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METHANE POTENTIAL OF MUNICIPAL SLUDGE IN ANAEROBIC CO-DIGESTION PROCESS BOOSTED WITH GLYCEROL

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At	bstract			
As	s a renewable resource, the production o	f biogas is increasingly value	d by various countries in the	
world. The goal of this thesis was to study the methane potential of municipal sludge in anaerobic				
co-digestion process boosted with waste glycerol. The municipal sludge was obtained from the				
	wastewater treatment plant Lehtoniemi (Kuopio Vesi) in Kuopio, Finland. The study compares the			
m	methane potential of municipal sludge and same feeding material boosted with glycerol as raw ma-			
te	terials in the anaerobic co-digestion process to produce biogas, and compare the performance of bi-			
og	ogas produced from different sources.			
Two materials were used for the experiment, which were municipal sludge and waste glycerol from				
biodiesel production. Municipal sludge is an experimental sample, and it requires determining me-				
thane potential. The inoculum was mixed with municipal sludge in a reactor to get the anaerobic di-				
gestion process started properly.				
Ad	According to the results of the experiment, for municipal sludge without glycerol 11 Nm ³ methane			
са	can be produced (per 1000 kg fresh mass). For municipal sludge boosted with glycerol 22 Nm ³ me-			
	ane can be produced.		5,	
	In general, the municipal sludge added with 1% glycerol produces almost 100% more methane			

In general, the municipal sludge added with 1% glycerol produces almost 100% more methane compared to sludge without glycerol. This study shows that boosting municipal sludge with glycerol in the anaerobic co-digestion process has a good potential for increased methane yield.

Keywords

Biogas production, circular economy, organic matter, waste.

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1 INTRODUCTION

One of the major environmental issues in today's society is the increasing production of municipal sludge, traditional methods of treating municipal sludge always cause environmental pollution. Uncontrolled waste dumping isn't acceptable anymore. Even controlled landfill disposal and incineration of municipal sludge are not optimal methods anymore. With environmental standards are becoming increasingly strict, recovering energy from municipal sludge is aimed.

Biogas production can be achieved through the anaerobic digestion (AD) of municipal sludge and various digestible organic wastes. Recent research shows that adding glycerol to municipal sludge can help it produce more biogas. Glycerol is a simple polyol compound and a by- product from the industry, it is a non-toxic viscous liquid that is sweet-tasting colorless and odourless.

Anaerobic digestion is a process of microbial decomposition of organic matter. It is common in many natural environments in the absence of oxygen. It is currently used mainly in the production of biogas in sealed reactors, commonly referred to as biogas digesters. The anaerobic process involves a wide range of microorganisms, there are two main end products in anaerobic process: biogas and digestate. Biogas is mainly composed of carbon dioxide and methane, and it also contains a small amount of other gases.

Using municipal sludge to produce biogas in anaerobic digestion not only solves the problem of municipal sludge treatment, but also minimises cause environmental pollution. Faced with the global energy shortage, great progress in exploring effective and sustainable energy sources in recent years was done. Biogas is considered to be an important energy source among different types of energy available. It is a sustainable source of fuel for heat, the production of electricity as well as, energy used fr vehides. Therefore, study the methane potential of municipal sludge has a huge significance.

The main objective of this study was to determine the efficiency of biogas produced from municipal sludge after the addition of glycerol, compare the methane potential of municipal sludge and the same feeding material boosted with glycerol as raw materials in anaerobic co-digestion process to produce biogas.

2 LITERATURE REVIEW

2.1 Biogas production around the world

2.1.1 Current situation of biogas and biomethane production in the European Union (EU)

According to the European Biogas Association (EBA), there were 17,240 biogas plants in the EU in 2014. This corresponds to a total installed capacity of 8,293 MWe. FIGURE 1 shows these data per country. The greatest producers are Germany (10,786 plants with an average installed capacity of 400 kWel), Italy (1,491 plants) and the United Kingdom (813 plants). Spain has currently 39 biogas plants. Biogas combustion produced 63.3 TWh in the EU in 2014, and this is equivalent to spending 14.9 million European households annually (Hernandez and Chamorro 2016).

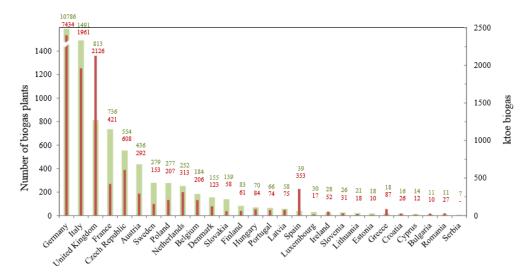


FIGURE 1 Biogas plants in the EU (green bars) and annual production of primary energy from biogas in 2014 expressed in ktoe (tonne of oil equivalent, red bars, Hernandez and Chamorro 2016)

2.1.2 The United States

There are more than 2,000 sites producing biogas in the United States: 239 anaerobic digesters in the farm, 1,241 sewage treatment plants using anaerobic digesters (currently about 860 use their biogas) and 636 landfill gas projects. There are 8,002 dairy farms and swine farms, and 2,440 wastewater treatment plants support biogas digesters and 450 untapped landfill gas projects, nearly 11,000 sites available for development. (Biogas opportunities roadmap 2014).

2.1.3 China

China's domestic biogas had some ups and downs in the 80 years. Efforts to create biogas in China can be traced back to the 1930s. Some companies tried to use biogas as imported kerosene alternative lighting. Thousands of low-cost biogas digesters were built in 1958. Although the initial results were satisfactory, most of these devices were discarded after a few years, mainly due to problems caused by low-grade building materials. At the time, the supply of cement was insufficient. On the

contrary, the so-called "trinity mixture fill" was widely used, including lime, clay and sand. (Xia 2013).

Until around the year 1970, biogas was widely disseminated and promoted. About seven million biogas plants were built after a large-scale massive campaigns. However, due to technical deficiencies, these only play a minor role. In 1975, people started to use biogas technology on a large scale and incorporated biogas into the national economy plan. Part of this promotion is the training of technicians, the manufacture of appliances and equipment, and the experiment of different digester designs (Biogas technology in China 2014).

Since 1982, standards must be developed and applied to the construction of biogas projects. Although there have been some improvements in the diffusion strategy, many problems and high failure rates still exist in the 1980s. About 5 million domestic household biogas digesters operate well, about 1.7 million biogas plants were put into operation in Sichuan until 1992. In the 1990s, investment increased dramatically, during the Ninth Five-Year Plan period, the Chinese government invested 6 billion yuan in biogas development. By the end of the 1990s, a total of 9.8 million household digesters were operating in China (Biogas technology in China 2014).

The annual out of biogas was approximately 1.55×10^{10} m³ in 2010, and it was calculated to be equal to approximately 5.55×10^{11} MJ of heat (the heat of methane combustion is 35.822 MJ/m³), as shown in TABLE 1 (Feng et al. 2012).

TABLE 1. Number of household biogas digesters built and annual output of biogas in China (Feng et al. 2012)

Year	The number of biogas digesters (10 ⁴)	Annual output of biogas (10 ⁸ m ³)
1996	602.1	16.3
1997	638.2	17.7
1998	688.8	19.8
1999	763.5	22.5
2000	848.1	25.9
2001	956.8	29.8
2002	1109.9	37.0
2003	1288.9	45.8
2004	1541.0	55.7
2005	1800.0	69.0
2006	2200.0	85.0
2007	2650.0	102.0
2008	3050.0	122.0
2009	3507.0	124.0
2010	4118.0	155.0

As shown in FIGURE 2, In general, biogas digesters in rural biogas digesters account for a large proportion of total biogas production and show long-term stable growth. However, according to the data by the Ministry of Agriculture of China. The generation of household biogas seems to have slowed to pre-2011 levels in recent years (calculated from MOA 1989–2014). In contrast, agricultur- al biogas projects show a clear growth trend and play an increasingly important role in China's bio-

gas production. In addition, biogas from biogas projects of industrial biogas projects fluctuate slightly, and after 2004 the agricultural sector surpassed biogas projects (calculated from MOA 1989– 2014).

In the past few decades, China has accelerated the pace of urbanization, industrialization, and even the transformation of energy and agriculture. Therefore, biogas production in China is undergoing a dynamic and complex transition that is quite different from that of developed countries and is different from those countries that rely on traditional farming and animal husbandry. (Gu et al. 2016).

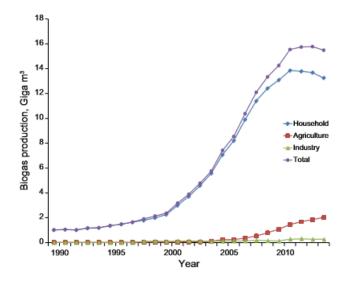


FIGURE 2. Biogas production for different sectors in China from 1989 to 2014 (Gu et al. 2016)

2.2 The benefits of biogas technologies

Building a 10 m³ anaerobic digestion reactor can solve the annual energy consumption of 5 - 8 homes in the countryside, reduce the burning cost of more than 5 tons of firewood or straw, and protect 4 acres of forest or 5.4 acres of young trees, protect forest vegetation, biogas technologies play a good role in protecting natural forest land (Significance of developing biogas 2013).

Anaerobic digestion (AD) requires biogas production to operate the equipment and maintenance it. Anaerobic digestion involves raw material collection and transportation, technical equipment manufacturing, and biogas plant construction. This means that the development of a national biogas sector will help create new businesses, some of which have significant economic potential, increase rural incomes and create new jobs (Al Seadi et al. 2008). Biogas project not only can develop new energy sources but also can improve soil structure and improve rural environmental sanitation (Li 2013).

The production and development of biogas provide the society with environmental and social economic benefits. It improves the standard of living and contributes to economic and social development. As a renewable resource, it has great potential for future energy use. Biogas is a biofuel that is naturally produced by the decomposition of organic waste. When organic matter (such as food residues or animal waste) decomposes in an anaerobic environment, gas mixtures are released, the gas includes mainly methane and carbon dioxide, and small amounts of other gases. Since this decomposition takes place in an oxygen-free environment, the process of producing biogas is also called anaerobic digestion. (Benzaken 2018). TABLE 2 shows the composition of biogas.

Compound	Formula	%
Oxygen	O ₂	0-0.5
Hydrogen sulfide	H ₂ S	0.1-0.5
Hydrogen	H ₂	0-1.5
Nitrogen	N2	0-9.5
Carbon dioxide	CO ₂	25-50
Methane	CH ₄	50-70

TABLE 2. The typical composition of biogas (Zhao 2017)

2.4 Anaerobic digestion

Anaerobic digestion is a series of biological processes in which microorganisms decompose biodegradable materials under anaerobic conditions. The final product is mainly biogas. Biogas can generate heat and electricity through combustion. A series of anaerobic digestion technologies convert food waste, livestock manure, high-strength industrial wastewater, municipal wastewater solids and oils (Fat, oil and grease - FOG), fats, residues, and various other organic waste streams into biogas. The separated digested solids can be composted and applied directly to the farmland or converted to other products. Nutrients in the liquid stream are used as fertilizers in agriculture. FIGURE 3 shows the anaerobic digestion process (What is anaerobic digestion 2018). Anaerobic digestion is becoming a popular recycling option, because it can be used as a method for obtaining energy from waste organics and controlling methane emissions. (Greene 2015). Anaerobic digestion requires three stages:

- Hydrolysis
- Acidogenesis
- Methanogenesis

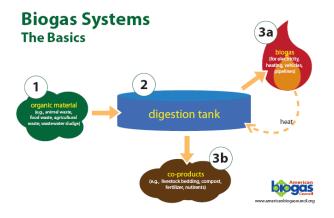


FIGURE 3. Anaerobic digestion process (What is anaerobic digestion 2018)

2.4.1 Hydrolysis

The first step of biological conversion of solid wastes is depolymerisation of the macromolecule solid substrates. This step is also known as hydrolysis. Hydrolysis is the slowest stage, so it is the rate-limiting step in the entire anaerobic digestion process (Cvetkovski and Litonjua 2012). In most cases, biomass consists of a large number of organic polymers. For bacteria in anaerobic digesters to capture the energy potential of a material, these chains must first be broken down into smaller components. Through hydrolysis, complex organic molecules are broken down into monosaccharides, fatty acids and amino acids (Gao 2016).

2.4.2 Acidogenesis

Under the effect of hydrogen-producing acetogen, the product of the previous stage is further converted into hydrogen, acetic acid, new cell material, and carbonic acid in process of acidogenesis. Some of its reaction formulas are as follows (Gao 2016):

$CH_{3}CHOHCOO^{-}+2H_{2}O \longrightarrow CH_{3}COO^{-}+HCO_{3}^{-}+H^{+}+2H_{2}$	$\Delta G'^0 = -4.2 \text{KJ/mol}$
$CH_3CH_2OH + H_2O -> CH_3COO^- + H_+ + 2H_2O$	$\Delta G'^0 = 9.6 \text{KJ/mol}$
$CH_{3}CH_{2}COO^{-}+2H_{2}O-> 2CH_{3}COO^{-}+H^{+}+2H_{2}$	ΔG' ⁰ =48.1KJ/mol
$CH_{3}CH_{2}COO^{-}+3H_{2}O^{-}>CH_{3}COO^{-}+HCO_{3}^{-}+H^{+}+3H_{2}$	$\Delta G'^0 = 76.1 \text{KJ/mol}$
$4CH_{3}OH+2CO_{2} \rightarrow 3CH_{3}COO^{-}+2H_{2}O$	ΔG' ⁰ =-2.9KJ/mol
$2HCO_3^{-}+4H_2+H^{+}->CH_3COO^{-}+4H_2O$	ΔG'0=-70.3KJ/mol

 $\Delta G^{\prime 0}$ = Gibbs free energy, the Gibbs free energy is a thermodynamic potential that can be used to calculate the maximum of reversible work that may be performed by a thermodynamic system at a constant temperature and pressure (i.e. isothermal, isobaric).

2.4.3 Methanogenesis

In the last step, methanogenic bacteria convert substrates such as formic acid, acetic acid, methylamine, and methanol into methane through different pathways, the most important one being acetic acid (Interactive Encyclopedia). The main methanogenic reaction:

$CH_3COO^-+H_2O->CH_4+HCO_3^-$	ΔG'0=-31.0KJ/mol
$HCO_{3}^{-}+H^{+}+4H_{2}->CH_{4}+3H_{2}O$	ΔG ⁷⁰ =-135.6KJ/mol
$4CH_{3}OH -> 3CH_{4} + CO_{2} + 2H_{2}O$	ΔG'0=-312KJ/mol
$4HCOO^{-}+2H^{+}->CH_{4}+CO_{2}+2HCO_{3}^{-}$	ΔG ^{′0} =-32.9KJ/mol

2.5 Parameters governing anaerobic processes

In the process of biogas fermentation, it is necessary to control parameters governing anaerobic processes, such as temperature, pH, etc. Appropriate conditions can maximize the production of biogas. If a certain condition does not meet the requirements, it may often cause the entire biogas production system process to fail.

2.5.1 Anaerobic environment

Bacteria that decompose organic substances and produce biogas are methanogens, which are all anaerobic bacteria. They are particularly sensitive to oxygen and they do not require air during their life activities such as growth, development, reproduction, and metabolism. Oxygen in the air inhibits theirs life activities and even causes death. Therefore, the construction of biogas digesters must be sealed, watertight, and gas-free. This is the key to biogas production. This is not only the need to collect biogas and storage of raw materials for biogas fermentation but also to ensure that biogas microorganisms live well under anaerobic ecological conditions so that biogas digesters can produce gas (The basic conditions for biogas fermentation 2015).

When oxygen is brought into the anaerobic digestion reactor when feeding, aerobic bacteria and facultative anaerobic bacteria will be rapidly consume dissolved oxygen, thus recreating good anaerobic conditions.

2.5.2 Temperature

The temperature of the biogas fermentation directly affects the digestion speed and the gas production rate of the raw materials. When the temperature is appropriate, the bacteria will better work and the activity will be strong. The anaerobic decomposition and the generation of methane will occur at a rapid rate. The range of temperature suitable for biogas fermentation is relatively wide. Generally, the anaerobic digestion reaction can produce biogas in range of 8-70 °C. Less than 10 °C or higher than 60°C in anaerobic digestion, it will seriously affect the survival of microbiology. The biogas fermentation is divided into three fermentation zones: 10-30 °C as psychrophilic, 33-38 °C as mesophilic, and 50-55 °C as thermophilic (TABLE 3) (The basic conditions for biogas fermentation 2015). The peak gas production temperature is about 35 °C and another is about 54 °C. TABLE 3. The three fermentation zones

fermentation principle and conditions 2011).

normal temperature	medium temperature	high temperature
10-30 °C	33-38 °C	50-55 °C

significantly. If the temperature changes suddenly, the gas production process will stop (Biogas

2.5.3 The hydraulic retention time (HRT) and the retention time of the solids (SRT)

The two parameters have important significance in the design of biological treatment process. Hydraulic retention time (HRT) is the average range of retention in the digester when the bottom layer of the anaerobic digestion process comes into contact with biomass (bacterial substances). Solids retention time (SRT) is a measure of the ability of a biological system to provide some measure of effluent and/or the ability to maintain a satisfactory biodegradation rate of a contaminant.

2.5.4 Nutrients

In the process of anaerobic fermentation, the raw material is the substrate for the production of biogas and the source of nutrients for the biogas fermentation microorganisms to survive. Various organic substances such as human and animal excreta, crop straw, leaf weeds, domestic sewage, and industrial waste containing organic substances can be used as raw materials for fermentation of biogas digesters.

Nitrogen-rich raw materials: This kind of raw material is fully digested by human and animal gastrointestinal system, generally small particles, high water content, easy anaerobic decomposition, fast gas production, short fermentation period.

Carbon-rich raw materials: These raw materials are rich in cellulose, hemicellulose, pectin and hardly degradable lignin and plant waxes.

Nitrogen is an important raw material that constitutes the cytoplasm of microbes. Carbon not only constitutes the cytoplasm of microbes but also provides energy for life activities. TABLE 4 shows some materials about nitrogen-rich raw materials and carbon-rich raw materials.

nitrogen-rich raw	Human waste, pig manure, chicken manure, cow dung, horse manure, sheep	
materials	manure, sugar cane, distiller's grains, etc.	
carbon-rich raw	Straw, straw, corn stalks, sorghum stalks, dried sweet potato vines, etc.	

TABLE 4. The example of nitrogen-rich raw materials and carbon-rich raw materials ((Zhao 2017)	
TABLE 4. THE Example of hitrogen-ficit raw materials and carbon-ficit raw materials ((ZI IAU ZUI7)	ŧ.

materials		
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2.5.5 The effect of pH value in bioreactor

The level of pH is an important factor in biogas fermentation. Normally, the suitable pH value of the methanogenic bacteria in the biogas digester is in the range of 6.5 to 7.8. Changes in the pH value will directly affect the survival and metabolism of the methanogens. Under normal circumstances, the pH of the digester should be maintained preferably around 7.2. No gas is produced when the pH is lower than 6 or higher than 9 (Zhao 2017).

2.5.6 Inoculum

The performance and stability of methanogenesis during anaerobic digestion is also affected by the quality and quantity of inoculum. Lower inoculum levels may lead to the imbalance due to the more rapid growth rate of acid-forming bacteria (compared to methanogens) and lowering of pH. Inoculum may also become imbalanced when exposed to toxic substances or environmental stress factors (e.g. abnormal temperature and heavy metals like arsenic, cadmium and chromium) for which they are not acclimated (Cvetkovski and Litonjua 2012).

2.6 Utilization of biogas

Biogas can be used for a variety of purposes. It can be used directly for lighting, cooking, It can also be used for requires that gas is used in internal combustion engines, boilers or fuel cells after the raw biogas cleanup or upgrading. The FIGURE 4 shows alternative biogas utilization and required cleanup.

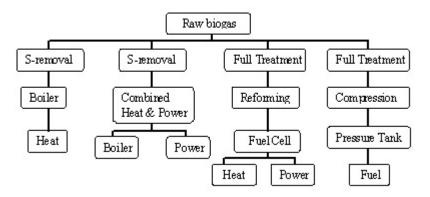


FIGURE 4. Alternative biogas utilization and required cleanup (Frazier 2015)

2.6.1 Production of heat or steam

The most direct use of biogas is heat energy. In areas where fuel is scarce, small biogas systems can provide heat for basic cooking and hot water heating. Gas lighting systems can also use biogas for lighting (Frazier 2015).

Biogas combustion power generation is a biogas utilization technology that has emerged along with the continuous development of large-scale biogas tank construction and comprehensive utilization of biogas. It uses biogas from anaerobic fermentation processes on engines and is equipped with an integrated power plant to generate electricity and heat.

The most common use is for biogas to burn internal gas engines in a combined heat and power (CHP) plant to generate electricity and heat. Compressed gas is used as automotive fuel and there are many biogas refueling stations for cars and buses in Sweden. Natural gas can also be upgraded and used for gas supply networks. The application of biogas in solid oxide fuel cells is also under study (Zafar 2015).

2.6.3 Biogas fuel cell technology

Biogas fuel cells (FIGURE 5) are the latest generation of clean, efficient, low-noise electrical devices. Compared with biogas generators, it not only has high power efficiency and energy efficiency, but also has low vibration and noise, and has low concentrations of nitrogen oxides and sulfides. Therefore, it is a promising biogas utilization process. Using biogas for fuel cell power generation is an important way to effectively use biogas resources.

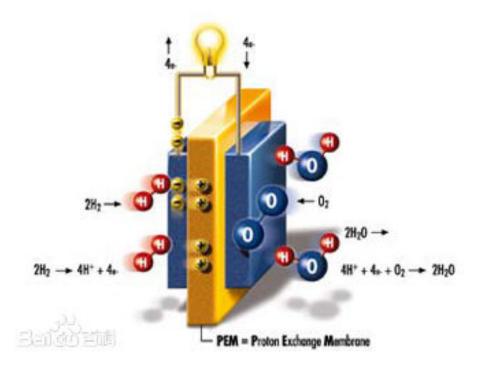


FIGURE 5. Biogas fuel cells (Baidu Wikipedia)

2.6.4 Vehicle fuel

Gasoline vehicles can use biogas as fuel, as long as the biogas is upgraded to the natural gas quality of vehicles that use natural gas for adjustment. In addition to ordinary gasoline fuel systems, most vehicles in this type of vehicle have already installed gas tanks and gas supply systems. However, special vehicles (using only biogas) are more efficient than these modifications (Frazier 2015).

2.7 Sludge characteristics

Sludge is a semi-solid slurry that can be produced as a sewage sludge in a sewage treatment process or as a suspended sediment obtained from conventional drinking water treatment and many other industrial processes. (Gao 2016). There are several classifications of sludge, such as according to the characteristics of sludge composition, according to different processes for separating sludge from sewage, or depending on source of sewage (TABLE 5, Cui et al. 2016).

Classification	Examples
According to the characteristics of sludge com- position	Organic sludge and inorganic sludge
According to different processes for separating sludge from sewage	Sludge from primary sedimentation tank, activat- ed sludge, humic sludge, chemical sludge and primary settling sludge
According to the source of sewage	Municipal sludge and industrial sludge

TABLE 5. Sludge classification and examples (Cui et al. 2016)

The main characteristic of sludge is high water content (up to 99%), high organic content and the smaller particles, colloidal liquid. Sludge contains a number of organic substances, such as benzene, chlorophenols, polychlorinated biphenyls, polychlorinated dibenzofurans and polychlorinated dibenzodioxins. Toxic metals such as cadmium, chromium, copper, zinc, etc. If sludge is handled improperly, it might cause secondary pollution to the environment (Status analysis 2017).

The composition of the sludge is very complex. There are certain differences in the sludge characteristics of different components, and there is also a great influence on the treatment and disposal methods of the selected sludge. Therefore, it is very important to analyse the composition of the sludge. The composition of domestic sewage sludge in different regions and seasons is different. Different industries and different technological processes will have a huge impact on the composition of industrial sludge.

2.8 Status of sludge treatment and disposal around the world

2.8.1 The United States

There are approximately 16,000 plants that disposal sludge in the United States and it serves 230 million people. The daily sewage treatment volume is 150 million cubic meters. The annual output of sludge is 35 million tons (80% moisture content). There are 650 centralized anaerobic digestion facilities to treat 58% of the sludge; 700 aerobic fermentation stabilization facilities handle 22% of the sludge; the final disposal of sludge is distributed as 60% for agriculture, 3% for ecological restoration, 17 % landfill and 20% incineration (FIGURE 6, Gao 2016).

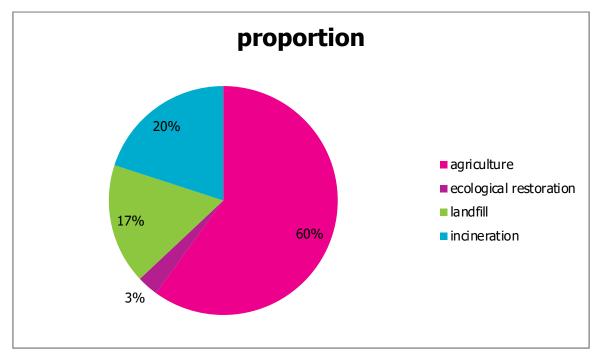


FIGURE 6. The percentage of different methods used of for sludge disposale sludge sludge in USA (Gao 2016)

2.8.2 Japan

Japan's useful land area is small, the land area is about 37.8×10^4 km², but the available area is only 7.95×10^4 km², agricultural land is 4.78×10^4 km², road land is 1.32×10^4 km², commercial residential land is 1.12×10^4 km², and industrial land is 0.16×10^4 km², other land for construction 0.57×10^4 km² (Guo 2014), Therefore, the main method of sludge treatment is incineration in Japan. Since 1980, about two-thirds of the sludge have been treated by incineration and eventually used in cement raw materials, roadbed and construction materials. In recent years, due to the continuous development of sludge pyrolysis, dry distillation, and gasification technologies, some adjustments have been made in the treatment and disposal of sludge, reduce the incineration of sludge and pay more attention to the biomass energy utilization of sludge. The future development trend of sludge treatment and disposal in Japan focuses on the resource utilization of sludge (Ding 2017).

2.8.3 Europe

The primary approach to sludge treatment is landfill and land use in Europe. The trend has an increasing proportion of land use in recent years, and most European countries are increasingly supporting land use for sludge. At present, Germany, the United Kingdom and France produced 2.2 million tons, 1.2 million tons and 850,000 tons of sludge each year (all dry weight), and the proportion of land-use has reached 40%, 60% and 60% (Lu et al. 2016).

2.8.4 China

At present, sludge treatment and disposal is still not developed in China. There are less than onehalf of sludge stabilization treatment facilities in the existing sewage treatment facilities, less than one-tenth of the complete treatment processes and ancillary equipment, A smaller number is normally operational and mainly concentrated in large-scale wastewater treatment plants with the daily capacity of over 150,000 m³ (Xu 2010). But in recent years, the treatment and disposal of sludge have gradually received attention.

- 2.9 Municipal sludge treatment and disposal technologies
- 2.9.1 Aerobic fermentation

Aerobic fermentation technology is a biological treatment technology that utilizes the microorganisms in sludge for fermentation. It does not pollute the environment in practical application (Wang 2013). It can oxidize a portion of organic matter to inorganic matter, provide the energy needed for the growth of microorganisms, and convert some organic matter into nutrients needed by microorganisms to synthesize new cells. The aerobic fermentation process is shown in FIGURE 7.

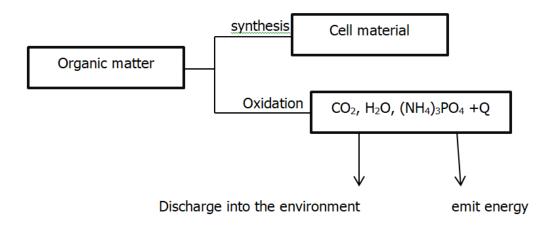


FIGURE 7. Aerobic fermentation process (Wang 2013)

2.9.2 Landfill

This treatment method is simple, and cheap. Sludge does not require a high degree of dehydration, However, there are also some problems with landfill, especially landfill leachate and gas formation. Leachate is a heavily contaminated liquid which can pollute the groundwater environment if landfills are not sited or operated properly. The gas produced in landfills is mainly methane, which can cause explosions and burns (Zhao 2018). In terms of operation and management, it is necessary to strictly limit the admission of hazardous wastes, to compact and cover the wastes that are buried daily, and to pay more attention to the maintenance and management of landfills after closure (FIGURE 8).



FIGURE 8. Landfill (Gao 2016)

2.9.3 Aerobic composting

Aerobic composting is the process by which aerobic microorganisms convert organic matter into humus under conditions of full contact with air (Sludge treatment method 2016). In general, the temperature of aerobic compost is high, and it is better to be within 55 - 60 °C, so aerobic compost is also called high-temperature compost. It has a high degradation rate for organic matter, a short amount of days for composting, and a low amount of odor (FIGURE 9).



FIGURE 9. Aerobic composting (http://slideplayer.com/slide/3938129/)

2.9.4 Sludge heat drying

Sludge heat drying refers to the use of thermal energy to heat and dehydrate the dewatered sludge to make it a dry product. The process of sludge drying can be divided into indirect and direct drying (Ding 2017). It is divided into direct heating and indirect heating according to the heat transfer method (Gao 2016). After heat drying, the sludge can be used as fertilizers, soil conditioners, build-ing materials, landfills and alternative energy sources.

2.10 Glycerol

Glycerol (FIGURE 10) is a side product from manufacturing biodiesel industry. Glycerol is also known as glycerin or glycerine. All names refer to the same chemical compound, propane-1,2,3-triol (IUPAC - $C_3H_8O_3$). It was obtained from the undisclosed source. It is a colorless, odorless, viscous liquid

that is sweet-tasting and non-toxic. Glycerol can be used accelerate biogas production, the percentage of 1% glycerol was added to municipal sludge. TABLE 6 shows the properties of glycerol.

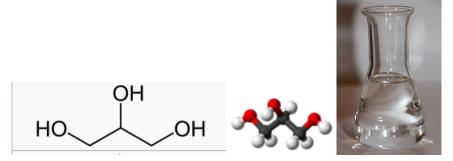


FIGURE 10. Glycerol (Gao 2016)

TABLE 6.	The properties	of glycerol	(Gao 2016)
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Chemical formula	C3H8O3
Melting point	17.8 °C (64.0 °F; 290.9k)
Magnetic susceptibility	-57.06·10 ⁻⁶ cm ³ /mol
Solubility in water	Miscible
Density	1.261 g/cm ³
Refractive index	1.4646
Boiling point	290 °C (554 °F; 563k)
Appearance	Colorless liquid hygroscopic
Vapor pressure	0.0003mmHg(50 °C)
Molar mass	92.09 g⋅mol ⁻¹
Odor	Odorless
Viscosity	1.412Pa·s

3 RESEARCH OBJECTIVES

The main objective of this study was to determine the efficiency of biogas produced from municipal sludge after the addition of glycerol, compare the methane potential of municipal sludge and the same feeding material boosted with glycerol as raw materials in anaerobic co-digestion process to produce biogas. The specific research objectives were:

- Study biogas production and biogas process. Learn to understand the basics of biogas production and feeding materials.
- Determine biogas yield or potential from municipal sludge.
- Determine methane potential of municipal sludge boosted with glycerol.
- Determine municipal sludge degradation and mass reduction.
- Determine municipal sludge with glycerol degradation and mass reduction.
- Compare the performance of biogas produced from different sources.

4 MATERIALS AND METHODS

In this section, the materials and methods used for the experiment will be introduced. The sample for the experiment is municipal sludge. Inoculum and glycerol was used in the experiment too, the methane potential of municipal sludge will be determined through biogas batch tests. In a biogas batch test a sample of organic material is tested to determine how much biogas the tested material produces in certain circumstances within the certain time period. The test was planned to continue for five weeks.

4.1 Inputs for bioreactor

Three materials were used for the experiment, which were inoculum, municipal sludge and glycerol. Municipal sludge was an experimental sample, which required determining methane potential. The inoculum was mixed with municipal sludge in test reactor in order to get the anaerobic digestion process started properly.

Nine anaerobic reactors (volume = 5 L) were set up for comparison, these reactors were divided into three groups, and the reactors in each group are filled with the same materials. The first three reactors were filled with inoculum only, the fourth to sixth reactors were filled with the inoculum and municipal sludge, and the last three reactors were filled with the inoculum, municipal sludge and glycerol (FIGURE 11). TABLE 7 shows the actual amounts of material added to each reactor in the experiment.

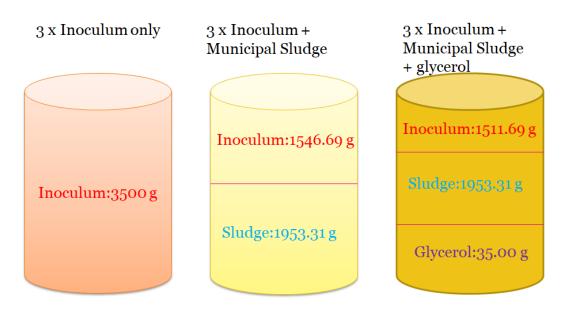


FIGURE 11 Bioreactors setup

Number of reactor	Municipal Sludge, g	Glycerol, g	Inoculum, g
1.0	—		3509.3
1.1	—	_	3500.5
1.2	—	_	3499.2
2.0	1954	_	1545.5
2.1	1961	_	1553.4
2.2	1953	_	1545.6
3.0	1957	34.2833	1523.5
3.1	1953	34.8163	1511.2
3.2	1953	35.2532	1510.6

TABLE 7. Exoerumental plan

4.1.1 Inoculum

The inoculum was collected in January 2018 from biogas plant owned by Natural Resources Institute Finland (Luke) which is located to Maananka Finland (FIGURE 12 and FIGURE 13). It was required to start the biogas process rapidly at the beginning of the batch tests. Before using in the experiments, the inoculum was tested for original total solids (TS) and volatile solids (VS) values (measured according to standard SFS3008), which amounted to 7.59% total solids and 5.82% volatile solids. The inoculum initial pH was measured to be 7.754.



FIGURE 12. The biogas plant of Maaninka (Qiuwang Fan 2018)





FIGURE 13. Taking inoculum samples (Qiuwang Fan 2018)

4.1.2 Municipal sludge

The municipal sludge was obtained from municipal wastewater treatment plant Lehtoniemi (Kuopio Vesi, FIGURE 14 and FIGURE 15) in Kuopio. The main strategy of Kuopion Vesi is to treat the wastewater adequently to requirements and reduce amount of sludge produced. The FIGURE 16 shows the amount of sludge produced per year and TS content in the plant from 2010 to 2016. The municipal sludge composition used in biogas batch test was following: 6.23% TS and 4.61% VS. The municipal sludge pH was 7.127.

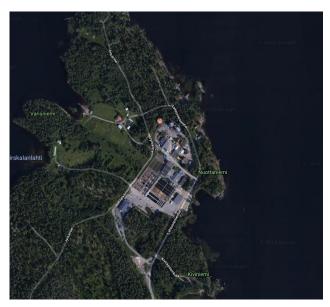


FIGURE 14. Location of Lehtoniemi wastewater treatment plant in Kuopio (maps.google)



FIGURE 15. Acquiring municipal sludge samples (Qiuwang Fan 2018)

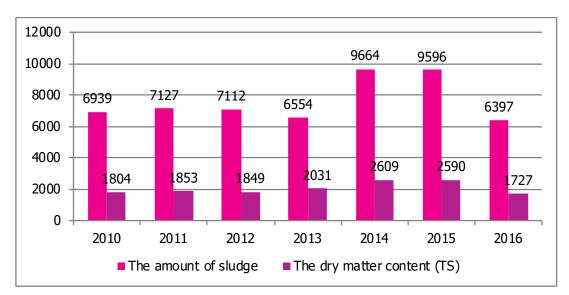


FIGURE 16. The amount of sludge (kg) and TS (kg) content in Lehtoniemi wastewater treatment plant from 2010 to 2016

4.2 Laboratory equipment used in experiments

The following equipment was be used during the experiment:

• The Memmert drying oven (FIGURE 17)

Thermostatically controlled with forced air ventilation, maintaining the temperature of 105 ± 5 °C, used for drying fresh matter in the stage of TS determination.

• Carbolite muffle kiln (FIGURE 18)

Muffle kiln which can be adjusted to temperature 550 ± 25 °C degrees, used for burning dry matter in the stage of VS determination.



FIGURE 17. Memmert UE 400 drying oven (Qiuwang Fan 2018)



FIGURE 18. Carbolite ELF 11/14B muffle kiln (Qiuwang Fan 2018)

• Desiccator (FIGURE 19)

With active drying agent such as silica gel, Used to cool the sample to room temperature in the stage of VS and TS determination.

• Memmert heating cabinet (FIGURE 20)

Used to give the reactor constant temperature of 40 degrees Celsius.



FIGURE 19. Desiccator (Qiuwang Fan 2018)



FIGURE 20. Memmert UF1060^{plus} Heating cabinet (Qiuwang Fan 2018)

• Water-sealed container (FIGURE 21)

Used for measuring the volume of the gas.

• Geotech GA2000 Plus analyzer (FIGURE 22)

Used for determining gas volume and content of the gas (i.e. CH_4 , CO_2 , O_2 , H_2S ...).



FIGURE 21. Water- sealed container (Qiuwang Fan 2018)



FIGURE 22. Geotech GA2000 Plus analyzer (Qiuwang Fan 2018)

• Pure nitrogen gas (FIGURE 23)

Used to remove oxygen from reactor.

• WTW pH3210 pH meter (FIGURE 24)

Used for measuring pH.



FIGURE 23. Pure nitrogen gas (Qiuwang Fan 2018)



FIGURE 24. WTW pH3210 pH meter (Qiuwang Fan 2018)

- Ohaus Adventurer Precision Balance (FIGURE 25)
- Scaltec Analytical balance SBC 31 (FIGURE 26)







(Qiuwang Fan 2018)

FIGURE 26. Scaltec Analytical balance SBC

(Qiuwang Fan 2018)

- Glass reactors (FIGURE 27), each with volume of 5 litres. Three reactors per one sample, used as bioreactor.
- Gas-tight corks with the sealed entry for gas outlet line (FIGURE 28).
- Numbered gasbags with gas lines and gas sealed valves (FIGURE 29).



FIGURE 27. Glass reactor (Qiuwang Fan 2018)



FIGURE 28. Gas-tight cork (Qiuwang Fan 2018) (Q



ork FIGURE 29. Numbered gasbags (Qiuwang Fan 2018)

4.3 Methods

Working phases of the biogas batch test are shown in FIGURE 30, including the calculation of results and conclusions, batch test lasted for five weeks.

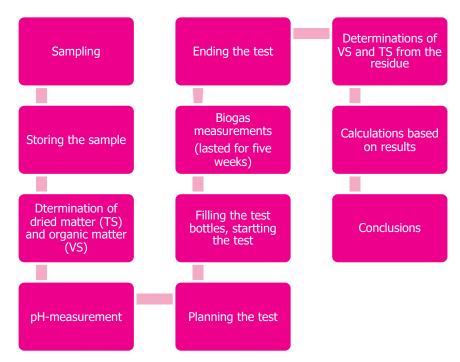


FIGURE 30. Working phases of the biogas batch test

4.3.1 Storage

During storage, samples may change (for example, carbon dioxide, moisture, and other volatiles are ingested or released). These changes may distort results. Bioactive samples should be analyzed within 3 days. If used during this period, samples should be stored at about 4°C, otherwise stored directly at a maximum of -18°C. Other samples can be stored in closed containers between 0°C to 4°C (Janhunen 2014).

4.3.2 Determinations of the dried matter (TS), organic matter (VS) and pH

The batch test begins by determining dry matter (TS - Total Solids) and organic matter (VS - Volatile Solids) concentrations of the sample and inoculum. The batch test is done by using the mixture of sample and inoculum in the test reactors. The dried matter (TS) and organic matter (VS) concentrations are needed for calculating the proper dose of the sample and inoculum in the test reactors. Necessary steps to determine TS- and VS-concentrations are shown in FIGURE 33 (Rantala et al. 2014).

In addition, the pH-level of the mixture should be kept in suitable range (pH 6-8) for the methanogenic microbes. If the pH is too low after filling the reactors it should be neutralized, for example by adding necessary amount of 20% sodium hydroxide into the reactors in order to raise the pH-level between 6 to 8. In addition, inoculum should never be exposed in the pH values under 5. Otherwise methanogenic microbes will be destroyed and test cannot be performed properly (Rantala et al. 2014).

The dry matter of the samples was determined by evaporation and drying the known amount of sample at 105 °C degree and weighing the residue (FIGURE 31). Ash is determined by drying resi-

due of sample at 550 °C degree and weighing the residue (FIGURE 32). The analysis should performance in triplicate samples.

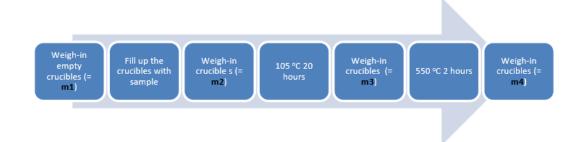




FIGURE 31. Samples after drying fresh matter, in 105 °C, for 20 hours (Qiuwang Fan 2018)

FIGURE 32. Samples after burning dry matter, in 550 °C, for 2 hours (Qiuwang Fan 2018)

Samples are dried to constant mass in an oven at 105 ± 5 °C. The difference in mass before and after the drying process is used to determine the dry matter and the water content. The annealing temperature is selected 550°C because it is sufficiently high to break down organic substances without any disappearance of inorganic substances (Janhunen 2014).





Calculating the dry matter and organic dry matter concentrations are expressed as a percentage of mass or grams per kilogram using the following equations:

$$TS = \frac{m_3 - m_1}{m_w}$$
(1)

$$Ash = \frac{m_4 - m_1}{m_w}$$
(2)

$$VS = TS - Ash$$
(3)

where:

4.3.3 Calculation of the dose for the bioreactor

The batch tests are made in 5-liter glass reactors, in which case the total dose of the sample and inoculum together is recommended to be around 3500g. The amount of inoculum used in biogas batch tests is roughly about half of the test reactor filling volume. However, the precise ratio of the inoculum and the sample can be calculated based on initial VS-concentrations of both materials.

4.3.4 Procedure of loading the bioreactor

Phases and procedure of preparing bioreactor for testing were the following:

- Mark the reactors and the gasbags with masking tape. Prepare three parallel samples for each test. Parallel test reactors loaded only with inoculum were marked 1.0, 1.1 and 1.2. Parallel test reactors filled with inoculum and municipal sludge mix were marked 2.0, 2.1 and 2.2. Parallel test reactors filled with mix of inoculum, municipal sludge and glycerol were marked 3.0, 3.1 and 3.2.
- Measure weight of each empty reactor.
- Measure the sample and the inoculum pH separately.
- Mix the municipal sludge properly and measure weight of the pre-calculated amount of the municipal sludge to the reactor.
- Mix the inoculum properly and add the pre-calculated amount of the inoculum to the reactor.
- Mix the mixture and measure the pH.
- Use nitrogen gas to remove oxygen from the reactor.
- Close the reactor with a cork.
- Attach the empty gas bag to the cork and open the valve.
- Place the reactor in the heating cabinet.



FIGURE 34. Preparationse to add substances in lab (Qiuwang Fan 2018)



FIGURE 35. All substances have been added (Qiuwang Fan 2018)



FIGURE 36. The beginning of experiment on 25.2.2018 (Qiuwang Fan 2018)

4.3.5 Biogas measurement

The content and the volume of the gas produced during the batch test are measured with GA2000 Plus analyzer, FIGURE 37 shows its accuracy (according to manufacturer). The analyser determines the methane (CH₄), carbon dioxide (CO₂), oxygen (O₂) and other gases (BAL) percentage values in the gas. In addition, the analyser gives the values for hydrogen sulfide (H₂S), ammonia (NH₃), temperature and air pressure have to be recorded so the results can be altered to match normal conditions (NTP) (Rantala et al. 2014).

Gas Accuracy*	CH ₄	CO2	0 ₂
0-5%	±0.5%	±0.5%	±1.0%
5-15%	±1.0%	±1.0%	±1.0%
15% - Full Scale	±3.0%	±3.0%	±1.0%



FIGURE 37. Gas measure accuracy with meter GA2000

FIGURE 38. Measuring biogas composition with GA2000 plus analyzer (Qiuwang Fan 2018)

The biogas batch test lasted for five weeks. At the beginning of the biogas batch test, more gas was produced and it was measured in every two days, but in the end, only once a week. FIGURE 39 shows the changes in reactor 1.0 during the experiment, FIGURE 40 shows the changes in reactor 2.1 during the experiment, FIGURE 41 shows the changes in reactor 3.1 during the experiment. No

color change was found in the three reactors, after a period of reaction, a thick material will form in the reactor, so it is necessary to shake the reactor every day to ensure sufficient mixing, otherwise the reaction will slow down but it will not stop.





02/02/2018 12/02/2018 FIGURE 39. Reactor 1.0 (Qiuwang Fan 2018)



01/03/2018





09/02/2018 02/02/2018 FIGURE 40. Reactor 2.1 (Qiuwang Fan 2018)



16/02/2018



02/02/2018



16/02/2018 FIGURE 41. Reactor 3.1 (Qiuwang Fan 2018)



01/03/2018

4.3.6 Ending the test and close procedure

After the final gas measurements the following steps are taken:

- Measure weight of each reactor.
- Measure the pH value of the tested mixture. •
- TS and VS concentrations are determined in the same way as the beginning of the test.



FIGURE 42. Measuring pH of reactor (Qiuwang Fan 2018)



FIGURE 43. Weighing the reactor (Qiuwang Fan 2018)

5 RESULTS AND DISCUSSION

5.1 Mass reduction of inputs for bioreactor

The mass reduction is at the normal level, it is always under 5 % in the wet-digester process. TA-BLE 8 shows the average of three sets of parallel experiments mass reduction. The mass reduction represents the of material efficiency biodegradation, the greater the mass reduction, the higher the efficiency of material biodegradation. It can be seen from TABLE 8, that highest mass reduction is 3.0%. This means that the efficiency of the reactor with inoculum, sludge and glycerol is the highest.

TABLE 8. The average mass reduction of inputs obtained in experiments

Inputs for bioreactor	Average mass reduction (%)
Inoculum	1.1 ± 0.1
Inoculum & sludge	2.0 ± 0.7
Inoculum & sludge & glycerol	3.0 ± 0.1

5.2 Calculation of methane potential

The methane potential of inoculum, municipal sludge and the methane potential of glycerol added to municipal sludge has been obtained.

TABLE 9. The methane potential of inoculum

Nm ³ CH ₄ /t FM	Nm ³ CH₄/t TS	Nm³ CH₄/t VS
2.37	31.19	40.63

TABLE 10. The methane potential of municipal sludge with inoculum

Municipal sludge with inoculum			
Number of reactors	Nm ³ CH ₄ /t FM	Nm ³ CH ₄ /t TS	Nm³ CH₄/t VS
2.0	7.04	103.06	136.80
2.1	6.61	96.81	128.50
2.2	8.62	126.19	167.50
Average	7.42	108.68	144.26
standard deviation	1.06	15.48	20.54

TABLE 11. The methane potential of municipal sludge without inoculum

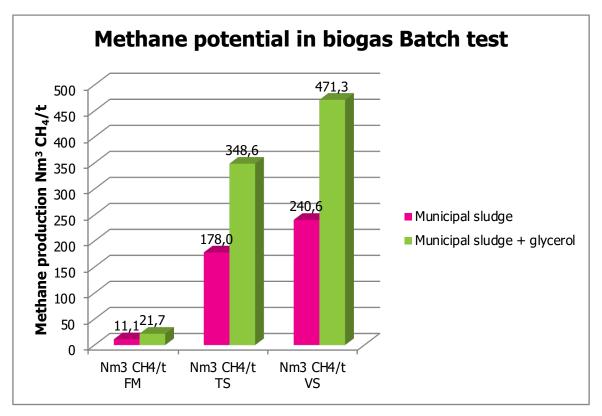
Municipal sludge without inoculum			
Nm³ CH₄/t FM Nm³ CH₄/t TS Nm³ CH₄/t VS			
11.1 178.0 240.6			

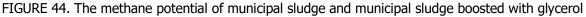
Municipal sludge boosted with glycerol			
Number of reactors	Nm ³ CH₄/t FM	Nm ³ CH ₄ /t TS	Nm ³ CH ₄ /t VS
3.0	28.70	196.36	260.68
3.1	25.01	201.10	267.00
3.2	23.54	194.17	257.80
Average	25.75	197.21	261.83
standard deviation	2.66	3.54	4.7

TABLE 12. The methane potential of municipal sludge boosted with glycerol with inoculum

TABLE 13. the methane potential of municipal sludge boosted with glycerol without inoculum

Municipal sludge boosted with glycerol without inoculum			
Nm³ CH₄/t FM Nm³ CH₄/t TS Nm³ CH₄/t VS			
21.7	348.6	471.3	





The Biogas production of raw materials can be used with almost any kind of organic material. The desirable features: high-calorie content, easily degrade, simple handling. TABLE 14 shows the methane potential of some feedstocks, in which the animal by-products had the highest methane yield, and it can produce 150 Nm³CH₄ from per ton of fresh matter, and 570 Nm³CH₄ from per ton of organic dry matter.

TABLE 14. The methane potential of some feedstocks (Janhunen 2017)

FEED	Nm ³ CH ₄ / Mg FM	Nm ³ CH ₄ / Mg VS
------	-----------------------------------------	-----------------------------------------

Animal by-products	150	570
Biowaste	100-150	500-600
Crops	30-150	300-450
Manure, pig	17-25	300-400
Manure, cow	7-14	100-250
Sewage sludge	5-12	200-400

5.3 Discussion of results

Anaerobic digestion has been maintained at around 40 °C throughout the process. Glycerol was added as an easily biodegradable carbon source to the sample for comparison, and it is a by-product of the biodiesel industry. Based on the results of the study, the following conclusions can be made:

TABLE 10 shows the methane potential of municipal sludge with inoculum. The calculated relative deviation is relatively high, the reason for this result may be that when the composition of the gas was measured on January 29, the gas bags marked 2.1 were damaged, so the gas was not obtained. Reactor 2.3 got a higher value, it may be that the inoculum or municipal sludge is not fully mixed when inoculum or municipal sludge was added to the reactor.

Compared to the various raw materials in TABLE 14, the potential of municipal sludge without glycerol is similar to the sewage sludge. The potential for municipal sludge plus 1% glycerol is between animal by-products and biowaste, in other words, Adding 1% glycerol to municipal sludge can greatly help biogas production.

TABLE 10 and TABLE 12 shows that the methane potential does not deduct the methane potential of the inoculum. FIGURE 44 shows the results without inoculum's methane potential, it indicated that the municipal sludge added with 1% glycerol shows double the methane potential without adding glycerol. This is primary was result obtained by other researchers.

There are several works evaluating the use of glycerol in anaerobic digesters. Mixtures of cow dung with 2% glycerol significantly increase the specific methane production (224 to 382 CH 4 g -1 volatile solids d-1) and higher organic material removal compared to the digester without the addition of additives (37 to 51%) Glycerine (Santibáñez et al. 2011). Batch testing also confirmed that the use of smaller amounts of glycerol (0.5% and 1%) produced the highest biogas and methane production. Glycerol is also recommended as a good co-substrate for anaerobic digestion of swine manure (Wohlgemut 2018). Previous studies using small reactors have shown that co-digestion of feces with a small amount of glycerol (about 1-2%) can double biogas production, but if glycerol exceeds 2% (volume-based), toxicity can occur. (Sell et al. 2011). As can be seen in other examples from literature, the amount of biogas produced is different for different for substrates with the same amount of glycerol, the amount of biogas produced is different for

same substrates with the different amount of glycerol. The substrate used in this thesis was municipal sludge, the result is similar to hog manure as a substrate.

TS-VS reduction may have higher uncertainty. The reason for this result is not enough shaking the reactor before analysis, and the supervisor guide that the TS/VS-determination after biogas batch test should do two to three parallel experiments, but this study just do one time.

38 (41)

6 CONCLUSIONS AND RECOMMENDATION

6.1 Conclusions

The main purpose of the study was to compare the methane potential of municipal sludge and same feeding material boosted with glycerol as raw materials in anaerobic co-digestion process to produce biogas and to compare the performance of biogas produced from different sources. Based on the results obtained, municipal sludge added with 1% glycerol has a great methane potential in anaerobic co-digestion, the following conclusions can be made:

- Municipal sludge has a good potential for biogas production, it can produce 11.1 Nm³CH₄/ t FM, 178.0 Nm³CH₄/ t TS and 240.6 Nm³CH₄/ t VS.
- Adding 1% glycerol can approximately double the amount of biogas and methane than municipal sludge without glycerol, it can produce 21.7 Nm³CH₄/ t FM (or 95.5% more), 348.6 Nm³CH₄/ t TS (or 95.8% more) and 471.3 Nm³CH₄/ t VS (or 95.9% more).
- The mass reduction of municipal sludge is 1.9%, the mass reduction of municipal sludge with glycerol is 3.0%.

6.2 Recommendation

As raw materials for biogas production, municipal sludge is qualified, and it is recommended that municipal sludge should be reasonably used to produce biogas. Municipal sludge with 1% glycerol has a great potential, but it is unclear what the optimal amount of glycerol added to municipal sludge is, for future study, it is recommended to add varying amounts of glycerol in the municipal sludge to find the optimal amount.

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