main factors for plant growth. The source of water in the soil are a natural rainfall, irrigation and it can also rise to the active root zone from groundwaters. Properties of gases and water are important for determining soil quality supporting plant growth (Kuncoro et al., 2014).

Soil is the result of multiple physical, chemical and biological interactions in the soil (Regelink et al., 2015). These mechanisms, according to the method of the substances fixation in soil, are classified in five categories, when every soil uses a different one to sorb the nutrients. Mechanical sorption is carried out by mechanical retention of particles in the

fine, narrowed or dead ending pores. The attraction between the adsorbate and adsorbent exists by the formation of intermolecular electrostatic (Taufarová et al., 2014). It has a limited relevance for the plant nutrition. Physical sorption is related to the surface phenomena at the interface and it has a relatively low degree of specificity (Rouquerol et al., 2013) It is manifested by increasing concentration of the molecules at the surface of the solid phase and its decreasing in the soil solution while lowering the surface free energy (Taufarová et al., 2014). Chemical sorption binds ions forming under the given conditions slightly soluble compounds stored in the pores of the adsorbent. Physico-chemical sorption is manifested by the exchange of adsorbed cations for cations in the soil solution. The main exchangeable cations in soils are calcium, magnesium and hydrogen in acidic soils. Exchangeable cations sorption pH dependent soil solution (Richter, 2004). Biological sorption occurs as a result of the edaphon life activities (Taufarová et al., 2014). During its life cycle, soil organisms consume a considerable part of plant nutrients in the soil. These accumulate in the mass of their bodies, so the amount of nutrients acceptable by plants is reduced (Richter, 2004).

#### **2.3.1.2 Porosity**

Soil porosity is defined as the ratio of the volume of pores to the volume occupied by the soil (Rouquerol et al., 2013). Pores are that part of the bulk volume that is not occupied by either mineral or organic matter but is open space occupied by either air or water (FAO, 2015). According to used aggregate hierarchy theory, soil pores and soil aggregates are formed in a hierarchical manner meaning that microaggregates (< 0.25 mm) are the building blocks for macro-aggregates (> 0.25 mm) (Regelink et al., 2015). There are four kinds of pores generally used to describe the accessibility of porosity – open pores, closed pores, transport pores and blind pores (Figure 3). The pore size is an important characteristic affecting their function. The porosity for loamy sand is in between 35 – 45% and 45 – 55% for loam (Taufarová et al., 2014).

#### **2.3.1.3 Soil texture**

Texture indicates the relative content of particles of various sizes, such as sand, silt and clay in the soil and influences the amount of water and air it holds and the rate at

which water can enter and move through soil. Soils may be assigned to textural classes depending on the proportions of sand, silt and clay-size particles. These textural classes are defined in Table 4 (FAO, 2015).

**Table 4: USDA texture classes of soil [%]**

<b>Textural class</b>	<b>Sand</b>	<b>Silt</b>	<b>Clay</b>
Sand	86-100	0-14	0-10
Loamy sand	70-86	0-30	0-15
Sandy loam	50-70	0-50	0-20
Loam	23-52	28-50	7.27
Silty loam	20-50	74-88	0-27
Silt	0-20	88-100	0-12
Clay loam	20-45	15-52	27-40
Sandy clay loam	45-80	0-28	20-35
Silty clay loam	0-20	40-73	27-40
Sandy clay	45-65	0-20	35-55
Silty clay	0-20	40-60	40-60
Clay	0-45	0-40	40-100

Source: FAO (2015)

Loam is a type of soil, which is characterized by smaller grains in diameter than sandy soils. These types of soil have a good retention capacity. They are moderately to cultivate, since they have nearly the same proportion of sand in the clay particles. Loamy soils feel moderately cohesive, plastic and sticky. They have usually a deep soil horizon and are agriculturally beneficial (Smith, 2000)..

Loamy sand soils are soils dominated by sand. This group can be further divided into coarse, medium, fine and very fine loamy sand soils according to dominant size of grains. Loamy sands feel gritty, slightly cohesive. A sample can be molded into a ball when have a sufficient moisture content. It is characterized by low retention of nutrients and usually low retention of water as well. They are easy to cultivate because of lack of clay particles (Smith, 2000).



#### **2.3.1.4 Structure of soil**

Soil structure has been defined as the size, shape and characteristics of soil particles, aggregates, and pores across the size-range from nanometers to centimeters (Regelink et al., 2015) and it is one of the most important soil physical properties, referring to the retention and transport of solutions, gases, and heat. It is determined by the distribution and the size of solid particles and pore spaces (Danielson and Sutherland, 1986). The optimal soil structure is one with stable soil aggregates of diameter of 2-10 mm. For restoring the soil structure, it can contribute loosening and crumbling of the soil, appropriate moisture, liming, appropriate crop rotation using perennial forage crops and fertilizing by quality organic fertilizers (Taufarová et al., 2014).

### **2.4 Use of digestate in soil**

Using the digestate in soil has effect on its wide range of physical, chemical and biological properties. This mainly depends on the soil types. When comparing properties of digestate to the other organic materials, on the bases of OM degradability, digestate has similar OM degradability to compost, higher degradability than digested sludge and ingestate (Makádi et al., 2008).

#### **2.4.1 Use of digestate as fertilizer**

If the output material from the BGS is applied on agricultural land for the purpose of fertilization in accordance with Act no. 156/1998 Coll., on fertilizers, soil conditioners, auxiliary plant preparations and substrates and on agrochemical testing of agricultural land or if it is further processed as organic fertilizer and then applied on agricultural land, it needs to be proceed according to the relevant regulations of the Ministry of Agriculture (MENDELU, 2008). The regulations, which are related to the application of digestate and other biosolids on agricultural land make imperative a, prior application via biological, chemical or thermal technology, storage for an extended period, or any other procedure capable of significantly reducing the fermentative capacity of biosolids and any risk to health arising from application to land. Application of biosolids to the land requires them to be stabilized (Goméz et al., 2005). Composting can be considered as an adequate post-

treatment stabilizing method. It is able to stabilize their residual organic matter, reducing by the way their phyto-toxicity and improving their humic potential (Teglia et al., 2011). Abdullahi et al. (2008) claims, that the phytotoxicity is reduced by the reduction of available biodegradable organic matter in the compost, this means that phytotoxicity decrease with a increase of biological stability. The application of digestate as fertilizer on agriculture land must be done on the basis of a fertilization plan of the farm. Inappropriate handling, storage and application of digestate can cause ammonia emissions, nitrate leaching and overloading of phosphorus (Holm-Nielsen et al., 2009). Therefore, storage and land spreading operations with digestates must be carefully controlled to avoid negative environmental impacts (Albuquerque et al., 2012). Chiew et al. (2015) in his study proves that use of the digestate as fertilizer makes a higher net contribution to the GWP and cause more eutrophication and acidification than chemical fertilizers. Pezzolla et al. (2012) do not confirm the Chiew's results when he claims that the GHG emissions are not affected by digestate application into soil. On the other hand, Chiew et al. (2015) believe, if some improvements in the digestion system are implemented successfully, digestate as fertilizer can be better than chemical fertilizers in terms of lowered GWP. However, it would still cause more eutrophication and acidification than chemical fertilizer use. Eutrophication happens mainly due to nitrogen leaching during cultivation and the contribution to acidification mainly arise from ammonia emissions when spreading digestate (Ahlgren et al., 2010). Against the use of chemical fertilizers are Owamah et al. (2014), who describe the large scale use of chemical fertilizers as the main cause of soil quality and environmental degradation. The use of digestate as fertilizers is, according to his findings, one of the important components of integrated nutrient management. Anaerobic digestate usually contains microorganisms like *Samonella*, *Shigella*, *Bacteriodes*, *Aspergillus* and *Bacillus*. These organisms runs the microbial processes in the soil faster and increase the availability of nutrients that can be assimilated by plants.

The digestate is considered to be an organic fertilizer, which means that the material goes through a mineralization process in soil, where it becomes a source of energy for soil microorganisms (Vaněk, 2009). This energy is used partly in the process of humification, which is a synthesis process, in which the high humic acid, fulvic acid and humic acid are synthesized from fragments of organic matter. The process of humification cannot occur

without previous humus mineralization. The faster the organic matter is digested, the more it supports microbial activity in soil, and it also supports humification and the mineralization process. The organic fertilizer is then evaluated as having a higher quality (Kolář et al., 2011). According to Pezzolla et al. (2012) application of digestate to the land yields positive benefits in terms of crop production, thanks to the release of nutrients for plant uptake and to the improvement in the physical properties of the soil. In contrary, Kolář et al. (2008) in their study claims, that digestate from effectively operating BGS loses its most valuable labile particles of organic matter in the digester, which the biogas is produced from. Labile organic constituents are mostly degraded, which leads to an increase in the stability of the remaining OM (Albuquerque et al., 2012). These compounds that are hard to be mineralized. For this reason, digestate is not considered such as high quality organic fertilizer. If a biogas plant does not work effectively enough, it produces admittedly less biogas but the digestate left has more labile fractions of organic matter, which are mineralized. Such a digestate can be used as a good organic fertilizer (Kolář et al., 2008). But in that case, when the production of a digestate that is not completely exhausted in terms of easily-degradable organic compounds, it can later cause problems during storage and have unfavourable impacts on the soil-plant system (Albuquerque et al., 2012). In contrary, Makádi et al. (2008) argue, if the organic loading rate of a biogas plant is high and the hydraulic retention time is short, the digestate will contain a considerable amount of undigested OM, which is not economic and does not result in a good amendment material. According to later experiments, Kolář et al. (2010) designates the solid phase of the digestate as not being an organic fertilizer because its organic matter is too stable and it cannot be an expeditious source of energy for the soil microedaphon and neither a mineral fertilizer because all available nutrients were passed to the liquid phase.

Due to the anaerobic digestion in the digester of a biogas plant, is 50% of originally organic nitrogen in the mineral form, released from unstable organic matter. That is why the digestate is considered to be a mineral nitrogen fertilizer. This mineral nitrogen which can be used by plants, is contained almost entirely in the fugate. The liquid phase of the digestate typically consists from 0.04 to 0.4% nitrogen. The amount of other nutrients (P, K, Ca, S) released, is relatively small. The solid phase of the digestate contains slowly hydrolysable, organic nitrogen, which is almost inaccessible for plants (Vaněk et al., 1995).

Nevertheless, Pezzolla et al. (2012) claim that even repeated digestate applications do not affect the soil composition in terms of soil total and mineral nitrogen. Use and dosage of digestate as fertilizer is largely similar to using a dose of manure, but also taking into account the actual content of nutrients and needs of cultivated plants (MENDELU, 2008).

#### **2.4.2 Use of digestate as soil amendment**

Except for fertilizer, solid digestates can be used directly after anaerobic digestion in agriculture as a soil amendment. The soil amendment is a material added to soil, where it is supposed to improve its physical properties such as water retention, permeability, water infiltration, aeration or structure (Teglia et al., 2011) and to contribute in maintaining the soil humus balance (Tambone et al., 2009). The roots environment is improved and plant development as well. The addition of organic amendments into soil restores the quality of degraded soils and soil agricultural productivity (Teglia et al., 2011). Fertilizing by digestate is a great way to possibly achieve lightening and aeration of the soil. Better access of air to the roots and improvement of soil hydro limits can increase the crop yield (Tlustoš et al., 1998). Another way to use digestate is outside the agricultural and forest land as reclamation materials, e.g. on the surface near recreational and sports centers, in urban parks or for the reclamation of industrial zones and landfills. Reclamation digestate must meet some special quality characteristics, such as the maximum moisture content 98% of its weight and the pH should be between 6.5 to 9.0 (MENDELU, 2008).

### 3 Aims of the Thesis

Objectives of the thesis were created according to already known facts about soil water sorption and digestate and changes of its properties in soil, which were obtained from scientific literature.

As was already said before, the physical structure of the soil plays a crucial role in the processes that facilitate soil functions such as food production and water retention. The main objective of the thesis was therefore to investigate the possibility of a change in the soil physical properties by volume changes of the digestate briquettes embedded in the soil which could support the idea of returning the organic matter coming from the process of anaerobic digestion back to the soil and wider use of digestate briquettes for non-energetic purpose.

On the basis of the main objective, there were two specific objectives of the thesis determined:

- To monitor how the retention capacity of briquettes changes in different types of soil and its influence on the soil physical properties
- To quantify changes in the volume of digestate briquettes depending on time, moisture and type of soil

In order to achieve specific objectives the following hypotheses were stated:

- $H_0$ : There is no statistically significant difference between the moisture of briquettes in loamy soil and the moisture of briquettes in loamy sand soil
- $H_0$ : There is no significant linear relationship between moisture of soil and moisture of briquettes
- $H_0$ : There is no statistically significant difference between the change in volume of briquettes in loamy soil and the change in volume briquettes in loamy sand soil
- $H_0$ : There is no significant linear relationship between change in volume of briquettes and moisture of briquettes

## **4 Material and Methods**

First of all, secondary data were explored to summarize literature review and give a clear idea, how to create research design to obtain primary data. After all the experiments were carried out and all the necessary data were collected, the data were firstly organized and then processed by using Microsoft Office Excel 2010. Furthermore, moisture content and density were calculated. To exploit the data was also used software STATISTICA 12.

### **4.1 Secondary data collection**

Literature review has been based on data obtained from scientific articles placed in scientific databases as Web of Science, Science Direct and Scopus. As principal key words for searching in those databases were: water sorption, digestate, volume changes of briquettes, density of digestate briquettes, type of soil, loam, loamy sand

### **4.2 Primary data collection**

#### **4.2.1 Material**

There are two materials, which have been used for further experiments: digestate and miscanthus, both in form of briquettes. Digestate, as the crucial material for running all the experiments, derived from commercial biogas plant in Krásná Hora nad Vltavou, Petrovice farm, Czech Republic. The biogas plant is a large-scale plant, using technology of company FARMTEC a.s., with an output of 800 kW. The digester of the BGS was fed (with a possibly error of about 5%) by 30% maize forage, 35% of cattle slurry and 35% grass cuttings. The grass cuttings and corn forage were put fresh into the digester. The sample was collected in spring 2014 and delivered already in solid form with moisture content between 78 – 80%. The other material, *Miscanthus sinensis*, was used as the comparative material due to digestate briquettes properties and was chosen because of material qualities similar to digestate and also with regard to its availability in the Czech Republic. Materials were both tested at the same time. Miscanthus is a perennial grass about 4 meters height. Botanically it belongs to the family Poaceae. It can optimally utilize water, nutrients and light assimilation. Average of the yield is around 15-18 t/ha (Holub,

2007). Its yield, elemental composition, carbohydrate and lignin content and composition are of high importance to be reviewed for future biofuel production and development (Brosse et al., 2012).

Both of materials were obtained as raw materials, means both of them had to be compressed into shape of briquettes first. Before compressing, material had to be dried. At the beginning drying was carried out in ambient room temperature in which the material had been stored, followed by drying in a dryer at temperatures up to 95 °C to 14-15% of final moisture content before processing. The granulating process was carried out in April 2014 in premises of the Faculty of Engineering, CULS Prague by using hydraulic piston press BrikStar CS25 (Briklis Ltd., Malšice, Czech Republic) belonging to the FTA. All briquettes were pressed from the stated materials without any other additives and as well as were produced under the same working conditions. Since digestate particles were sufficiently small for crushing, there was no need for a special treatment of the material. Digestate briquettes of diameter 60 mm (D) and different length between 30-50 mm (L) had been produced. In case of miscanthus, its dry stems had to be, prior to pressing, crushed using the hammer crusher 9FQ40C with 3.8 mm diameter sieve. Miscanthus biomass was pressed into briquettes of two diameter sizes – 50 mm and 60 mm and length between 30-50 mm. Briquettes made from digestate and Miscanthus used for running the experiments had laboratory moisture between 8-10%. The mechanical strength of briquettes was determined according to the CEN/TS 15210-2 standard.

#### **4.2.1.1 Specification of the briquettes physical properties**

For the description of briquettes properties, samples were analyzed in the laboratory of CULS and measured values were recorded into tables for further analyses. The nomenclature of properties according to EN ISO 17225 has been used. The analyses were done to assess the effectiveness of briquettes. The following properties were determined within samples (for values obtained see Table 5 below):

Moisture content – Quantity of water contained in briquettes was calculated from differences of briquettes initial and final weights.

Volume and density –Weight, length, diameter of each briquette in initial and terminal phase (before insertion into the soil and after removal from the soil) were measured using a micrometer. The volume of the briquettes was calculated as the volume of a cylinder with the dimensions (length and diameter) measured and density of briquettes was calculated on the basis of these dimension measurement and weighing. Briquette density represents the ratio between the sample mass and its volume, including pore volume. For drawing up the results there were finally used volume measurements, the values which are part of the density formula (mass of material per unit volume).

**Table 5: Characteristics of material used**

Material	Diameter [mm]	Moisture [%]	Calorific value [MJ/kg]	Density [g/cm <sup>3</sup> ]
Digestate	60	8.6	16.18	0.879
Miscanthus	50	11.3	17.81	0.863
Miscanthus	60	6.8	18.12	0.746

Source: Testing made by author and doc. Ing. Josef Pecen, CSc.

Detailed characteristics of digestate used in the experiments were measured apart of the experiments to know exact contents of the material (Table 6) and particles distribution (Table 7). The characteristics of *Miscanthus sinensis* as the material for comparison were summarized from scientific literature. *Miscanthus sinensis* had following characteristics: 2.44% ash, 7.9 g/kg K, 1.5 g/kg Cl, 4.6 g/kg N of dry weight, cellulose (40 to 60 % wt) and lignin (10 to 30 % wt) depending on the harvest period (Brosse et al., 2012).

**Table 6: Nutrient content of digestate [%]**

Material	DM	Ash	N x 6.25	Lipids	Fibre	OM	NFE
Briquettes	91.31	10.90	11.20	0.27	31.74	80.41	37.21
In 100% dry matter	100	11.93	12.27	0.30	34.76	88.07	40.75

DM = Dry Matter

OM = Organic Matter

NFE = Nitrogen-free extract

Source: Testing made by doc. Ing. Josef Pecen, CSc.



**Table 7: Distribution of the digestate in a 1 kg sample according to particle size [%]**

size of the particles [%]							
10 mm	5 mm	2.5 mm	1 mm	0.5 mm	0.25 mm	0.1 mm	< 0.1 mm
5.3	25.5	44.8	11.9	10.5	1.2	0.2	0.1

Source: Sieve analysis made by doc. Ing. Josef Pecen, CSc.

## **4.2.2 Water sorption in soil**

### **4.2.2.1 Water sorption in laboratory conditions**

During the first part of the experiment, which was carried out in laboratories of the CULS, room number C63, in the period of two months in between June 4<sup>th</sup> – May 18<sup>th</sup> 2014 (the first trial) and June 18<sup>th</sup> – 30<sup>th</sup> 2014 (the second trial), we tried to simulate soil conditions by keeping briquettes in wooden boxes, which had been filled in by soil. For reaching more exact results the experiment was repeated.

Digestate and miscanthus briquettes were horizontally placed into two wooden boxed with parameters of 30 cm x 70 cm x 30 cm. Before the briquettes were placed, the bottom and sides of both wooden boxes had to be covered by plastic foil to prevent the wooden material get wet. Briquettes were evenly covered by 25-30 cm wide layer of soil, where each box contained the same amount of briquettes (10 of each) and different kind of soil; see Figure 2. The box marked as S1 sample contained loamy soil. The other box marked as S6 sample contained loamy sand soil. There was no additional watering of soil in boxes when the experiment was running. In every 2-3 days, layers of soil were vertically removed from the box profile with two briquettes of each included and their physical properties, as increase in length, diameter and mass, have been measured. The values were measured five times per trial. The moisture content inside briquettes and moisture content of the soil environment were calculated from values (the initial and final weights) obtained after drying all the collected material in a drying oven, where the studied samples were subsequently dried at  $105^{\circ}\text{C} \pm 3^{\circ}\text{C}$  for 24 hours, until the constant weight is obtained. Those values were then tabulated for further data processing and evaluation.

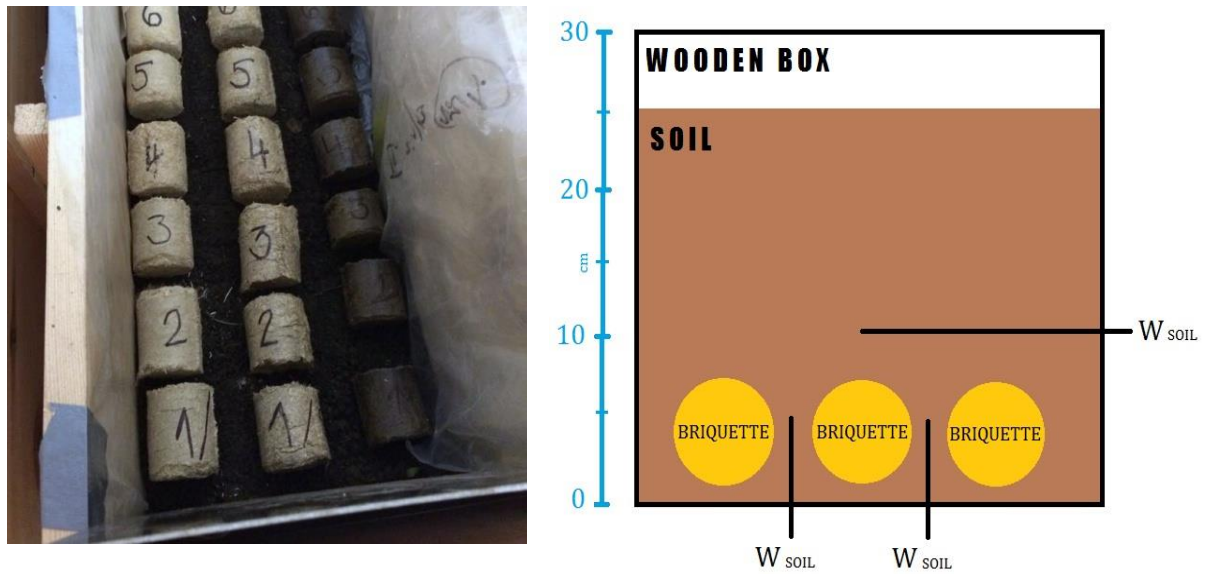


Figure 2: Placement of briquettes in the wooden box (Author)

#### 4.2.2.2 Water sorption in experimental plot conditions

The second part of the experiment took place on the experimental plot, which was established on demonstrative fields belonging to the CULS campus located in the north of Prague, Czech Republic (see Table 8). All the data were in this case collected in between June – October 2014 (the first trial 12<sup>th</sup> – 27<sup>th</sup> June and the second one 7<sup>th</sup> – 31<sup>st</sup> October), were carried. This time period was chosen taking in account the fact that according to Weather station of CULS (2014), in this area nearly 40 percent of the annual total precipitation falls from June to October.

**Table 8: Experimental plot characteristics**

Characteristics	Experimental field ČZU
Region	Prague
Municipality	Prague-Suchdol
Geographical coordinates	50° 7' N 14° 22' W
Altitude	284 m
Average annual temperature	9.3°C
Annual rainfall	472 mm
Soil type	Black soil

Source: Author's compilation based on Weather station of CULS (2014).

Digestate as well as miscanthus briquettes, were placed in three rows in total amount of 18 briquettes each into a 30 cm deep ditch and covered by soil; see Figure 3. The soil had the same composition as S6 sample used in previous experiment in laboratory. Three briquettes of each were taken every 3-5 days (interval was depending mainly on current weather conditions – in general six times per trial) and the same physical properties as in case of laboratory experiment (briquettes length, briquettes diameter, moisture of briquette and soil environment) have been measured. The dry matter content of digestate samples was determined by weight loss after drying in drying oven at  $105\text{ °C} \pm 3\text{ °C}$  for 24 hours, according to “ČSN P CEN/TS 14774-1 (-2, -3)” standard used in the laboratory of the FTA. As in the case of previous experiments, all obtained values were tabulated.

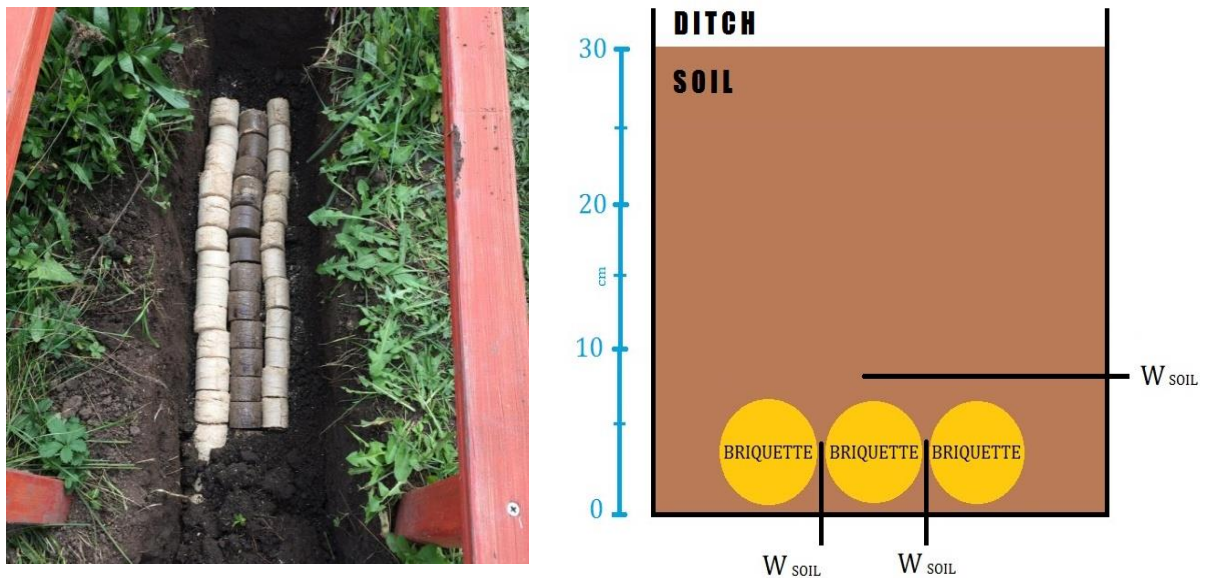


Figure 3: Placement of briquettes outside in the ground (Author).

#### 4.2.3 Methods of analyses

##### 4.2.3.1 Volume and density determination

The volume of each briquette was calculated from using formula for measured values of length and diameter (Eq. 1). For density calculation was used formula counting with obtained values of mass and volume (Eq. 2).

$$V = \frac{\pi D^2}{4} \times L \text{ [m}^3\text{]} \quad (1)$$

$$\rho = \frac{M}{V} \text{ [kg/m}^3\text{]} \quad (2)$$

where:  $V$  – Volume [m<sup>3</sup>]

$D$  – Diameter [m]

$L$  – Length [m]

$M$  – Mass [kg]

$\rho$  – Density [kg/m<sup>3</sup>]

#### 4.2.3.2 Moisture content determination

The moisture content of briquettes was determined according to the ČSN P CEN/TS 14774-1 using the oven drying method. The percentage of moisture content of briquettes and the moisture content of soil were calculated from the differences of the oven dry weight of the sample and the fresh weight of the sample, according to a following equation (Eq. 3):

$$w = \frac{m_w - m_d}{m_w} \times 100 \quad (3)$$

where:  $w$  - the moisture content [%]

$m_w$  - the total weight of the sample prior to drying [g]

$m_d$  - the total weight of the sample after drying [g]

#### 4.2.3.3 Statistical analyses

The obtained data were firstly characterized by descriptive statistics and tested for normality. Statistical analyses were carried out with the program Statistica 12.0. First, the descriptive statistics was made to identify and summarize information, processes it in the form of graphs and tables and calculates their numerical characteristics. The normal distribution of the data was checked by the Shapiro–Wilk test. The data of descriptive statistics we processed into several forms: Box Plot and Histogram. Box Plot is a graphical

representation of statistical measures like median, upper and lower quartiles, minimum and maximum data values. Histogram is defined as a display of statistical information that uses rectangles to show the frequency of data items in successive numerical intervals of equal size. The data were subjected to statistical hypothesis testing for T-test for Independent Means ( $P < 0.01$ ) and Time Series Correlation. In t-test for independent means there are two completely different (independent) groups of subjects that we want to compare to determine if they are significantly different from one another. Independent t-test is derived by assuming the mean of the sampling distribution or differences between means is zero for the null hypothesis population. In general, two-sample tests are less powerful because we are forced to estimate characteristics of the population. Time series are understood as sequences of materially and spatially comparable observations and their analysis is a set of methods that are used to describe dynamic systems of time series, or to predict their future behavior. We worked with residuals - residuals in one period are correlated with residuals in previous periods. One of the problems of time series analysis includes the choice of a suitable length of time series. The accuracy of work and the informative value of indicators are influenced by the length of time series (number of measurements). In correlation, we recognize three most important values: The *R-square* value, which is an indicator of how well the model fits the data, the correlation coefficient *R* as a degree to which a predictor (*X*) is related to the dependent (*Y*) variable and *B* coefficients which interpret the direction of the relationship between variables (StatSoft, 2015).

## 5 Results

All the experiments describing digestate sorption properties were carried out in completely limited environment of the soil. This means the briquettes were accompanied by the occurrence of extensive and stable pressure (smaller than 1 MPa). These limited space conditions leads to a tension and a visible deformation of this space (Pecen et al., 2014). The overall process of the briquettes sorption is defined by two main characteristics – retention capacity of briquettes increase in time and volume change of briquettes in time. These dependencies are expressed in detail by line charts in Chapter 5.1, 5.2 and 5.3 below. For the all experiments, the stated results are results of multiple repeating. The stated data in the line charts, because of the small size of the every set of data which contained only two or three values per the exact period obtained have represented the arithmetic values of the monitored quantity, all the statistical testing have been calculated using median values of the data sets. The overall research was focused on determining the sorption qualities of the digestate and also of one more material with similar properties for the purpose of comparison. For experiments, which were carried on in laboratory conditions, there were two different types of soil used: S6 – loamy soil and S1 – loamy sand. For exact analysis of soil particles distribution of each soil type used see Annex 1.

### 5.1 Water sorption in laboratory conditions

#### 5.1.1 Water retention capacity

The briquettes water content and soil water content in the area around briquettes was calculated for each briquette before inserting and after removal from the containers using the weight of the sample after removal from soil ( $m_w$ ) and weight of the sample after drying ( $m_d$ ). From 60 values for S6 and 60 values for S1 central tendency and variability were determined. For detailed values see Annex 2 and Annex 3. The average moisture of briquettes after the removal from S6 sample was 19.01% and 16.27% after the removal from S1 sample. Even higher the arithmetic means were in case of digestate: 20.29% for S6 and 16.63% for S1. To be sure the arithmetic mean did not distort the results, Box Plot was constructed and median calculated. See Figure 4. Measured moistures of briquettes are shown on Y- axis. The confidence limit for the mean was calculated on the significance level  $\alpha = 0.05$ . We can say with 95% confidence the average briquettes moisture after

sorption was between 17.87% and 20.15% in case of S6 (then highest one for digestate briquettes 17.99% - 22.59%) and in between 15.28% and 17.25% in S1 (14.72% - 18.55% in case of digestate briquettes). The edges of the box are the 25th and 75th percentiles. There were 25% of the samples with a value less than or equal to 16.30% (S6), 13.95% (S1) and 75% of the values is smaller than or equal to 22.24% (S6), 18.57% (S1). The data does not show any presence of extremes or outliers in the collected data. Non-outlier range was in the interval (8.17; 27.46% for S6 and 7.84; 22.67% for S1). The central mark is the median, which was in both cases situated around the center of a rectangle; it means data were in normal distribution. This is also confirmed by the fact, the values of arithmetic mean were close to values of median (19.67% for S6 and 16.75% for S1). Further, the histogram was made from obtained values, which also confirmed the normal distribution of the sample (see Annex 4 and Annex 5). This result was finally confirmed by Shapiro-Wilk test, accepting the null hypothesis ( $H_0$  = the data under investigation comes from the population with normal distribution) where in both cases *p-value* 0.450 (S6) and 0.091 (S1) was greater than significance level  $\alpha = 0.05$ .

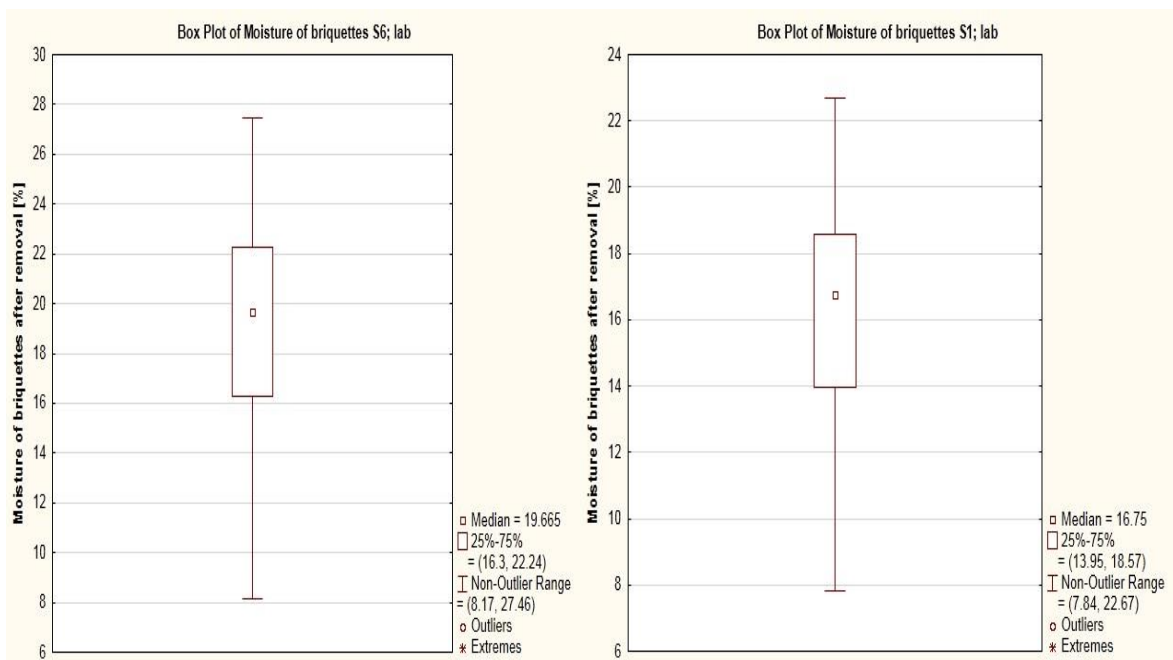


Figure 4: Box plot of moisture of briquettes after removal from soil in S6 sample and in S1 sample in laboratory (Author)

The sorption process in two different types of soil is graphically described in Figure 5 and 6 with data from first set of trials (4.6.2014). From the obtained and further

calculated values (see Annex 17 and 18), line charts were processed to draw up the dependence of moisture gain (in case of the briquettes) and moisture loss (in case of soil) over the time period. The similar results were obtained when the trials were repeated in two weeks interval after the first set of trials (see Annex 19 and 20). It was observed the sorption might run differently in the different type of soils. While the decrease in the soil moisture in the area around briquettes has shown an uniform decrease for both types of soil in about 2.25-2.5% of the moisture content from the initial values (before the briquettes were insert into the soil environment), increase in the moisture of briquettes at the end of the testing period varied from 15.1% in S6 sample to 10.5% in S1sample in total briquettes moisture content compared to initial values. The higher retention capacity of briquettes stored in the soil was observed in the S6 sample with higher soil moisture. Regardless the type of soil or the soil moisture, the highest performing retention capacity occurred in digestate briquettes, when the increase in their moisture was 18.45% for S6 and 12.19% for S1 compared to miscanthus  $\varnothing$  50 mm briquettes (11.97% for S6 and 7.72% for S1) and miscanthus  $\varnothing$  60 mm (15.02% for S6 and 11.85% for S1 sample). The sorption process stopped approximately after 12 – 14 days, when the briquettes reached their maximum retention capacity, were not able to sorb water anymore, so were followed by a decreasing tendency in graph which describe the ability of briquette desorption (visible in Figure 6).

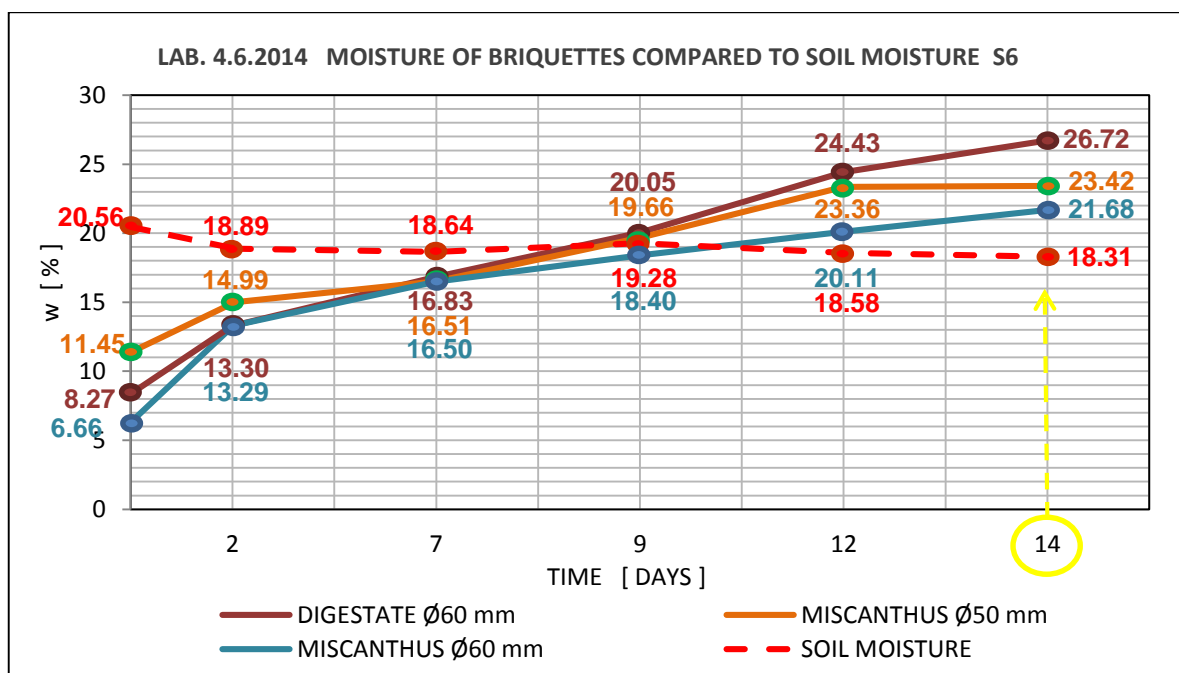


Figure 5: Increase in digestate and miscanthus briquettes moisture and decrease in soil moisture in time, S6 sample in laboratory (Author)



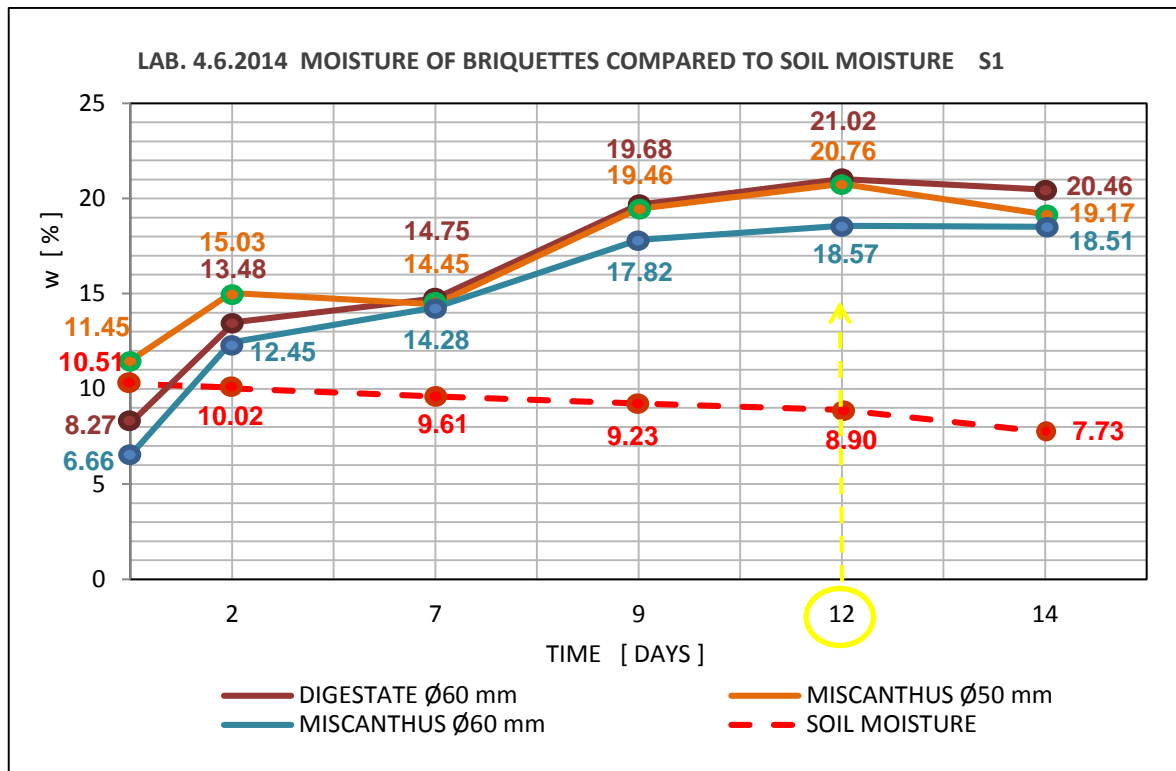


Figure 6: Increase in digestate and miscanthus briquettes moisture and decrease in soil moisture in time, S1 sample in laboratory (Author)

### 5.1.1.1 Comparison of water uptake in loamy soil (S6) and loamy sand soil (S1)

To confirm the assumption, if there is the difference between the means of two moisture samples different enough to say that some other characteristic (type of soil) could have caused it, *P-value* was tested by the use of the T-Test for Independent Means. See the values in Table 9. The null and alternative hypotheses for the moisture of briquettes after the removal from the soil were stated:

- $H_0$ : There is no statistically significant difference between the moisture of briquettes in loamy soil and the moisture of briquettes in loamy sand soil
- $H_1$ : There is a statistically significant different between the moisture of briquettes in loamy soil and the moisture of briquettes in loamy sand soil

**Table 9: T-test for Independent Samples – Moisture of briquettes S6 and S1, lab**

		T-test for Independent Samples – Moisture of briquettes S6 and S1 [%] lab.								
Group 1 vs. 2		Mean Group 1	Mean Group 2	t-value	df	p	Valid N Group 1	Valid N Group 2	Std.Dev. Group 1	Std.Dev. Group 2
W briq. S6 vs. S1		19.00	16.26	3.64	118	0.0003	60	60	4.39	3.81

Source: Author

Among the briquettes after the sorption in the soil, there was a statistically significant difference between the moisture of briquettes in S6 sample and the moisture of briquettes in S1 sample, S6 ( $M = 19.01$ ,  $SD = 4.40$ ) and P1 ( $M = 16.27$ ,  $SD = 3.81$ ),  $t(118) = 3.65$ ,  $p \leq 0.01$ .

**$p < \alpha$ ;  $H_0$  is rejected**

Therefore, we reject the null hypothesis that there was no difference in between the moisture of briquettes in S6 sample and the moisture of briquettes in S1 sample. With the significant difference at  $p = 0.01$  we can be reasonably confident that the samples do differ from one another, a difference at 99% level is "highly significant".

### 5.1.1.2 Dependence of the moisture of soil on the moisture of briquettes

Regression function was calculated for two types of soil – loam (S6) and loamy sand (S1). Since the both variables were primary dependent on time, the regression was calculated through residual values of the variables, which are the deviation of a particular point from the regression line.

The results of regression for S6 are listed in Table 10. An *R-square* was 0.18. It means that the variability of the Y values around the regression line were 1 - 0.18 times the original variance. The correlation coefficient R was 0.42 which shows a medium dependence. The regression coefficient for dependent variable moisture of S6 sample was -0.190, which shows a negative dependence on the moisture of briquettes stored in soil. The regression equation was  $y = - 0.00 - 0.19x$ . If the size of independent variable changes by one unit, the value of the dependent variable would be changed on average by regression coefficient. It means, for example if the moisture of briquettes increases of 10%, the moisture of soil decreases of 1.9%.

**Table 10: Regression summary for moisture of soil S6**

		Regression Summary for Dependent Variable: Residual w soil (S6)					
		R= .42312223 R2= .17903242 Adjusted R2= .16487781 F(1.58)=12.648 p<.00076 Std.Error of estimate: 1.0216					
N=60		b*	Std.Err. of b*	b	Std.Err. of b	t(58)	p-value
	Intercept			-0.0000	0.1318	-0.0000	0.9999
	Residual w briquettes S6	-0.4231	0.1189	-0.1896	0.0539	-3.5564	0.0007

Source: Author

The results of regression for S1 were tabulated into Table 11. An *R-square* is 0.28 so we know that the variability of the Y values around the regression line was 1 - 0.28 times the original variance. The correlation coefficient was 0.53 which shows a medium dependence. The regression coefficient for dependent variable was -0.130, which shows a negative dependence on the moisture of briquettes. The regression equation was  $y = -0.00 - 0.13x$ . It means, for example if the moisture of briquettes increases of 10%, the moisture of soil decreases of 1.3%.

**Table 11: Regression summary moisture of soil S1**

		Regression Summary for Dependent Variable: Residual w soil (S1)					
		R= .53399474 R2= .28515038 Adjusted R2= .27282539 F(1.58)=23,136 p<.00001 Std.Error of estimate: .49392					
N=60		b*	Std.Err. of b*	b	Std.Err. of b	t(58)	p-value
	Intercept			-0.0000	0.0637	-0.0000	0.9999
	Residual w briquettes S1	-0.5339	0.1110	-0.1282	0.0266	-4.8099	0.0000

Source: Author

To evaluate obtained results, the null and alternative hypothesis has been stated:

- $H_0$ : There is no significant linear relationship between moisture of soil and moisture of briquettes
- $H_1$ : There is a significant linear relationship between moisture of soil and moisture of briquettes

**$p < \alpha$ ;  $H_0$  is rejected**

Since the P-value for S6 (0.0007) and S1 (0.0001) were less than the significance level (0.05), the null hypothesis was rejected (95% confidence interval limits do not include 0).

### 5.1.2 Change in volume

Basic statistics was determined from two samples: 60 values of volume change in S6 sample and 60 values for S1 sample. Exact values were tabulated in Annex 8 and Annex 9. The average volume increase of briquettes in percentage in S6 sample was 50.41% and 38.17% after the removal from S1 sample. The Box Plot construction and mean calculation did show us, that the arithmetic mean did not distort the results. See Figure 7. The changes in volume are shown on Y- axis. Non-outlier range was in the interval (11.62; 98.52% for S6 and 9.42; 88.94% for S1). There was no presence of extremes or outliers. The 25% of the samples occurred with a value less than or equal to 32.16% (S6), 23.93% (S1) and 75% of the values is smaller than or equal to 64.34% (S6), 51.91% (S1). The confidence limit for the mean was defined on the significance level  $\alpha = 0.05$ . Due to significance level, we are able to say with 95% confidence the average volume change after sorption was between 44.85% and 55.96% in case of S6 and in between 33.35% and 42.98% in S1 sample. In the determination of the median value of the volume change, the greatest inaccuracy is applied, since the briquettes do not have regular shape due to their internal inhomogeneity. Median was located almost in the middle of the rectangle and values of arithmetic mean were close to median (52.11% for S6 and 37.27% for S1). The null hypothesis ( $H_0$  = the data comes from the population with normal distribution) was confirmed by Shapiro-Wilk test, when *p-value* 0.120 (S6) and 0.065 (S1) was greater than significance level  $\alpha = 0.05$ .

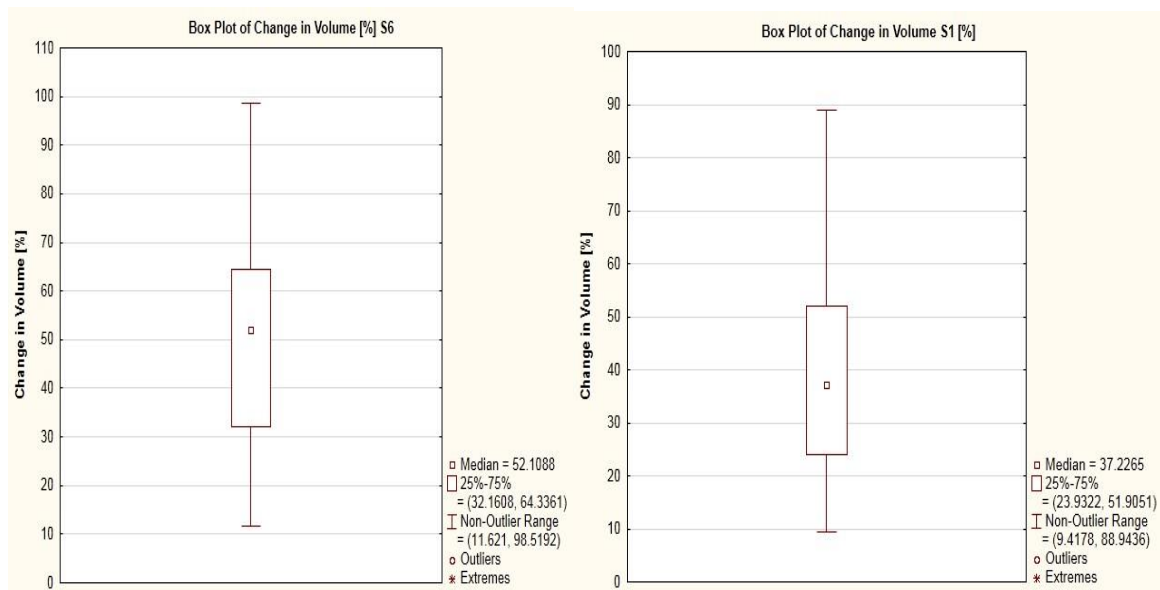


Figure 7: Box plot of change in volume of briquettes in S6 and S1 in laboratory (Author)

From the collected data (tabulated in Annex 17 and 18) the line charts were made to draw the course of the dependence of the volume changes of briquettes in two different types of soil of on time. The enlargement of the original volume of briquettes in the soil due to water uptake briquettes it is graphically described in Figure 8 and Figure 9. The similar course of dependence was also followed by the data, which were collected when the laboratory experiments were repeated in two weeks interval (see Annex 19 and 20). We have observed the change in volume of briquettes might run differently in the different type of soils. The higher increase in volume changes was observed in the S6 sample with higher soil moisture. Digestate briquettes, in both cases, had the greatest volume change, when at the end of the experiment, after 14 days, the increase in volume was 97.12% in S6 and 64.03% in S1 sample. Since the sorption of briquettes stopped approximately after 12 – 14 days, the briquettes are also reaching the maximum volume increase in this time interval.

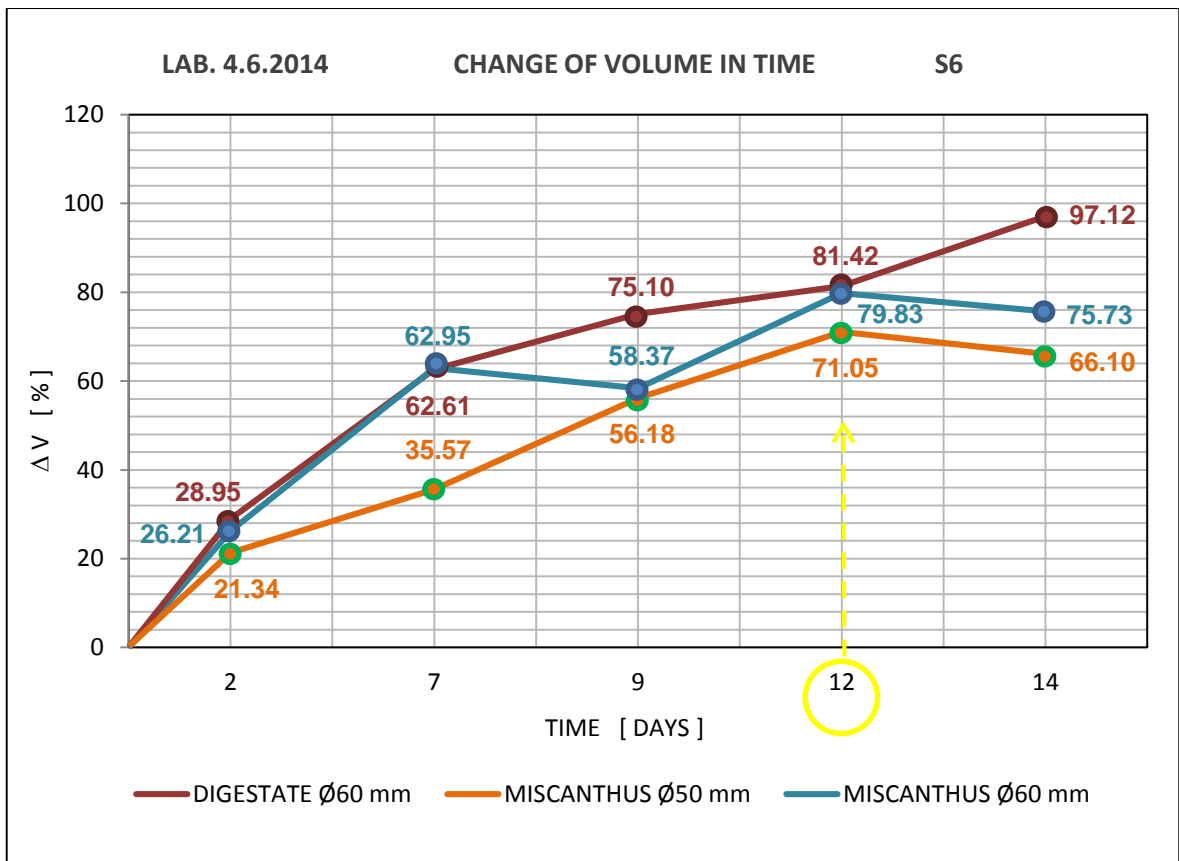


Figure 8: Change in volume of briquettes in time, S6 sample in lab (Author)

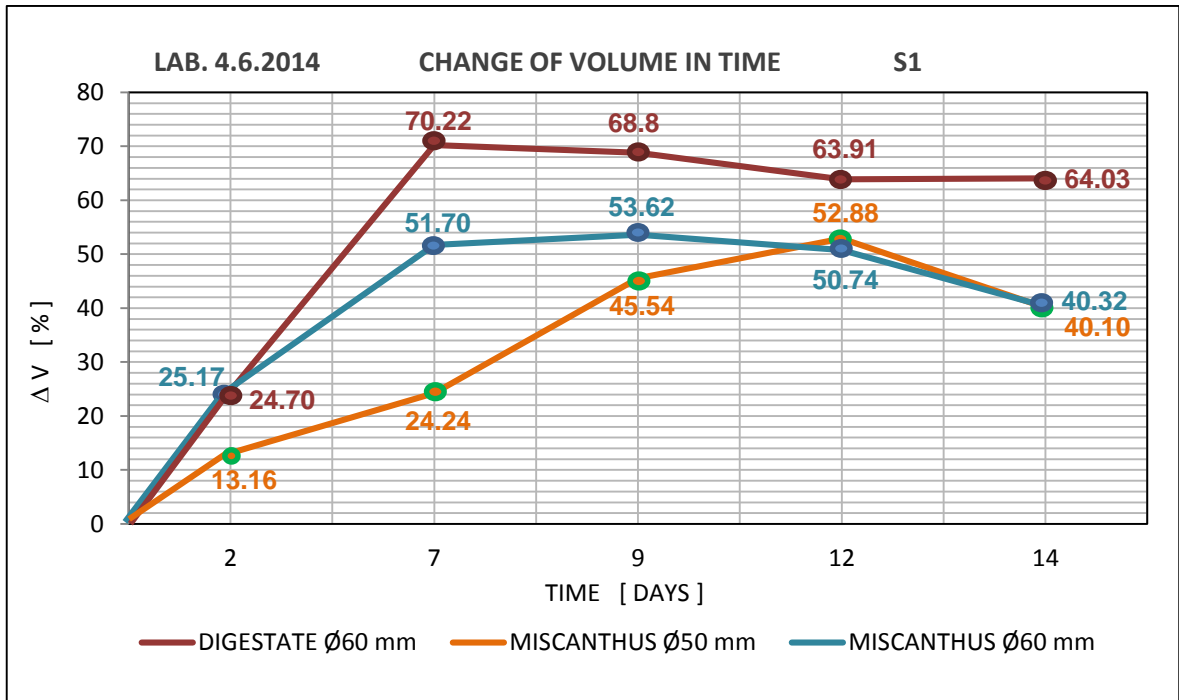


Figure 9: Change in volume of briquettes in time, S1 sample in lab (Author)

### 5.1.2.1 Comparison of change in volume in loamy soil (S6) and loamy sand soil (S1)

To make certain of what was observed from the line charts above, that the briquettes change the volume differently in different types of soil, the null and alternative hypotheses for the changes in volume of briquettes after removal from the soil were determined:

- $H_0$ : There is no statistically significant difference between the change in volume of briquettes in loamy soil and the change in volume briquettes in loamy sand soil
- $H_1$ : There is a statistically significant different between the change in volume of briquettes in loamy soil and the change in volume briquettes in loamy sand soil

**Table 12: T-test for Independent Samples – Volume change of briquettes S6 and S1 sample; lab.**

		T-test for Independent Samples - Volume change S6 x S1 [%] lab.								
Group 1 vs. 2		Mean Group 1	Mean Group 2	t-value	df	p	Valid N Group 1	Valid N Group 2	Std.Dev Group 1	Std.Dev. Group 2
V change S6 vs. S		50.412	38.167	3.334	118	0.001	60	60	21.498	18.632

Source: Author

Under the testing the *P-value* by T-Test for Independent Means (see Table 12), it was found out, that there was a statistically significant difference between the change in volume of briquettes in S6 sample ( $M = 50.41$ ,  $SD = 21.50$ ) and the change in volume of briquettes in S1 sample ( $M = 38.17$ ,  $SD = 18.63$ ), where  $t(58) = 2.28$  and  $p \leq 0.01$ .

**$p < \alpha$ ;  $H_0$  is rejected**

According to the T-Test for Independent Means results, we reject the null hypothesis that there is no statistically significant difference between the change in volume of briquettes in S6 and the change in volume briquettes in S1 sample. The significant difference at  $p = 0.01$  confirms the samples do differ from one another, a difference at 99% level is "highly significant" but there is nearly a 1% chance of being wrong in reaching this conclusion.

#### **5.1.2.2 Dependence of change in volume of briquettes on the moisture of briquettes**

Regression function was calculated for change in volume of briquettes and the moisture of briquettes in loamy soil (S6) and loamy sand soil (S1). Since the both variables were primary dependent on time, the regression was calculated through residual values of the variables.

To evaluate the results, the null and alternative hypothesis has been developed:

- $H_0$ : There is no significant linear relationship between change in volume of briquettes and moisture of briquettes
- $H_1$ : There is a significant linear relationship between change in volume of briquettes and moisture of briquettes

The results of regression for S6 are listed in Table 13 below. The variability of the Y values around the regression line was 1 - 0.04 times the original variance. The correlation coefficient R was 0.22, which implies weak dependence. The regression coefficient for dependent variable of S6 sample was -1.9, which shows a negative dependence on the moisture of briquettes stored in soil.

**Table 13: Regression summary for volume of briquettes S6**

		Regression Summary for Dependent Variable: Residual volume change					
		R= .22184845 R2= .04921674 Adjusted R2= .03282392 F(1.58)=3.0023 p<.08846 Std.Error of estimate: 20.955					
N=60		b*	Std.Err. of b*	b	Std.Err. of b	t(58)	p-value
Intercept				-0.0000	2.7052	-0.0000	0.9999
Residual moist S6		-0.2218	0.1280	-1.8954	1.0938	-1.7327	0.0884

Source: Author

Obtained results during the regression for S1 sample are tabulated in Table 14. The variability of the Y values around the regression line was 1 - 0.01 times the original variance. The correlation coefficient R was 0.10, which implies almost no dependence.

**Table 14: Regression summary for volume of briquettes S1**

		Regression Summary for Dependent Variable: Residual volume change					
		R= .09916994 R2= .00983468 Adjusted R2= ---- F(1.58)=.57608 p<.45093 Std.Error of estimate: 18.728					
N=60		b*	Std.Err. of b*	b	Std.Err. of b	t(58)	p-value
Intercept				0.0000	2.4178	0.0000	0.9999
Residual moist S1		0.0991	0.1300	0.7672	1.0108	0.7589	0.4509

Source: Author

P-value for S6 (0.088) and S1 (0.451) were greater than the significance level (0.05), the slope was close to zero, we accepted the null hypothesis.

**$p > \alpha$ ;  $H_0$  is accepted**

The change in volume of briquettes stored in loamy soil (S6) and the change in volume of briquettes stored in loam sandy soil (S1) were found independent on change of briquettes moisture in the same soil sample.



## 5.2 Water sorption in experimental plot conditions

The trials on the experimental plot were carried out in two different periods of the year (June and October). These months were chosen due to expectation of different amount of precipitation over the periods when the experiments were running and its expected influence of the soil moisture and briquettes sorption properties. These expectations were based on the data, which were collected by Weather Station of CULS the year before our experiments were carried out, so the research was designed according to those data.

The central tendency and variability was obtained from 54 values for each of characteristics (moisture, volume) measured during the experiment in June and for 54 values measured during the experiment in October (values are tabulated in Annex 21 and Annex 22). For the detailed values of the descriptive statistics for the moisture content and change in volume see Annex 10 and Annex 11. To be sure the arithmetic mean did not distort the results, median was calculated. The confidence limit for the mean was calculated on the significance level  $\alpha = 0.05$ . We can say with 95% confidence the average briquettes moisture after sorption was between 16.06% and 17.70% and change in volume 30.42% and 37.33% in case of June and in between 50.58% and 61.74% for moisture and 84.10% 128.84% for volume change in October. Data collected during the experiment in October showed the presence of extremes and outliers. The normal distribution was tested by Shapiro-Wilk test, rejecting the null hypothesis ( $H_0$  = the data under investigation comes from the population with normal distribution) where in both cases *p-value* for moisture content of briquettes and change in volume of briquettes were less than significance level  $\alpha = 0.05$ .

The results of the experiments carried out in experimental plot are graphically evaluated in Figure 11 and Figure 12 (for measured data from which the line charts come from see Annex 21 and 22). The course of rainfall during the October trial is displayed in Figure 10. The line charts for moisture of briquettes compared to moisture of soil, the change in volume of briquettes and the course of rainfall during for the June trial are displayed in Annex 14, 15, 16. The soil moisture was in case of both experiments steady, influenced by precipitations. There was no visible decrease in the soil moisture in the area around briquettes over the experimental period as it has been measured within experiments

in laboratory conditions. Digestate briquettes as well as miscanthus briquettes reached their maximum retention capacity in 13 days after insertion into the soil. The large amount of precipitation between 7<sup>th</sup> and 13<sup>th</sup> day of the experiment was crucial for the results of the trial in October, when the moisture of soil shows 7% increase in this period and also briquettes reached their maximum sorption capacity. The maximum reached sorption capacity of briquettes after 13 days was also confirmed by further course of sorption, when the briquettes did not reacted to the rainfall in days 15 and 16 anymore, keeping the constant moisture.

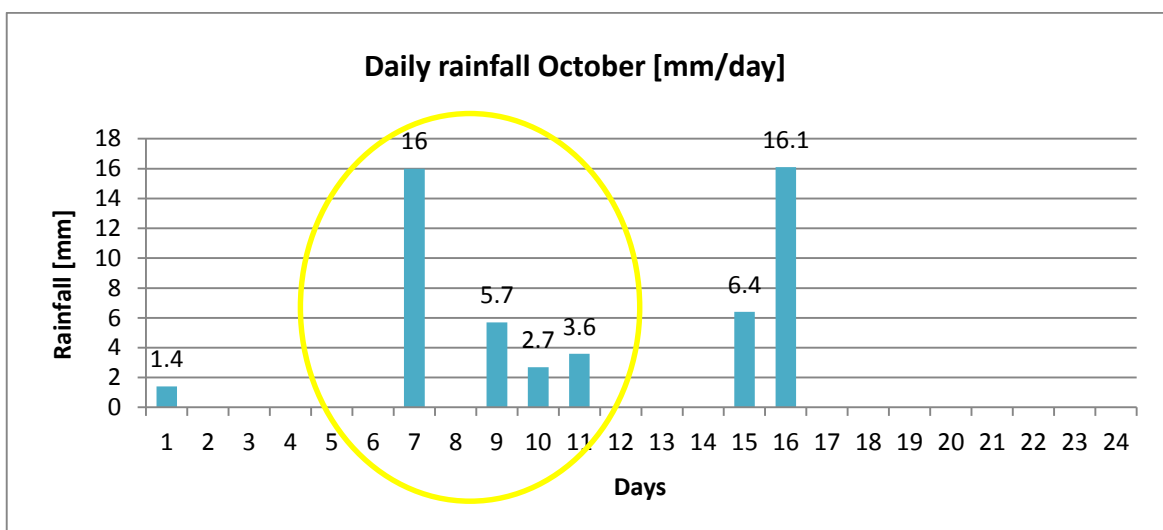


Figure 10: Daily rainfall during the experiment on experimental plot; October

From the Figure 11 below, we can see there was a higher increase in moisture and increase in volume of briquettes over the time during the experiment which was carried out in October, when moisture of digestate briquettes reached level of 69.9% compared to 21.66% and change in volume in June. There was almost no difference in water uptake between different kinds of briquettes used, when digestate gained 57.14% of water, miscanthus  $\varnothing$  50 mm 56.31% of water and miscanthus  $\varnothing$  60 mm 55.37% of water. Although the maximum of water uptake was reached in 13 days, the volume of briquettes was still increasing up to 20th day of the experiment, when change in volume raised up to 254.4% (digestate), 317.09% (miscanthus  $\varnothing$  50 mm) and 124.49% (miscanthus  $\varnothing$  60 mm).

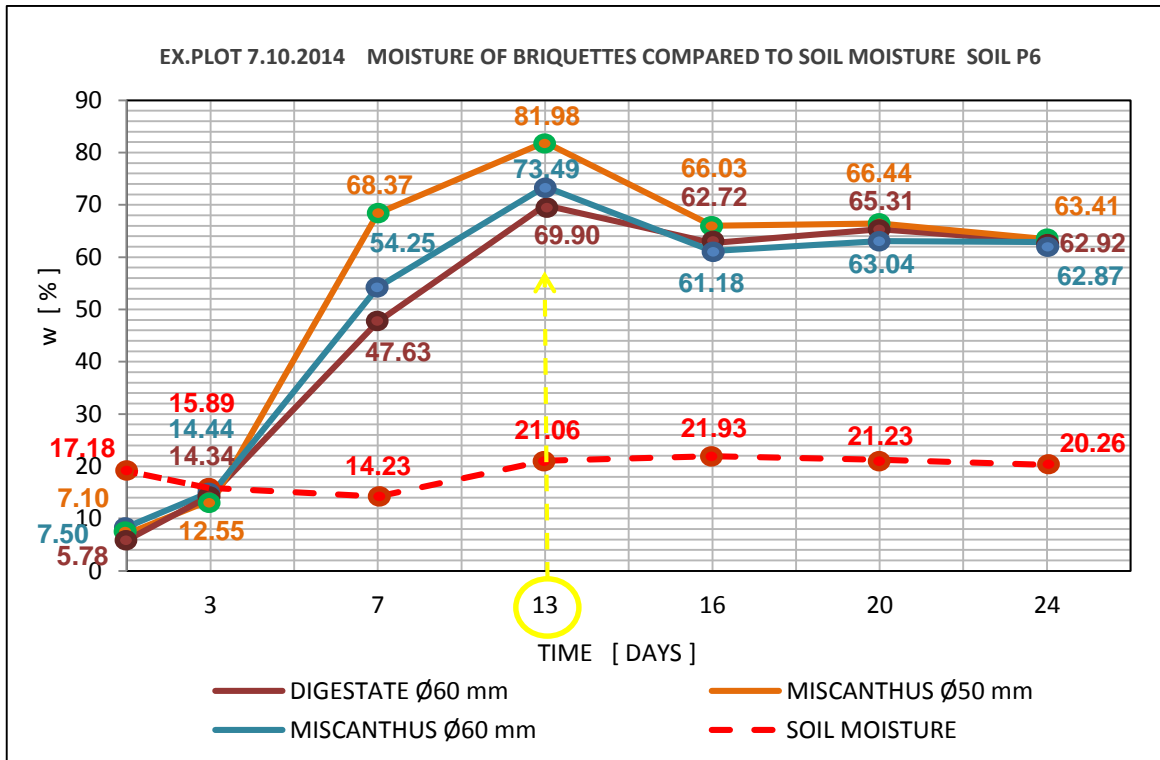


Figure 11: Increase in digestate and miscanthus briquettes moisture and decrease in soil moisture in time, S6 sample in experimental plot, October (Author)

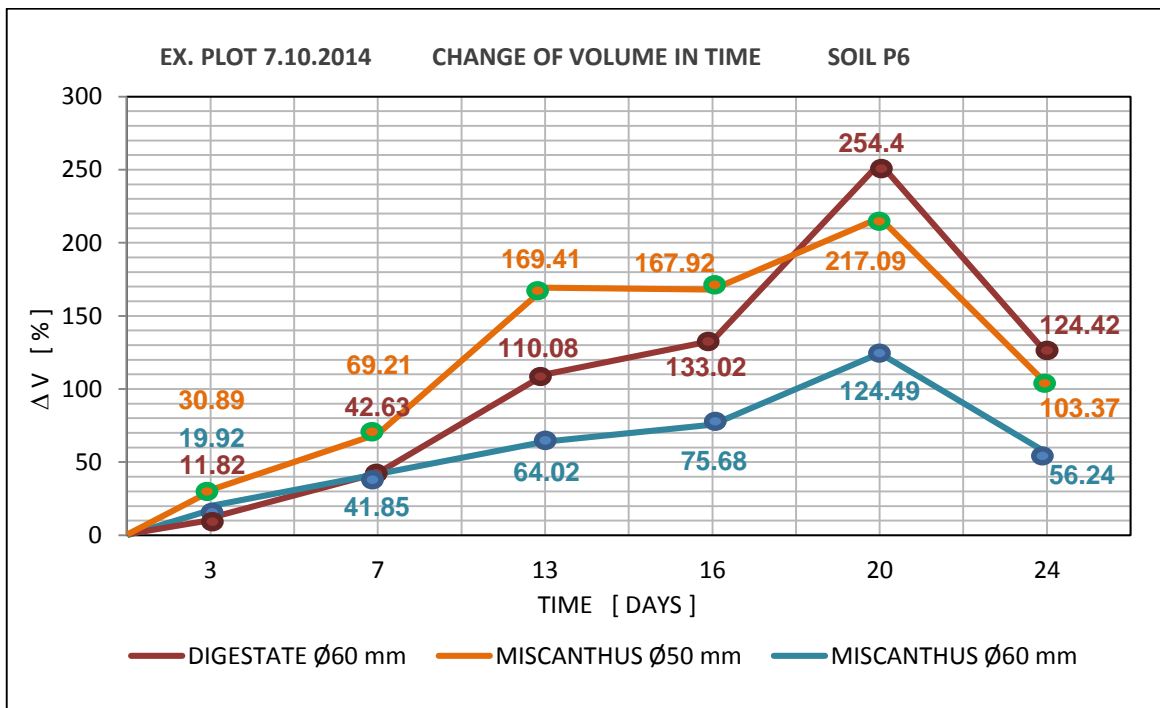


Figure 12: Change of Volume of briquettes in time, S6 sample in experimental plot, October (Author)

## 6 Discussion

Following the obtained results it is possible to discuss over these topics:

### 6.1 Water retention capacity in laboratory conditions

The null hypothesis “There is no statistically significant difference between the moisture of briquettes in loamy soil and the moisture of briquettes in loamy sand soil” was tested by using T-test for Independent Samples. This hypothesis was rejected,  $p < 0.01$ , which means there was a significant difference between water uptake by briquettes in loamy soil and loamy sand soil used during the experiment. Our result confirms the Bortoluzzi et al. (2005) statement that available water content varies widely depending on soil composition and especially soil texture. According to Pecen et al. (2014) the outer conditions have a significant impact on a water retention capacity of the briquettes and on the speed of sorption. From their results it comes out, the briquettes water uptake reaches the maximum in 9 days, which is about 3 days less than what was found out during our experiment where the maximum moisture of briquettes occurs the twelfth day of storage in soil, even though the soil moisture reached the same level in both experiments. We could also observe a better capability of digestate briquettes to hold already sorbed water than it was in case of miscanthus briquettes. The water sorption in soil is predominantly limited by the physical conditions of the soil as a temperature or pressure. It was concluded by Hopmans and Dane (1986) that temperature influences water retention more than can be explained by surface tension changes of pure water only. Porosity of soil was not considered to be the primary cause of the larger temperature effect, which comes more likely from viscosity changes. Karunanithy et al. (2013) also claim the influence of sorption by temperature and relative humidity of the environment. Since the laboratory experiments comparing two different types of soil were running in the same ambient temperature and atmospheric humidity, it is not possible the water retention capacity of briquettes in loamy soil (S6) and loamy sand soil (S1) in our experiment differs because of those two conditions. The influence of the soil pressure depends in particular on the type of soil and moisture of the soil. Davidson et al. (1965) support the idea of the influence of sorption by pressure, analyzing the different moisture content from two soils, a silt loam and sandy loam and claims that the water content depends on the size of the applied capillary pressure. The significant difference of the moisture of briquettes in different types

of soil can be then explained by a different the rate at which water can move through the soil when loamy soils generally having higher moisture than sandy soils and have also a better drainage (FAO, 2015). Nevertheless Pecen et al. (2014) state that with higher compaction of the soil, which refers to different soil textures, the briquette moisture remains almost the same.

To analyze the dependence of the moisture of soil on the moisture of briquettes (taking into account there was a significant difference between water uptakes in soils used), there were regression for loam and loamy sand moisture calculated. The correlation coefficient for S6 sample was 0.42 which shows a medium dependence and the regression coefficient for dependent variable moisture was -0.190; the correlation coefficient for S1 sample was 0.53 which also shows a medium dependence. The regression coefficient for dependent S1 was -0.130. Both regression coefficients claim the negative dependence of the moisture of soil on the moisture of briquettes stored in soil. *P-value* was less than significance level  $\alpha = 0.05$ , the stated null hypothesis ( $H_0$ : There is no significant linear relationship between moisture of soil and moisture of briquettes) was rejected. Results of the regression analyzes confirm the finding of Pecen et al. (2014), that there has been a possibility to use the briquettes as a sorbent in wet soil, since the regression claims, when moisture of briquettes changes by one unit, the moisture of soil would be changed on average by regression coefficient. When analyzing the increase in moisture of briquettes, during our experiments was reached in about 30% less moisture content of briquettes compared to the similar study of Pecen et al. (2014), where the briquettes moisture were stabilized at 60%, i.e., the value was four times higher than the soil moisture, when in our experiment regardless of the kind of material and the soil moisture, the briquettes reached maximum moisture of 26%. Letey (1985) highlights the position of soil water potential, which has higher importance than soil moisture content. Different types of soil have different water potentials, which mean the energy with which is water retained by the soil and energy which is necessary for removing water from the soil differs between types of soil. The similar results as by Pecen et al. (2014) were obtained by Černá (2013), who claims the difference between the soil moisture and briquette moisture varies between 30% - 40%. According to Karunanithy et al. (2013) the moisture content of briquettes from biomass depends upon the species, variety, maturity, porosity and microstructure, specific

surface area, strength of the material and type of processing or treatment the material was subjected to. It means the difference between the moisture uptake during our experiment and the moisture uptake in experiment described by Černá (2013) and Pecen et al. (2014), could be partly caused by different variety of miscanthus plants in case of miscanthus briquettes (*Miscanthus sinensis* versus *Miscanthus x giganteus*) and the different feedstock for used for the digestion in case of digestate briquettes, where composition of our sample was: 30% maize forage, 35% of cattle slurry and 35% grass cuttings, versus 60% corn silage, 10% of pig slurry and 30% manure (Černá, 2013). Nevertheless these slight differences in material could not cause the difference 30% moisture content. The values probably differ because of white sand, by which were the briquettes backfilled and a soft absorbent paper, in which were the briquettes packed for better water uptake (Černá, 2013). Although there is no evidence about the exact properties of the absorbent paper, which was used during the experiment and we are not able to confirm this assumption, the comparison of sorption capacity of briquettes with and without additional material used as an absorbent comes up with possibly interesting finding. If the use of absorbent paper can increase the briquettes water uptake, this combination of biomass briquettes sorption and other absorbent material sorption could be used for absorbing other fluids too, especially oils and greases, e.g. using an oil absorbent paper. An oil sorbent material combined with digestate, but also briquettes from other kinds of biomass, could help with treating of oil spills into soil, which have tremendous effects on environment, ecology, economy, and the society as a whole since the processes involved in cleaning oil spill were normally very complicated and not environmentally friendly. An absorbent of synthetic or natural materials brings an effective cost saving option (Shamsudin, 2015). Especially, when manure is used on farms, it could cause the pharmaceutical chemicals are transported into topsoil and further into groundwater systems (Zhang et al., 2011). The possibility of adsorption of antibiotics from soil by biosolids is still little know, even though there has been several experiments carried out already in different types of soil, since persistence of antibiotics in the terrestrial environment differs from a soil structure it was sorb into (Rabølle and Spliid, 2000). For example, according to Thaller and Kennouche (2005), desorption of tylosin (the most commonly used antimicrobial chemical used in swine, cattle, and poultry production) from silty clay loam soils over the four-day period was less than 0.2% of the amount added into soil, which suggest that once tylosin gets trapped in these soils, there is only a low chance of desorption. The results of the research made by Jeong et al. (2011) indicate a

considerably higher amount of tylosin remained after desorption in the corn field soil than in the forest soil, which was attributed to the high pH and silt content of the former and that biochar amendments enhance the retention and reduce the transport of tylosin in soils.

## 6.2 Change in volume in laboratory conditions

The greatest inaccuracy applies to the determination of the volume of briquettes in all cases of water sorption, since briquettes do not have regular shape due to their internal inhomogeneity. As in the previous case, T-test for Independent Samples was used to test the null hypothesis “There is no statistically significant difference between the change in volume of briquettes in S6 sample and the change in volume of briquettes in S1 sample”. With the *p-value* < 0.01, the null hypothesis was rejected, which means there was a significant difference between the change in volume of briquettes in loamy soil and loamy sand soil during our experiment. The water is absorbed by the briquette up to a limit. As a consequence, the volume of the briquettes increases (Pecen et al., 2014). There was a change in density too and, according to Černá (2013), there is assumption of complete decomposition of briquettes in the soil with the prediction of two years. In case of our experiment the absorption limit in loamy soil was reached in 12 days by miscanthus briquettes and 14 days by digestate briquettes, which also performed the greatest increase in volume compared to miscanthus briquettes, when there was a double size of digestate briquettes reported. The lowest change in volume occurred with miscanthus briquettes with diameter of 50 mm. These briquettes were pressed with a greater strength, which caused their higher density and became less porous and led to the lower performance in volume change. After reaching the sorption limit there was almost no gain in volume of briquettes observed. Even though the maximum in volume was reached when they reached the sorption limit, the briquettes kept about the same constant volume in soil close to their maximum volume till the end of the experiment. In loamy sand soil the briquettes have a steeper increase in volume, when digestate briquettes reaching its volume maximum the 7<sup>th</sup> day of the trial already. This might be cause by the texture of the loamy sand soil, which has, compared to loamy soil, a lower cohesion of soil mechanical particles and lower water retention (Smith, 2000). This could allow briquettes to develop the volume increase faster than in loamy soil regardless the lower moisture of the loamy sand soil. It is also important to mention, with the increasing absorbed water, the briquettes partially fell apart

mechanically, when the greatest instability was reported at digestate briquettes, which was also claimed by Pecen et al. (2014). According to Briklis (2011) the speed of volume changes of briquettes depends on the kind of material and the size of its particles. The dependence of volume changes on a kind of material used is clearly visible only for volume changes in loamy sand soil. In loamy soil, digestate and miscanthus briquettes refer almost to the same sorption speed. Also, the degree of compression of the material and the temperature created during compression play important role when talking about the sorption speed. Kaliyan and More (2010) claim that the main responsible actors in briquettes durability are solid bridges created by highly viscous binders during the densification process. Adhesion forces at the interfaces between the solid particles and the viscous binder, and cohesion forces within the viscous binder can bond the solid particles until the weaker of the two fails and the briquettes changes the volume.

The correlation coefficient for loamy soil (S6) was 0.22, which implies weak dependence and the correlation coefficient for loamy sand soil (S1) was 0.10, which implies almost no dependence. P-value for S6 (0.088) and S1 (0.451) were greater than the significance level (0.05), we accepted the null hypothesis ( $H_0$ : There is no significant linear relationship between change in volume of briquettes and moisture of briquettes). The change in volume of briquettes stored in loamy soil and the change in volume of briquettes stored in loam sandy soil were found independent on change of briquettes moisture in the same soil sample. This finding was in contrary to the Zhang and Guo (2014) statement that increasing the moisture content led to a decreased density, durability, and impact resistance and an increased compressive strength.

### **6.3 Water sorption in experimental plot conditions**

The experiments in the experimental plot were carried out mainly for a comparison of results obtained in laboratory condition, since the loamy soil sample (marked as S6) used for laboratory trials came from the experimental plot, where we carried out the outdoor trials. From the drawn up dependence of the moisture of briquettes on time, it was possible to observe the similar speed of sorption, which regardless the soil moisture reached the maximum after 12 – 14 days. We could also say comparing the course of



sorption in laboratory and outdoor conditions of the trial in June, even though the trial was slightly influenced by rainfalls (which were during the experimental period 14.8 mm only), the increase in the moisture of briquettes was almost uniform except the fact, there was no decrease in the moisture of soil caused by briquettes and their water uptake performed. When comparing these results with the course of water sorption of briquettes during the outdoor experiment in October, again regardless the soil moisture which, despite of the large amount of precipitation among the initial and terminal date of the trial (51.9 mm), was oscillating around the same value as the moisture of soil during laboratory trials, we could notice the moisture of briquettes was four times higher. This briquettes moisture is similar to the moisture of briquettes observed by Černá (2013), when during her laboratory experiments where such an increase in the briquettes moisture were mainly caused by using an absorbing paper. The enormous increase of the briquettes moisture in our October outdoor experiment might be caused by the direction of the sorption, when the precipitation went through the soil profile straight into the briquettes. These findings might open a new possibility of use of biomass briquettes as water deposits in soil under low or variable rainfall conditions, since they are able to catch water before running in groundwater, hold it over a time period and return the water back into the soil slowly (Pecen et al., 2014). FAO (2005) promotes efficient soil moisture management as a good way for improving water-use efficiency, especially in tropics where soils are subjected to a cycle of wetting and drying associated with seasonality (Bruand et al, 2005). It might also be used as a new element in approaches managing soil evaporation by modifying the soil microclimate, when inserting biomass briquettes into soil (FAO, 2005).

## **7 Conclusion and recommendation**

The carried out research over physical properties of digestate and miscanthus briquettes stored in loamy and loamy sand soils has confirmed the good sorption properties of all the examined biomass briquettes, especially highlighting the digestate course of sorption, when this material have performed the highest ability to absorb the water and hold it in the internal space and also the highest increase in its proportions and volume up to 3 times of the initial briquette volume. The trials took a place in two different soils in laboratory and outdoor conditions. Regardless the soil moisture and conditions of the environment, the speed of sorption remained the same, reaching the sorption maximum in 12 - 14 days. The dependence of the water uptake and changes in volume on the soil texture has been proved, when there was a significant difference between changes of mentioned properties in loamy and loamy sand soil. Regression analyzes of values obtained in laboratory conditions have shown the medium negative dependence of the moisture of soil on the moisture of briquettes, confirming our assumption that with the increasing moisture of briquettes the moisture of soil decreases. The similar regression analyzes were made to test the estimated dependence for change in volume of briquettes on moisture of briquettes. It was found out there was no dependence between those two variables.

In outdoor conditions, the water sorption differed mainly due to precipitation, which influenced the moisture of the experimental plot by unsteady addition of water during the experiment. There was no decrease in the soil moisture, but on the other hand the digestate briquettes performed great water storage ability. Even after reaching the maximum retention capacity the briquettes were able to store the adsorbed water over a period. This might have a possible use in dry soils of tropical areas with irregular rainfall, where the briquettes could be used as a water deposit in soils with the fast water flow, when first catching the water before flowing into the ground water and later on releasing the water back into the soil, sustaining the soil water regime. I recommend focusing in further research on more detailed observation of the change in physical and also chemical properties of soil due to digestate and other briquettes storage and also evaluate the economic sustainability of such as new advanced technology of a water regime regulation, especially with emphasis on subtropical and tropical areas.

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### Annex 1: Characteristics of S6 and S1 soil sample

Symbol	Soil type	Granularity				Colour	WRB classification
		0.25-2.0 mm	0.05-0.25 mm	0.01-0.05 mm	< 0.01 mm		
S6	Loam	7	36	42,5	14,9	brownish black	blacksoil
S1	Loamy sand	17	29	35	19	brown	fluvisoil

Source: Testing made by doc. Ing. Josef Pecen, CSc.

### Annex 2: Descriptive Statistics – Moisture of briquettes after removal [%], S6 lab.

Variable	Descriptive Statistics -Moisture of briquettes after removal [%],S6 lab.											
	Valid N	Mean	Confidence -95,000%	Confidence 95,000%	Median	Minimum	Maximum	Variance	Std.Dev.	Coef.Var.	Standard Error	
Misture of briquettes S6	60	19.009	17.872	20.145	19.665	8.170	27.460	19.350	4.395	23.141	0.567	
Digestate	20	20.291	17.992	22.589	20.825	11.480	27.060	24.110	4.910	24.202	1.098	
Mlscanthus 50 mm	20	19.536	17.513	21.559	20.830	9.780	24.400	18.680	4.322	22.123	0.966	
Mlscanthus 60 mm	20	17.599	15.841	19.357	17.775	8.170	22.740	14.113	3.756	21.345	0.840	

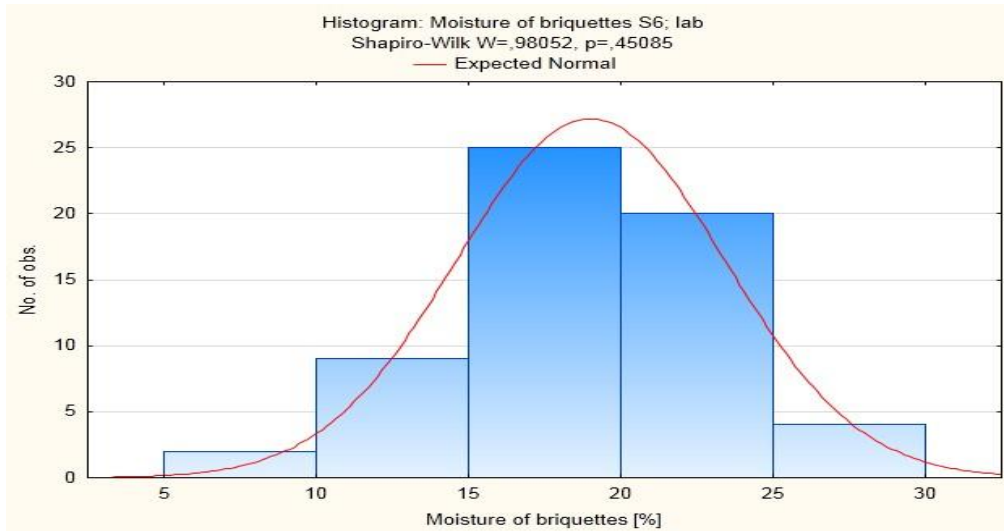
Source: Author

### Annex 3: Descriptive Statistics – Moisture of briquettes after removal [%], S1 lab.

Variable	Descriptive Statistics -Moisture of briquettes after removal [%], S1 lab.											
	Valid N	Mean	Confidence -95,000%	Confidence 95,000%	Median	Minimum	Maximum	Variance	Std.Dev.	Coef.Va	Standard Error	
Moisture of briquettes S1	60	16.266	15.282	17.251	16.750	7.840	22.670	14.522	3.810	23.426	0.491	
Digestate	20	16.633	14.716	18.549	17.285	7.840	21.850	16.772	4.095	24.622	0.915	
Mlscanthus 50 mm	20	16.602	14.893	18.310	17.300	7.950	22.140	13.333	3.651	21.994	0.816	
Mlscanthus 60 mm	20	15.565	14.050	17.079	16.820	8.040	19.410	10.465	3.235	20.784	0.723	

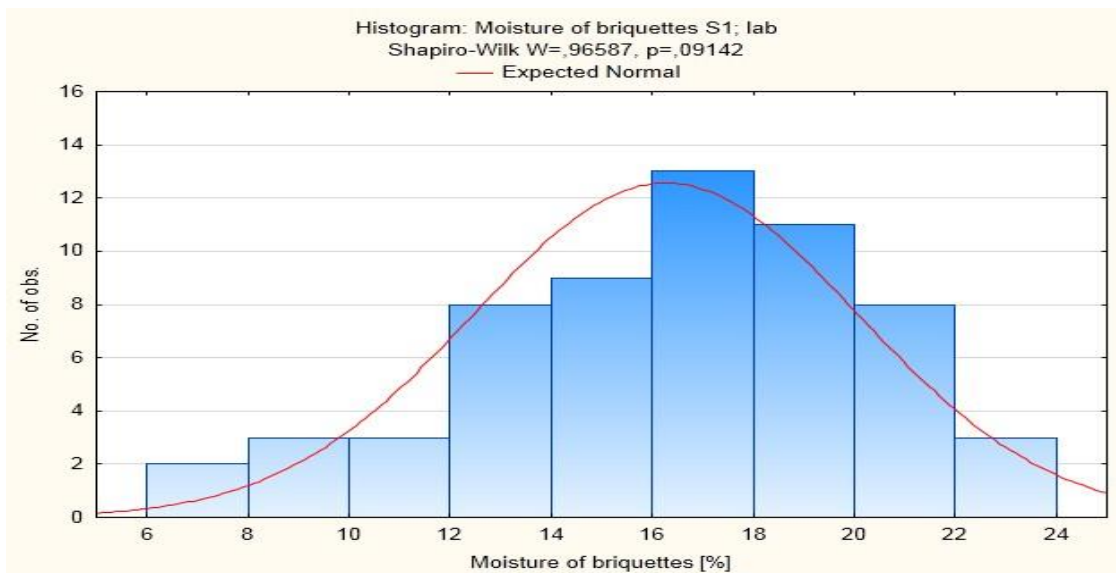
Source: Author

**Annex 4: Histogram – Shapiro-Wilk test for normality testing; Moisture of briquettes after removal, lab. S6**



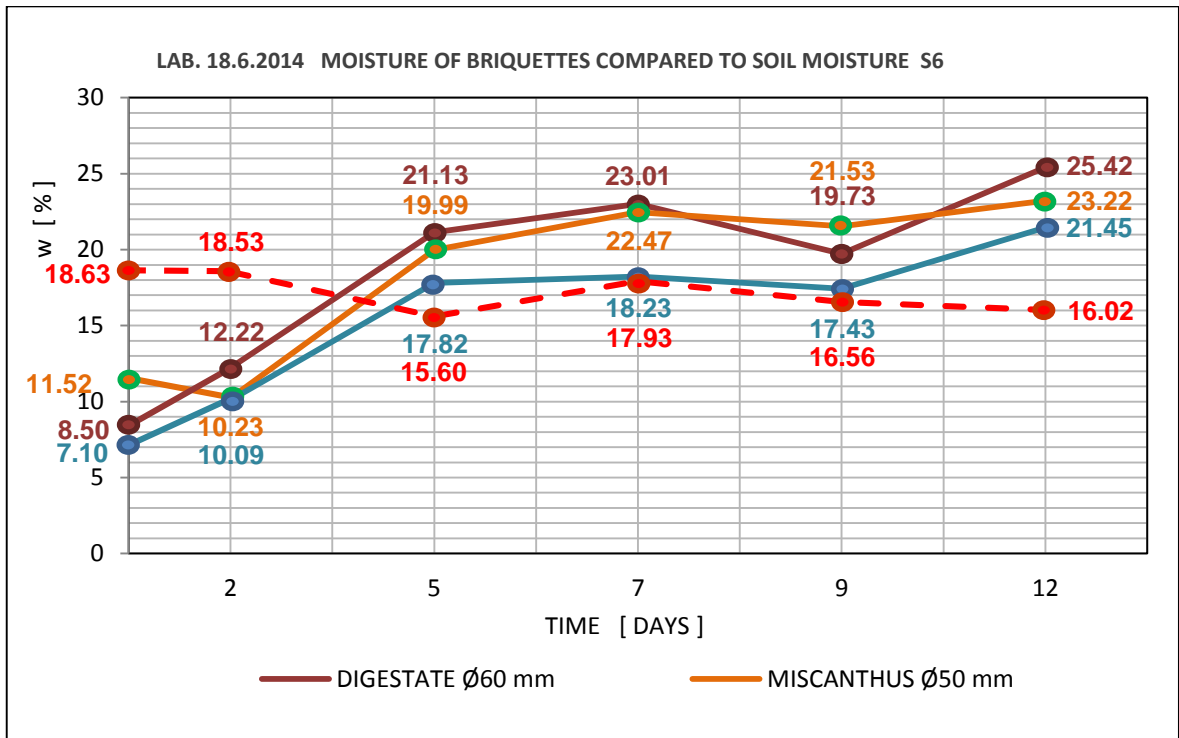
Source: Author

**Annex 5: Histogram – Shapiro-Wilk test for normality testing; Moisture of briquettes after removal, lab. S1**



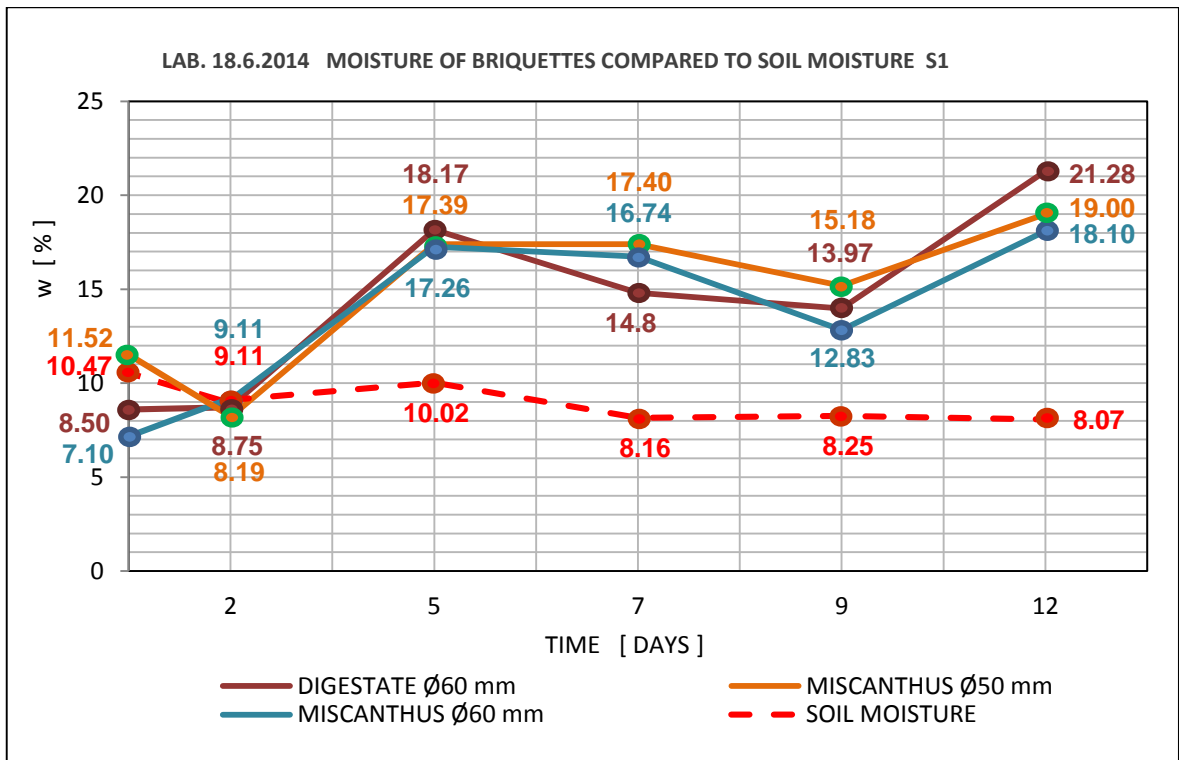
Source: Author

Annex 6: Line chart – Moisture of briquettes to soil moisture, S6, lab. 18.6.2014



Source: Author

Annex 7: Line chart – Moisture of briquettes to soil moisture, S1, lab. 18.6.2015



Source: Author

**Annex 8: Descriptive Statistics - Volume change of briquettes in S6 [%]; lab.**

Variable	Descriptive Statistics - Volume change of briquettes in S6 [%] lab.										
	Valid N	Mean	Confidence -95,000%	Confidence 95,000%	Median	Minimum	Maximum	Variance	Std.Dev.	Coef.Var	Standard Error
Volume change S6	60	50.412	44.859	55.966	52.108	11.627	98.519	462.194	21.498	42.645	2.775
Digestate	20	58.536	48.418	68.654	58.923	28.832	98.519	467.337	21.617	36.930	4.833
Miscanthus 50 mm	20	43.440	34.053	52.828	37.020	11.627	87.437	402.320	20.057	46.172	4.485
Miscanthus 60 mm	20	49.267	39.404	59.118	47.852	17.066	91.083	443.548	21.060	42.752	4.709

Source: Author

**Annex 9: Descriptive Statistics - Volume change of briquettes in S1 [%]; lab.**

Variable	Descriptive Statistics - Volume change of briquettes in S1 [%] lab.										
	Valid N	Mean	Confidence -95,000%	Confidence 95,000%	Median	Minimum	Maximum	Variance	Std.Dev.	Coef.Var	Standard Error
Volume change S1	60	38.167	33.354	42.987	37.226	9.417	88.943	347.179	18.632	48.817	2.405
Digestate	20	48.236	39.058	57.414	50.679	14.317	88.943	384.577	19.610	40.654	4.385
Miscanthus 50 mm	20	27.217	19.716	34.718	23.932	6.539	62.116	256.866	16.027	58.885	3.583
Miscanthus 60 mm	20	38.849	31.976	45.722	37.226	16.884	61.728	215.663	14.685	37.807	3.283

Source: Author

**Annex 10: Descriptive Statistics – Moisture of briquettes after removal [%], exp. plot June and October**

Variable	Descriptive Statistics - Moisture of briquettes after removal, experimental plot June (12.6.2014) and October (7.10.2014)										
	Valid N	Mean	Confidence -95,000%	Confidence 95,000%	Median	Minimum	Maximum	Variance	Std.Dev.	Coef.Var.	Standard Error
<b>Moisture of briquettes June</b>	54	16.886	16.065	17.702	16.505	11.780	23.780	8.944	2.990	17.711	0.406
Digestate June	18	17.656	16.247	19.070	16.915	13.630	22.000	8.091	2.844	16.111	0.670
Miscanthus 50 mm June	18	17.102	15.475	18.725	16.325	12.960	23.780	10.651	3.263	19.083	0.769
Miscanthus 60 mm June	18	15.900	14.544	17.256	14.820	11.780	20.830	7.436	2.727	17.150	0.642
<b>Moisture of briquettes October</b>	54	56.158	50.580	61.736	63.295	10.490	83.330	417.637	20.436	36.389	2.780
Digestate October	18	53.802	44.047	63.562	62.190	13.690	73.070	385.224	19.627	36.480	4.626
Miscanthus 50 mm October	18	59.796	48.535	71.057	65.980	10.490	83.330	512.807	22.645	37.870	5.337
Miscanthus 60 mm October	18	54.877	45.155	64.607	61.815	13.940	74.770	382.364	19.554	35.632	4.608

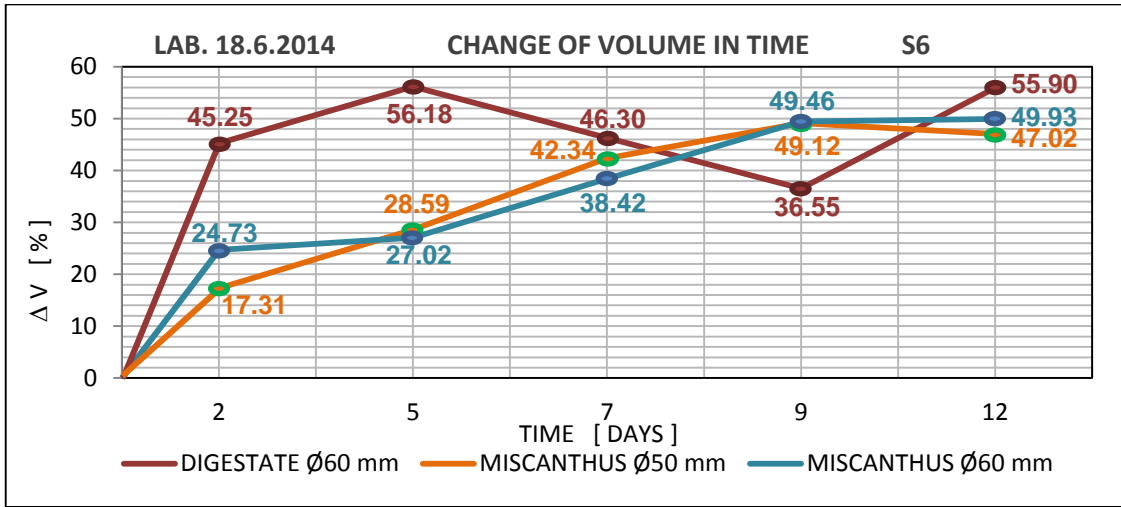
Source: Author

**Annex 11: Descriptive Statistics – Change in Volume [%], exp. plot June and October**

Variable	Descriptive Statistics - Change in Volume of briquettes; experimental plot June (12.6.2014) and October (7.10.2014)										
	Valid N	Mean	Confidence -95,000%	Confidence 95,000%	Median	Minimum	Maximum	Variance	Std.Dev.	Coef.Var.	Standard Error
<b>Change in volume June</b>	54	33.892	30.452	37.333	33.1	2.81	69.51	158.897	12.605	37.197	1.715
Digestate	18	38.348	32.037	44.659	38.735	16.00	69.51	161.060	12.690	33.093	2.991
Miscanthus 50 mm	18	27.793	21.527	34.060	29.20	2.81	56.28	158.797	12.607	45.338	2.970
Miscanthus 60 mm	18	35.536	30.267	40.805	34.28	20.93	65.08	112.266	10.595	29.810	2.497
<b>Change in volume October</b>	54	106.470	84.102	128.839	94.395	6.06	340.67	6716.307	81.953	76.972	11.152
Digestate	18	112.730	71.235	154.225	116.545	6.06	310.17	6962.638	83.442	74.019	19.667
Miscanthus 50 mm	18	142.982	94.854	191.110	135.725	26.72	340.67	9366.500	96.780	67.687	22.817
Miscanthus 60 mm	18	63.7	46.330	81.069	60.735	16.35	136.88	1219.990	34.928	54.832	8.232

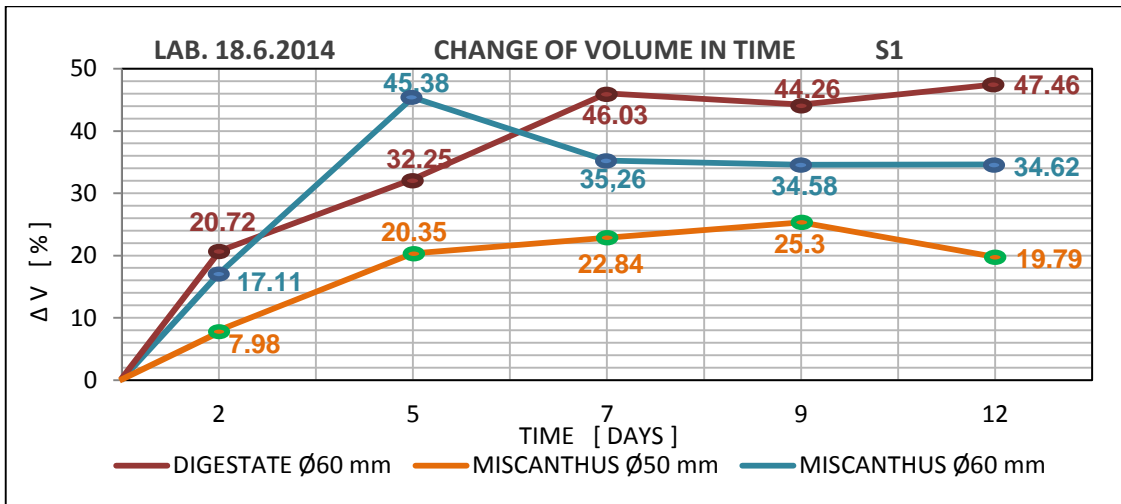
Source: Author

**Annex 12: Line chart – Change of Volume in Time, S6, lab. 18.6.2014**



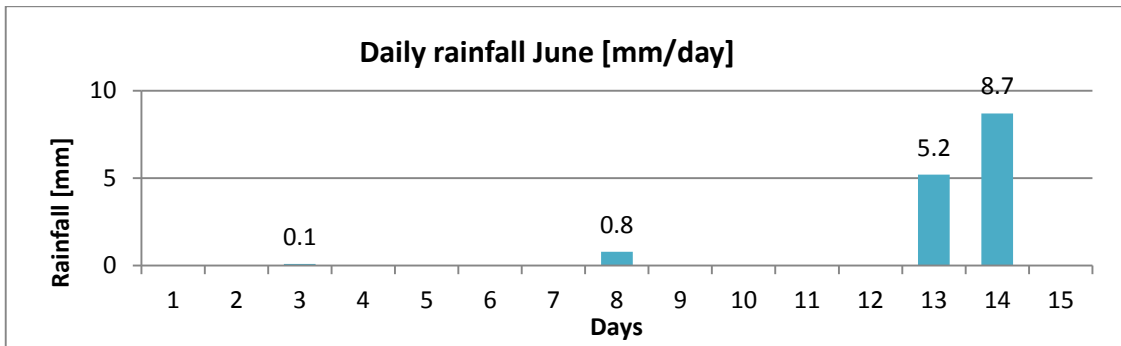
Source: Author

**Annex 13: Line chart – Change of Volume in Time, S1, lab. 18.6.2014**



Source: Author

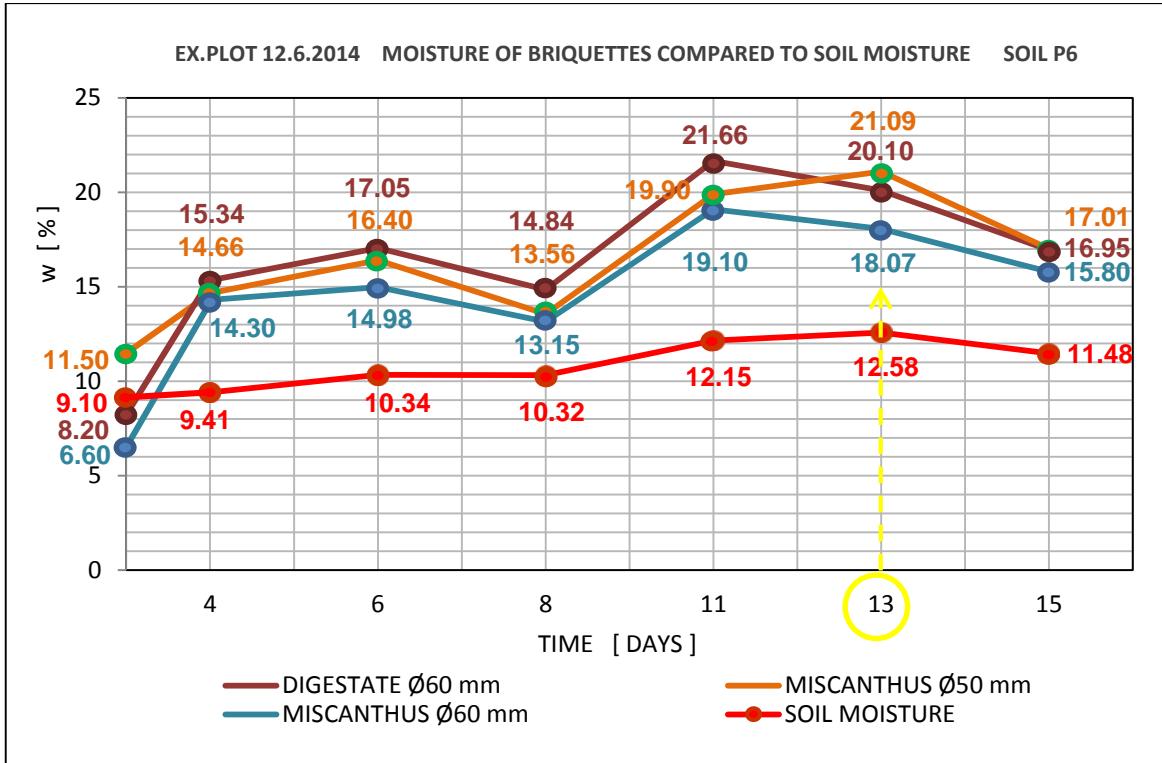
**Annex 14: Daily rainfall during the experiment on experimental plot; June**



Source: Author's compilation based on Weather station of CULS (2014)

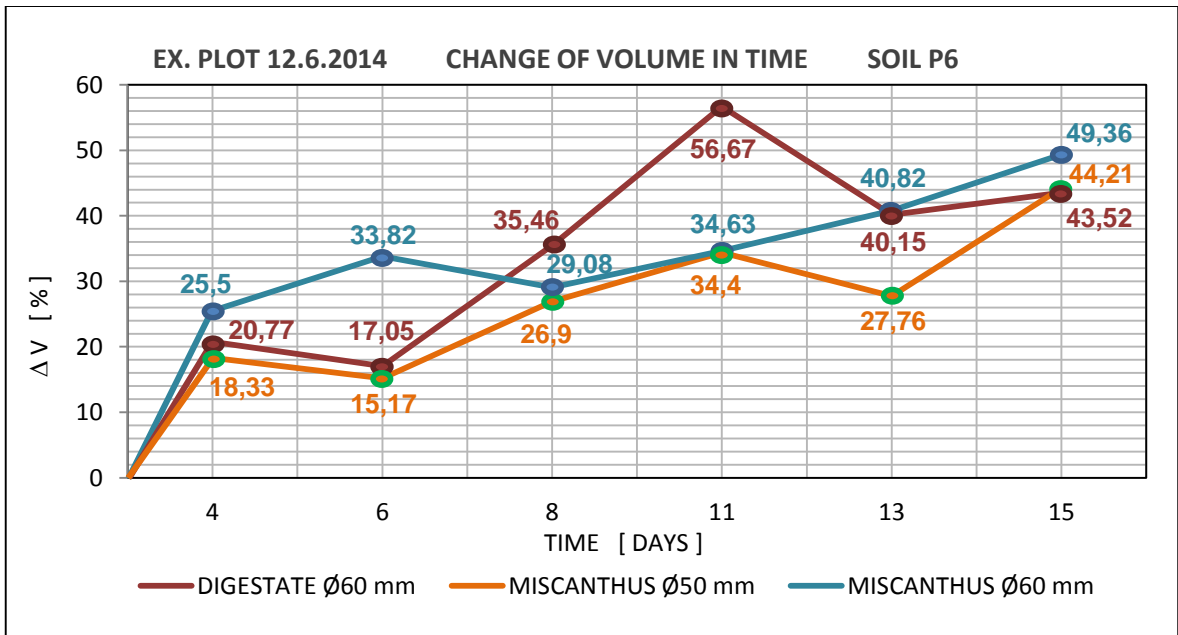


**Annex 15: Line chart - Increase in digestate and miscanthus briquettes moisture and decrease in soil moisture in time, S6 sample in experimental plot, June**



Source: Author

**Annex 16: Line chart – Change of Volume of digestate and miscanthus briquettes in time, S6 sample in experimental plot, June (Author)**



Source: Author

**Annex 17: Tabulated data – Laboratory experiment 4.6.2014 soil S6**

BOX 1						SOIL					S6	Established: 4.6.2014					June 2014				
DATE	Soil sample					Digestate 60 mm					Miscanthus sinensis 50 mm					Miscanthus sinensis 60 mm					
	ozn	w1	w2	w3	w	oz	m	V	density	w	oz	m	V	density	w	oz	m	V	density	w	
	l	g	g	g	%	l	g	cm3	g/cm3	%	l	g	cm3	g/cm3	%	l	g	cm3	g/cm3	%	
4.6.	S6	x	x	x	20.56	1	151.76	169.59	0.895	8.27	1	88.33	99.50	0.888	11.45	1	99.01	133.86	0.740	6.66	
						2	147.37	161.00	0.915	8.27	2	112.83	124.42	0.907	11.45	2	157.06	204.60	0.768	6.66	
						3	169.13	195.69	0.864	8.27	3	100.78	116.76	0.863	11.45	3	84.09	115.60	0.727	6.66	
						4	135.82	149.54	0.908	8.27	4	89.13	104.03	0.857	11.45	4	118.46	162.98	0.727	6.66	
						5	140.22	155.53	0.902	8.27	5	111.84	129.46	0.864	11.45	5	113.21	151.78	0.746	6.66	
						6	127.40	155.53	0.819	8.27	6	108.40	128.88	0.841	11.45	6	155.62	209.82	0.742	6.66	
						7	156.41	182.10	0.859	8.27	7	108.54	116.06	0.935	11.45	7	147.29	197.38	0.746	6.66	
						8	162.89	161.14	1.011	8.27	8	106.26	122.83	0.865	11.45	8	137.01	182.77	0.750	6.66	
						9	129.76	147.65	0.879	8.27	9	97.53	111.81	0.872	11.45	9	163.16	217.25	0.751	6.66	
						10	96.53	110.75	0.872	8.27	10	106.28	127.94	0.831	11.45	10	136.59	181.87	0.751	6.66	
6.6.	S6	x	x	x	18.89	1	161.14	218.89	0.736	13.17	1	92.62	120.96	0.766	14.22	1	107.73	169.20	0.637	13.78	
						2	156.37	207.42	0.754	13.43	2	117.74	150.69	0.781	15.75	2	169.32	257.81	0.657	12.79	
11.6.	S6	x	x	x	18.64	3	200.78	317.25	0.633	18.63	3	113.39	159.67	0.710	17.41	3	100.52	193.65	0.519	17.61	
						4	153.82	243.89	0.631	15.03	4	98.41	139.39	0.706	15.61	4	138.30	258.12	0.536	15.39	
13.6.	S6	x	x	x	19.28	5	164.61	280.41	0.587	20.22	5	123.72	205.16	0.603	19.71	5	134.81	262.50	0.514	19.68	
						6	151.15	264.26	0.572	19.88	6	120.25	198.33	0.606	19.61	6	177.38	301.67	0.588	17.11	
16.6.	S6	x	x	x	18.58	7	187.06	340.88	0.549	24.23	7	121.20	217.54	0.557	22.32	7	167.76	377.16	0.445	20.57	
						8	197.96	283.05	0.699	24.63	8	120.78	189.96	0.636	24.40	8	158.92	308.09	0.516	19.65	
18.6.	S6	x	x	x	18.31	9	159.47	288.98	0.552	26.37	9	112.82	199.85	0.565	23.69	9	194.63	391.44	0.497	21.77	
						10	119.36	219.86	0.543	27.06	10	120.85	196.34	0.616	23.15	10	162.76	311.51	0.522	21.59	

Source: Author

**Annex 18: Tabulated data – Laboratory experiment 4.6.2014 soil S1**

BOX		SOIL				S1	Established: 4.6.2014					June 2014								
DATE	Soil sample					Digestate 60 mm					Miscanthus sinensis 50 mm					Miscanthus sinensis 60 mm				
	oz	w1	w2	w3	w	oz	m	V	density	w	oz	m	V	density	w	oz	m	V	density	w
	l	g	g	g	%	l	g	cm3	g/cm <sup>3</sup>	%	l	g	cm3	g/cm <sup>3</sup>	%	l	g	cm3	g/cm <sup>3</sup>	%
4.6.	S1	x	x	x	9.51	1	150.26	183.53	0.819	8.27	1	104.89	121.56	0.863	11.45	1	138.21	209.23	0.661	6.66
						2	140.74	167.28	0.841	8.27	2	98.93	114.14	0.867	11.45	2	126.03	192.75	0.654	6.66
						3	147.16	170.03	0.865	8.27	3	107.15	128.77	0.832	11.45	3	151.91	228.34	0.665	6.66
						4	157.91	127.13	1.242	8.27	4	97.29	108.95	0.893	11.45	4	153.14	229.19	0.668	6.66
						5	141.18	158.74	0.889	8.27	5	112.78	130.61	0.863	11.45	5	167.77	258.94	0.648	6.66
						6	142.22	170.89	0.832	8.27	6	118.58	113.42	1.045	11.45	6	123.83	190.08	0.651	6.66
						7	130.36	158.53	0.822	8.27	7	121.85	141.97	0.858	11.45	7	108.26	168.61	0.642	6.66
						8	144.45	167.72	0.861	8.27	8	106.06	116.26	0.912	11.45	8	96.54	126.80	0.761	6.66
						9	154.97	178.33	0.869	8.27	9	98.60	119.14	0.828	11.45	9	141.57	216.83	0.653	6.66
						10	160.96	198.12	0.812	8.27	10	106.54	118.75	0.897	11.45	10	82.74	143.96	0.575	6.66
6.6.	S1	x	x	x	10.72	1	161.90	232.26	0.697	13.01	1	111.15	137.36	0.809	15.06	1	148.51	260.72	0.570	12.48
						2	149.25	205.51	0.726	13.95	2	104.81	129.36	0.810	14.99	2	134.71	242.36	0.556	12.41
11.6.	S1	x	x	x	9.61	3	166.72	257.60	0.647	15.07	3	117.07	142.47	0.822	14.55	3	183.01	353.74	0.517	14.75
						4	176.88	240.20	0.736	14.42	4	107.50	150.17	0.716	14.35	4	177.22	340.31	0.521	13.81
13.6.	S1	x	x	x	12.01	5	166.73	284.27	0.587	20.67	5	126.03	211.74	0.595	21.28	5	197.57	418.78	0.472	18.21
						6	164.57	270.90	0.607	18.69	6	130.04	146.28	0.889	17.63	6	142.99	276.59	0.517	17.43
16.6.	S1	x	x	x	8.90	7	153.12	255.77	0.599	21.75	7	138.02	215.76	0.640	22.14	7	129.99	261.63	0.497	19.41
						8	168.93	279.22	0.605	20.28	8	118.30	178.78	0.662	19.38	8	109.69	185.53	0.591	17.73
18.6.	S1	x	x	x	7.73	9	180.19	299.50	0.602	20.30	9	110.02	170.61	0.645	19.83	9	164.96	333.79	0.494	19.00
						10	186.74	317.20	0.589	20.61	10	116.80	162.69	0.718	18.51	10	92.29	182.39	0.506	18.01

Source: Author

**Annex 19: Tabulated data – Laboratory experiment 18.6.2014 soil S6**

BOX		SOIL					S6	Established: 18.6.2014					June 2014							
DATE	Soil sample					Digestate 60 mm					Miscanthus sinensis 50 mm					Miscanthus sinensis 60 mm				
	ozn	w1	w2	w3	w	oz	m	V	density	w	oz	m	V	density	w	oz	m	V	density	w
	l	g	g	g	%	l	g	cm3	g/cm3	%	l	g	cm3	g/cm3	%	l	g	cm3	g/cm3	%
18.6.	S6	x	x	x	18.63	1	83.93	104.48	0.803	8.50	1	119.31	138.16	0.864	11.52	1	136.03	215.64	0.631	7.10
						2	85.08	114.61	0.742	8.50	2	103.80	122.02	0.851	11.52	2	147.42	198.93	0.741	7.10
						3	104.12	127.98	0.814	8.50	3	94.88	120.90	0.785	11.52	3	140.30	223.34	0.628	7.10
						4	130.71	158.65	0.824	8.50	4	110.31	117.25	0.941	11.52	4	146.29	198.87	0.736	7.10
						5	146.52	174.35	0.840	8.50	5	101.48	119.11	0.852	11.52	5	145.65	242.86	0.600	7.10
						6	162.98	236.95	0.688	8.50	6	103.31	122.58	0.843	11.52	6	142.96	197.96	0.722	7.10
						7	128.43	223.08	0.576	8.50	7	113.87	127.77	0.891	11.52	7	128.82	205.54	0.627	7.10
						8	155.84	227.82	0.684	8.50	8	106.52	125.71	0.847	11.52	8	152.32	214.80	0.709	7.10
						9	145.20	207.16	0.701	8.50	9	100.59	113.43	0.887	11.52	9	137.93	181.24	0.761	7.10
						10	153.61	215.09	0.714	8.50	10	83.32	101.04	0.825	11.52	10	130.71	180.89	0.723	7.10
20.6.	S6	x	x	x	18.53	1	93.67	163.39	0.573	12.96	1	126.63	169.94	0.745	9.78	1	150.95	285.46	0.529	12.00
						2	92.30	153.71	0.600	11.48	2	110.14	136.20	0.809	10.67	2	158.26	232.88	0.680	8.17
23.6	S6	x	x	x	15.60	3	122.38	204.33	0.599	21.61	3	105.48	151.57	0.696	20.90	3	162.33	271.78	0.597	18.78
						4	149.24	242.25	0.616	20.65	4	120.25	154.55	0.778	19.09	4	162.05	263.21	0.616	16.85
25.6.	S6	x	x	x	17.93	5	174.03	280.04	0.621	21.18	5	114.31	175.54	0.651	23.16	5	170.24	357.92	0.476	19.56
						6	190.48	312.70	0.609	25.01	6	116.96	168.29	0.695	21.77	6	159.55	256.28	0.623	16.90
27.6	S6	x	x	x	16.56	7	156.22	292.50	0.534	21.00	7	128.18	211.56	0.606	20.76	7	152.26	314.40	0.484	17.94
						8	184.52	323.45	0.570	18.45	8	118.10	166.77	0.708	22.29	8	176.60	313.51	0.563	16.92
30.6.	S6	x	x	x	17.02	9	177.07	318.20	0.556	25.46	9	112.71	178.46	0.632	23.70	9	159.21	268.83	0.592	22.19
						10	189.30	340.25	0.556	25.37	10	95.32	138.12	0.690	22.74	10	148.80	274.09	0.543	22.74

Source: Author

**Annex 20: Tabulated data – Laboratory experiment 18.6.2014 soil S1**

BOX		SOIL				S1	Established: 18.6.2014					June 2014								
DATE	Soil sample					Digestate 60 mm					Miscanthus sinensis 50 mm					Miscanthus sinensis 60 mm				
	ozn	w1	w2	w3	w	oz	m	V	density	w	oz	m	V	density	w	oz	m	V	density	w
	l	g	g	g	%	l	g	cm3	g/cm3	%	l	g	cm3	g/cm3	%	l	g	cm3	g/cm3	%
18.6.	P1	x	x	x	8.47	1	150.18	183.33	0.819	8.50	1	116.67	135.17	0.863	11.52	1	125.91	224.88	0.560	7.10
						2	152.21	191.38	0.795	8.50	2	126.84	140.53	0.903	11.52	2	146.85	202.61	0.725	7.10
						3	166.21	218.91	0.759	8.50	3	101.99	118.59	0.860	11.52	3	153.06	194.74	0.786	7.10
						4	131.23	174.47	0.752	8.50	4	101.62	111.28	0.913	11.52	4	157.25	215.25	0.731	7.10
						5	151.66	182.30	0.832	8.50	5	113.91	141.26	0.806	11.52	5	144.02	239.46	0.601	7.10
						6	144.03	173.27	0.831	8.50	6	100.15	110.22	0.909	11.52	6	107.65	173.71	0.620	7.10
						7	153.14	190.63	0.803	8.50	7	102.52	121.34	0.845	11.52	7	137.52	180.88	0.760	7.10
						8	146.53	183.72	0.798	8.50	8	104.39	121.13	0.862	11.52	8	133.47	190.68	0.700	7.10
						9	146.24	180.71	0.809	8.50	9	101.69	121.20	0.839	11.52	9	146.02	228.05	0.640	7.10
						10	156.64	189.10	0.828	8.50	10	96.20	115.61	0.832	11.52	10	180.88	239.67	0.755	7.10
20.6.	S1	x	x	x	9.11	1	166.49	233.05	0.714	9.65	1	122.60	147.90	0.829	8.43	1	139.12	263.86	0.527	10.18
						2	163.38	218.77	0.747	7.84	2	130.48	149.72	0.871	7.95	2	163.37	236.82	0.690	8.04
23.6.	S1	x	x	x	10.02	3	184.41	306.30	0.602	18.03	3	109.50	147.29	0.743	18.46	3	174.63	313.49	0.557	18.16
						4	145.76	217.36	0.671	18.31	4	108.40	129.63	0.836	16.32	4	173.61	279.33	0.622	16.36
25.6.	S1	x	x	x	8.16	5	171.84	276.57	0.621	13.05	5	121.62	161.49	0.753	17.83	5	164.63	329.14	0.500	17.28
						6	160.99	243.16	0.662	16.54	6	108.18	144.77	0.747	16.97	6	121.43	231.13	0.525	16.20
27.6.	S1	x	x	x	8.25	7	175.02	285.67	0.613	14.21	7	111.18	154.43	0.720	15.81	7	156.76	271.15	0.578	13.95
						8	166.78	254.74	0.655	13.73	8	114.23	149.39	0.765	14.54	8	146.53	227.39	0.644	11.70
30.6.	S1	x	x	x	9.07	9	168.95	274.38	0.616	21.85	9	111.39	149.88	0.743	19.48	9	169.50	301.57	0.562	18.88
						10	180.88	270.57	0.669	20.70	10	102.00	134.01	0.761	18.53	10	203.86	328.35	0.621	17.31

Source: Author

**Annex 21: Tabulated data – Experimental plot June soil S6**

EX.PLOT		SOIL				S6	Established: 12.6.2014					June 2014								
DATE	Soil sample					Digestate 60 mm					Miscanthus sinensis 50 mm					Miscanthus sinensis 60 mm				
	ozn	w1	w2	w3	w	oz	m	V	density	w	oz	m	V	density	w	oz	m	V	density	w
	l	g	g	g	%	l	g	cm3	g/cm3	%	l	g	cm3	g/cm3	%	l	g	cm3	g/cm3	%
12.6.	S6	x	x	x	9,10	1	157.92	196.10	0.805	8.20	1	95.69	113.22	0.845	11.50	1	142.98	198.21	0.721	6.60
						2	141.65	176.63	0.802	8.20	2	115.85	125.66	0.922	11.50	2	142.09	194.87	0.729	6.60
						3	145.97	187.50	0.779	8.20	3	99.49	107.69	0.924	11.50	3	133.20	179.24	0.743	6.60
						4	155.47	192.63	0.807	8.20	4	103.97	121.16	0.858	11.50	4	138.45	184.65	0.750	6.60
						5	143.11	176.97	0.809	8.20	5	89.15	103.22	0.864	11.50	5	124.40	195.82	0.635	6.60
						6	156.31	179.76	0.870	8.20	6	96.38	116.42	0.828	11.50	6	142.16	187.77	0.757	6.60
						7	159.98	187.81	0.852	8.20	7	105.95	131.32	0.807	11.50	7	117.74	164.21	0.717	6.60
						8	161.25	197.30	0.817	8.20	8	105.09	125.05	0.840	11.50	8	149.77	208.96	0.717	6.60
						9	146.96	187.11	0.785	8.20	9	99.72	120.86	0.825	11.50	9	159.04	213.60	0.745	6.60
						10	159.42	178.14	0.895	8.20	10	118.92	133.54	0.891	11.50	10	144.41	186.20	0.776	6.60
						11	159.43	186.03	0.857	8.20	11	87.76	100.18	0.876	11.50	11	124.51	168.96	0.737	6.60
						12	149.61	187.06	0.800	8.20	12	98.34	101.75	0.966	11.50	12	157.02	224.26	0.700	6.60
						13	157.08	191.86	0.819	8.20	13	110.83	147.71	0.750	11.50	13	137.52	187.40	0.734	6.60
						14	147.70	186.76	0.791	8.20	14	102.00	121.28	0.841	11.50	14	143.72	197.16	0.729	6.60
						15	153.40	185.04	0.829	8.20	15	90.27	111.83	0.807	11.50	15	144.37	193.95	0.744	6.60
						16	172.78	208.01	0.831	8.20	16	117.66	129.33	0.910	11.50	16	132.34	167.41	0.791	6.60
						17	152.24	192.22	0.792	8.20	17	91.40	93.15	0.981	11.50	17	136.79	184.10	0.743	6.60
						18	149.76	193.31	0.775	8.20	18	119.51	149.57	0.799	11.50	18	137.65	216.53	0.636	6.60
16.6.	S6	x	x	x	9.41	1	175.60	253.61	0.692	16.67	1	101.99	121.84	0.837	15.87	1	156.01	266.20	0.586	14.77
						2	153.82	206.54	0.745	14.77	2	121.96	145.64	0.837	14.07	2	154.68	235.65	0.656	14.01
						3	158.87	217.62	0.730	14.57	3	104.94	141.59	0.741	14.05	3	145.40	217.39	0.669	14.12
18.6.	S6	x	x	x	10.34	4	173.84	253.07	0.687	17.16	4	112.17	146.17	0.767	16.50	4	153.62	253.57	0.606	15.66
						5	159.34	232.78	0.685	16.62	5	95.76	125.98	0.760	16.15	5	138.00	258.34	0.534	14.85
						6	173.76	247.44	0.702	17.36	6	100.04	119.69	0.836	16.54	6	156.41	248.25	0.630	14.43
20.6.	S6	x	x	x	10.32	7	181.06	254.42	0.712	14.55	7	114.37	174.24	0.656	13.58	7	131.88	213.81	0.617	13.58

23.6.						8	186.03	258.62	0.719	15.12	8	113.84	150.05	0.759	14.13	8	167.92	280.54	0.599	12.18
						9	165.60	261.62	0.633	14.86	9	107.89	154.74	0.697	12.96	9	176.12	262.26	0.672	13.69
	S6	x	x	x	12.15	10	182.31	280.45	0.650	21.16	10	128.35	170.20	0.754	19.97	10	162.90	265.30	0.614	19.40
						11	184.38	315.34	0.585	22.00	11	95.35	139.07	0.686	20.00	11	140.16	221.10	0.634	19.05
25.6.						12	172.75	267.60	0.646	21.82	12	106.39	139.31	0.764	19.72	12	176.89	292.76	0.604	18.86
	S6	x	x	x	12.58	13	182.60	255.30	0.715	20.08	13	122.06	176.74	0.691	17.10	13	155.45	256.91	0.605	17.86
						14	174.23	271.44	0.642	19.82	14	112.50	161.46	0.697	22.38	14	163.23	273.62	0.597	16.90
27.6.						15	180.96	262.84	0.688	20.39	15	100.07	145.93	0.686	23.78	15	166.04	284.32	0.584	19.46
	S6	x	x	x	11.48	16	202.98	304.07	0.668	20.72	16	131.20	202.12	0.649	21.00	16	146.57	248.50	0.590	11.78
						17	177.79	270.67	0.657	16.51	17	97.66	130.75	0.747	14.22	17	153.99	247.75	0.622	14.79
						18	177.53	277.55	0.640	13.63	18	130.11	203.41	0.640	15.82	18	159.96	357.44	0.448	20.83

Source: Author

#### Annex 22: Tabulated data – Experimental plot October soil S6

EX. PLOT						SOIL					S6	Established: 7.10.2014					October 2014				
DATE	Soil sample					Digestate 60 mm					Miscanthus sinensis 50 mm					Miscanthus sinensis 60 mm					
	ozn	w1	w2	w3	w	oz	m	V	density	w	oz	m	V	density	w	oz	m	V	density	w	
	l	g	g	g	%	l	g	cm3	g/cm3	%	l	g	cm3	g/cm3	%	l	g	cm3	g/cm3	%	
7.10.	S1	x	x	x	17.18	1	162.53	174.48	0.932	5.78	1	101.16	122.57	0.825	7.10	1	122.75	190.39	0.645	7.50	
						2	149.53	162.60	0.920	5.78	2	100.44	118.48	0.848	7.10	2	113.51	185.22	0.613	7.50	
						3	199.40	232.69	0.857	5.78	3	91.09	114.68	0.794	7.10	3	125.50	208.01	0.603	7.50	
						4	189.70	201.85	0.940	5.78	4	107.11	124.61	0.860	7.10	4	147.01	228.80	0.643	7.50	
						5	172.60	222.38	0.776	5.78	5	102.60	129.55	0.792	7.10	5	130.21	200.96	0.648	7.50	
						6	176.95	195.01	0.907	5.78	6	100.62	121.74	0.827	7.10	6	148.44	243.05	0.611	7.50	
						7	150.20	160.80	0.934	5.78	7	93.61	110.31	0.849	7.10	7	118.15	199.74	0.592	7.50	
						8	145.05	152.64	0.950	5.78	8	91.35	116.80	0.782	7.10	8	140.40	221.53	0.634	7.50	
						9	166.56	181.32	0.919	5.78	9	98.81	118.48	0.834	7.10	9	114.62	198.18	0.578	7.50	

						<b>10</b>	163.09	177.90	0.917	5.78	<b>10</b>	95.88	116.44	0.823	7.10	<b>10</b>	109.08	155.13	0.703	7.50
						<b>11</b>	178.38	195.01	0.915	5.78	<b>11</b>	100.82	118.48	0.851	7.10	<b>11</b>	137.34	210.64	0.652	7.10
						<b>12</b>	170.74	182.51	0.936	5.78	<b>12</b>	103.10	127.42	0.809	7.10	<b>12</b>	135.68	197.44	0.687	7.10
						<b>13</b>	151.33	159.28	0.950	5.78	<b>13</b>	92.08	106.03	0.868	7.10	<b>13</b>	100.28	151.60	0.661	7.10
						<b>14</b>	161.50	169.23	0.954	5.78	<b>14</b>	103.45	131.67	0.786	7.10	<b>14</b>	136.31	205.27	0.664	7.10
						<b>15</b>	201.80	243.27	0.830	5.78	<b>15</b>	97.75	127.42	0.767	7.10	<b>15</b>	138.33	250.32	0.553	7.10
						<b>16</b>	161.64	177.90	0.909	5.78	<b>16</b>	97.80	121.05	0.808	7.10	<b>16</b>	124.23	224.36	0.554	7.10
						<b>17</b>	176.66	191.57	0.922	5.78	<b>17</b>	97.26	130.16	0.747	7.10	<b>17</b>	115.54	193.91	0.596	7.10
						<b>18</b>	157.91	173.72	0.909	5.78	<b>18</b>	99.85	133.79	0.746	7.10	<b>18</b>	105.96	177.95	0.595	7.10
<b>10.10.</b>	<b>S1</b>	<b>x</b>	<b>x</b>	<b>x</b>	<b>15.89</b>	<b>1</b>	172.92	199.74	0.866	15.60	<b>1</b>	108.82	168.68	0.645	10.49	<b>1</b>	133.45	241.51	0.553	14.79
						<b>2</b>	156.36	186.86	0.837	13.72	<b>2</b>	106.65	152.05	0.701	13.75	<b>2</b>	120.81	215.51	0.561	13.94
						<b>3</b>	208.64	246.80	0.845	13.69	<b>3</b>	96.30	145.32	0.663	13.42	<b>3</b>	134.73	242.45	0.556	14.59
<b>14.10.</b>	<b>S1</b>	<b>x</b>	<b>x</b>	<b>x</b>	<b>14.23</b>	<b>4</b>	254.68	342.01	0.745	53.32	<b>4</b>	140.13	251.64	0.557	69.34	<b>4</b>	204.35	344.07	0.594	55.65
						<b>5</b>	195.36	289.02	0.676	44.56	<b>5</b>	116.82	189.65	0.616	70.27	<b>5</b>	159.83	272.05	0.588	56.12
						<b>6</b>	202.12	250.59	0.807	45.00	<b>6</b>	114.70	193.93	0.591	65.49	<b>6</b>	196.61	339.77	0.579	50.99
<b>20.10.</b>	<b>S1</b>	<b>x</b>	<b>x</b>	<b>x</b>	<b>21.06</b>	<b>7</b>	285.91	361.27	0.791	73.07	<b>7</b>	221.91	308.83	0.719	83.33	<b>7</b>	206.72	301.29	0.686	74.77
						<b>8</b>	244.58	297.22	0.823	65.90	<b>8</b>	231.22	299.68	0.772	82.52	<b>8</b>	293.41	407.15	0.721	73.20
						<b>9</b>	303.41	382.33	0.794	70.73	<b>9</b>	233.80	321.88	0.726	80.08	<b>9</b>	225.05	311.99	0.721	72.49
<b>23.10.</b>	<b>S1</b>	<b>x</b>	<b>x</b>	<b>x</b>	<b>21.93</b>	<b>10</b>	379.81	385.60	0.985	64.23	<b>10</b>	287.00	299.26	0.959	65.72	<b>10</b>	281.76	262.35	1.074	63.28
						<b>11</b>	421.26	453.46	0.929	61.83	<b>11</b>	317.08	331.86	0.955	63.83	<b>11</b>	334.09	366.94	0.910	61.70
						<b>12</b>	432.93	455.86	0.950	62.10	<b>12</b>	303.29	339.78	0.893	68.55	<b>12</b>	342.25	362.72	0.944	58.56
<b>27.10.</b>	<b>S1</b>	<b>x</b>	<b>x</b>	<b>x</b>	<b>21.23</b>	<b>13</b>	376.87	653.22	0.577	65.77	<b>13</b>	248.82	467.18	0.533	66.68	<b>13</b>	252.73	359.11	0.704	61.93
						<b>14</b>	452.36	476.35	0.950	64.17	<b>14</b>	272.38	489.69	0.556	66.24	<b>14</b>	344.10	421.54	0.816	61.23
						<b>15</b>	515.82	904.04	0.571	66.00	<b>15</b>	292.72	559.07	0.524	66.40	<b>15</b>	377.45	578.85	0.652	65.95
<b>31.10.</b>	<b>S1</b>	<b>x</b>	<b>x</b>	<b>x</b>	<b>20.26</b>	<b>16</b>	303.54	386.22	0.786	65.99	<b>16</b>	255.53	260.10	0.982	64.03	<b>16</b>	302.79	313.96	0.964	62.57
						<b>17</b>	404.04	414.44	0.975	60.48	<b>17</b>	240.74	261.84	0.919	63.60	<b>17</b>	279.22	318.09	0.878	62.73
						<b>18</b>	395.93	416.64	0.950	62.28	<b>18</b>	244.33	259.64	0.941	62.20	<b>18</b>	253.60	293.15	0.865	63.31

Source: Author