Heating Value and Energy Recovery Potential of Sewage Sludge and Suspended Solids in Municipal Wastewater Treatment Plant

Yahya Mahzoun

2018

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都市下水処理場における

下水汚泥及び下水中汚濁粒子の発熱量と

エネルギー回収可能性に関する研究

Yahya Mahzoun

2018

Abstract

Water is used for many human activities apart from being a key need for life. Wastewater treatment in modern societies is a necessity, but results in the production of sewage sludge.

Globally, million tons of sewage sludge is produced from municipal wastewater treatment plants (WWTPs) every year. In Japan, approximately 2.2 million tons in dry base of sewage sludge was produced in 2015.

Higher heating value (HHV) is an important parameter for energy recovery from sewage sludge. Thus, to make HHV analysis faster and easier, it is essential to predict it from other parameters such as volatile matter (V). An updated equation; HHV ＝ 4.186× (50.2V＋308) is proposed for sewage sludge HHV prediction. This equation is statistically different from that presented 30 years ago.

Thermogravimetry and differential thermal analysis (TG-DTA) is a fast and accurate method to evaluate sewage sludge volatile matter and lower heating value. The V contents measured by a typical sewage testing method and those deduced from the TG curve were strongly correlated. The average correlation error and standard deviation of lower heating value prediction were approximately 5 and 4%, respectively.

Finally, particle size distribution and their heating values in influent, primary sludge, and primary treated wastewater were evaluated and it was observed that small particles exerted the greatest effect on heating value characteristic of the wastewater.

Key words: Wastewater, Sewage sludge, Heating value, TG-DTA, Particle size distribution.

Introduction

Sewage sludge, the liquid phase of biological and non-biological solids removed from sewage, can be divided into two types: primary (produced during primary treatment) and secondary (produced during secondary treatment). The solid concentrations of raw primary and secondary sludge are 2–7% and 0.4–1.5%, respectively.

Several million tons of sewage sludge is produced from municipal wastewater treatment plants (WWTPs) annually. For example, approximately 1.1 million tons in dry base of sewage sludge was produced in the United Kingdom in 2012. In the United States, it is estimated that 12.6 million tons in dry base of sewage sludge was produced in 2017. In Japan, approximately 2.2 million tons in dry base of sewage sludge was produced in 2015.

Sewage sludge from municipal WWTPs contains organic matter. Researchers indicated that organic matters cause increase of heating value. Interest in recovering energy from sewage sludge is increasing due to the disadvantages of traditional methods of waste disposal.

Because sewage sludge contains organic matter, it may have utility as a renewable energy resource. In this study, the renewable energy potential of municipal WWTPs was investigated.

Dewatered sewage sludge was collected from municipal WWTPs throughout Japan in all seasons. Proximate and ultimate analyses of samples were conducted and their heating values were measured. The characteristics of sewage sludge samples were compared to those reported previously, and the effect of sewage developments was assessed. The relationship between heating value and the values determined by

proximate and ultimate analyses was investigated and an equation for estimating heating value was derived.

Thermogravimetric and differential thermal analysis (TG-DTA) is a novel method of investigating the effect of heat on sample characteristics. The feasibility of heating value analysis of organic matter (such as sewage sludge) was assessed in this work.

In the second phase of the study, TG-DTA of sewage sludge was performed. Sample ignition loss during heating (to 650°C) was compared to the volatile matter content analyzed by standard method. The relationship between sewage sludge treatment process and DTA peak shape was evaluated.

Energy recovery from primary sludge (PS) in primary sedimentation tanks (PST) was investigated. The particle size distribution of raw sewage is a key parameter for PST. Larger particles readily settle and can be removed by primary sedimentation; however, the heating values of particles of various sizes have not been determined due to insufficient sample quantities. Heating value analysis of each particle was performed using the DTA peak area, and the removal rates of particles of various sizes according to the PST surface area were calculated. Finally, the energy and mass balance of WWTP was calculated.

Materials and Methods

32 types of dewatered sludge were collected and sampling was conducted in each season and a total of 120 samples was collected. Volatile matter (V) and ash content (A), was conducted according to the Japanese Wastewater Examination Method and ultimate analysis was conducted using a CHN analyzer (JM-10, J-Science Lab Co. Ltd., Kyoto, Japan) and an X-ray fluorescence spectometer (XRF-1800, Shimadzu

Co. Ltd., Kyoto, Japan). The difference between V and other volatile parameters is indicating percentage of oxygen (O).

TG-DTA of dry dewatered sludge was performed using a differential thermal balance (Thermo Plus EVO 2, TG 8120; Rigaku). 10 mg –DS of the sample was heated from ambient temperature to 650°C at 10°C/min under 50 mL/min air flow (Air-zero A; Sumitomoseika).

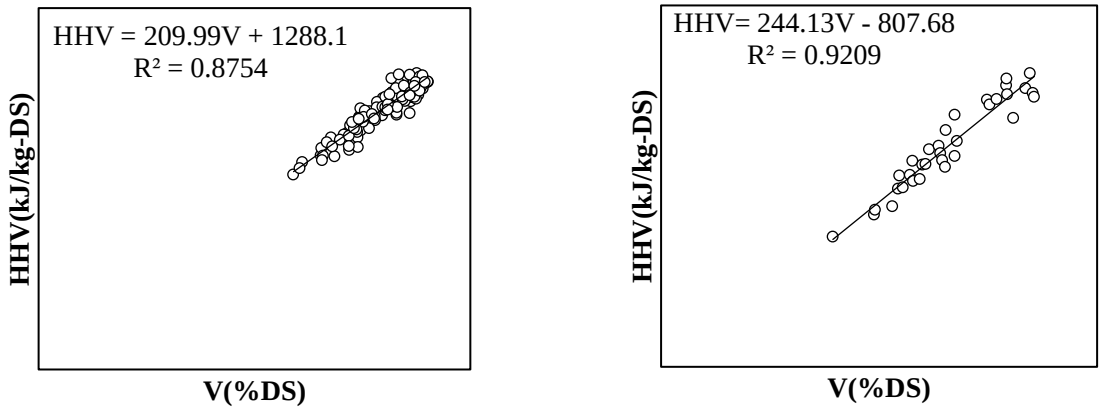
Using the obtained TG curve, the rate of weight decrease from room temperature to the temperature at which the weight reduction rate become almost zero was taken as the predicted V-value. In addition, the DTA curve area was calculated by integrating the exothermic peak area of sewage sludge obtained during heating that is exemplify lower heating value (LHV) of sample.

Particle size analysis was carried out by sieving and filtration. The samples were passed through 1,000-, 500-, 250-, 150-, 75-, 45-, and 20-μm sieves (JIS Z 8801, Tokyo Screen Co. Ltd., Tokyo, Japan), followed by vacuum filtering through a glass-fiber filter (GS-25; φ 47 mm, Advantec, Tokyo, Japan) to remove 1-μm particles. Finally heating value of each particle range was analyzed by DTA peak area.

Results and discussion

The volatile matter content, HHV, and organic matter content of sewage sludge in Japan has increased in the past 30 years. The quality of organic matter (HHV/V) has decreased due to increases in oxygen concentration. An updated equation: HHV ＝ 209.99×V+1288.1 is proposed for the HHV prediction for sewage sludge. This equation is statistically different from that presented 30 years ago (see Fig.1). This equation can predict the HHV of sewage sludge within an average relative error of

2.47% however, average relative error can be reduced to within 1.41% when C, H, and N are used as variables.



a b

Figure1: Correlation between volatile matter and HHV (a. this study and b. previous study).

The ability of TG-DTA to predict the V content and LHV of sewage sludge was investigated. The V contents measured by a typical sewage testing method and from the TG curve were strongly correlated (average relative error and standard deviation, 1.02 and 0.62%, respectively). Thus, the V content of sludge samples can be predicted by TG.

The LHV calculated by DTA and that determined by the standard method exhibited an approximately 1:1 correlation (Fig.2). The average correlation error and standard error of period A (spring) and B (winter) samples were 5.20% and 4.00%, and 5.17% and 4.08%, respectively. Thus, this method can be applied to sewage sludge samples obtained during other seasons, or from other WWTPs.

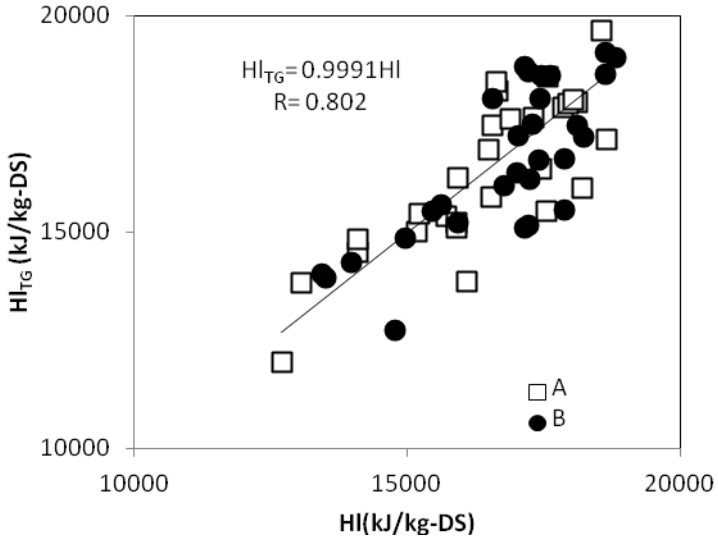


Figure2: Relationship between LHV obtained by the standard method (Hl) and that obtained by DTA (Hl_{TG}).

We analyzed the particle size distribution (PSD) and heating values of them in influent, PS, and primary treated wastewater (PTW) of two WWTPs in Japan. The particle size of wastewater decreased during the primary sedimentation process. The PSD influenced the heating value distribution of the wastewater. Small particles exerted the greatest effect on heating values characteristic of the wastewater suspended solids (SS), because they predominated over other particle sizes in the PTW. Approximately 75% of the energy in SS in influent can be recovered in the PST; the remainder is lost as SS in the PTW of both WWTPs. However, the energy recovery rate decreased with decreasing particle size, indicating that removal efficiency decreased with decreasing particle size, and the energy within those particles was lost.

Conclusion

In this study, we investigated heating value and energy recovery potential from sewage sludge and suspended solids of wastewater. It was illustrated that organic component of sewage sludge has changed during past 3 decades and heating value

of it increased. Heating value also can be estimated by DTA properly. However, particle size distribution is an important parameter to recover energy from sewage because smaller particles are harder to remove.

Contents

Chapter 1
General Introduction

1.1 Sewage sludge and its origin

Consumption of materials by humankind results in the production of various types of liquid and solid waste. Safe disposal of these materials requires much effort and planning.

Water is used for many human activities apart from being a key need for life. Much of the wastewater produced by these activities is sent to wastewater treatment plants (WWTPs). Wastewater consists of less than 0.01% solids, and so must be treated before being discharged to receiving water to prevent environmental contamination and adverse effects on humans and wildlife.

Wastewater treatment in modern societies is a necessity, but results in the production of sewage sludge. Sewage sludge is the concentrated phase of solids in wastewater produced by primary and secondary treatments. A flow chart of typical WWTP and sludge processing is shown in Figure 1-1.

Sewage sludge, the liquid phase of biological and non-biological solids removed from sewage, can be divided into two types: primary (produced during primary treatment) and secondary (produced during secondary treatment). The solid concentrations of raw primary and secondary sludge are 2–7% and 0.4–1.5%, respectively (Turovskiy & Mathai, 2006). Sewage sludge is considered to be biomass and contains abundant organic matter, which can be reused as fertilizer or for energy recovery (Burton *et al*., 2003).

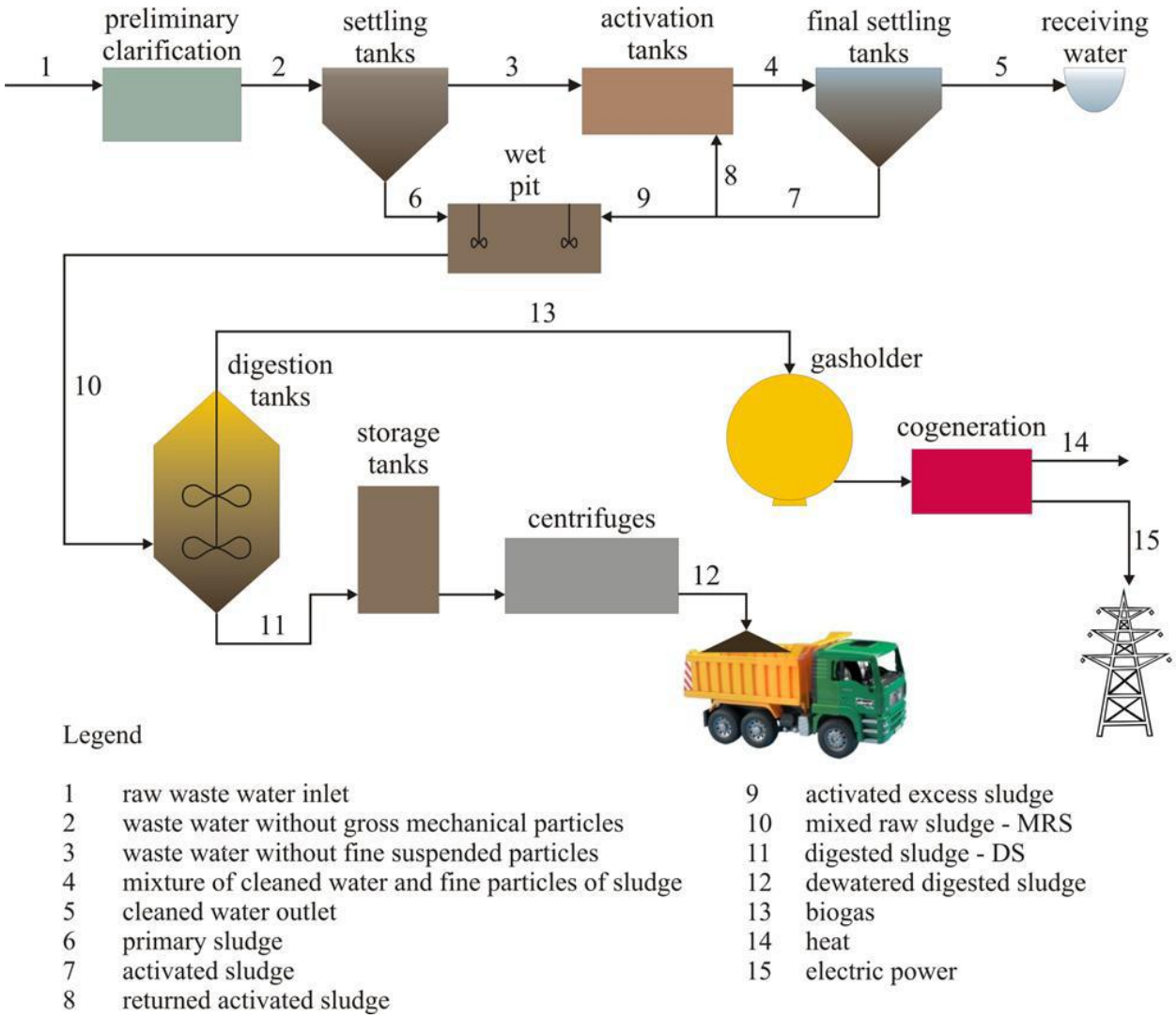


Figure 1-1: Scheme of typical WWTP and sewage sludge processing (Houdková et al., 2008).

Several million tons of sewage sludge is produced from municipal WWTPs annually. For example, approximately 1.1 million tons in dry base of sewage sludge was produced in the United Kingdom in 2012 (Eurostat, 2016). In the United States, it is estimated that 12.6 million tons in dry base of sewage sludge was produced in 2017 (Seiple *et al.*, 2017). In Japan, approximately 2.2 million tons in dry base of sewage sludge was produced in 2015 (MLIT, 2015). In the Beijing urban area, about 2,400 tons of dry sewage sludge is produced per day; by 2015, sludge production by urban WWTPs is expected to reach 3,300 tons/day. In Shanghai, the total sludge

production was 3,200 m^3/day in 2012 and 4,200 m^3/day 2015. In 2020, sludge production is projected to be 6,000 m^3/day (Asian Development Bank, 2012).

Management of produced sewage sludge is one of the most important function of WWTPs, and requires continuous effort and expenditure. Raw sewage sludge is over 95% water and so must be subjected to processing procedures such as concentration, disinfection, stabilization, and dewatering.

1.2 Sewage sludge as an energy resource

The imperative to reduce fossil fuel use and increasing global energy demand continues to drive international focus on renewable energy. Biomass is one of the most well-known sustainable energy resources.

About 80% of global energy consumption is supported by fossil fuels (Figure 1-2), compared with 20% by renewables. At least 10% of the energy consumed globally originates from biomass. Indeed, the total energy locked in biomass is eight-fold the annual global energy requirement. Moreover, the energy that could be extracted from biomass is equal to 1.1–1.5-fold the projected global energy demand in 2050 (Lins *et al.*, 2014; Midilli *et al.*, 2002; Ladanai & Vinterbäck, 2009).

Sewage sludge from municipal WWTPs contains much organic matter. Researchers indicated that organic matters cause increase of heating value; therefore, sewage sludge represents a potential source of energy (Rulkens, 2007). Interest in recovering energy from sewage sludge is increasing due to the disadvantages of traditional methods of waste disposal (landfill), such as leachate production and adverse effects on ground water and the atmosphere. Therefore, energy recovery from sewage sludge, which is a sustainable, clean, efficient, and environmentally friendly energy resource, is an important challenge for future generations. Sewage sludge production

is expected to increase in the next decade due to increases in population and urbanization (Salado, 2008).

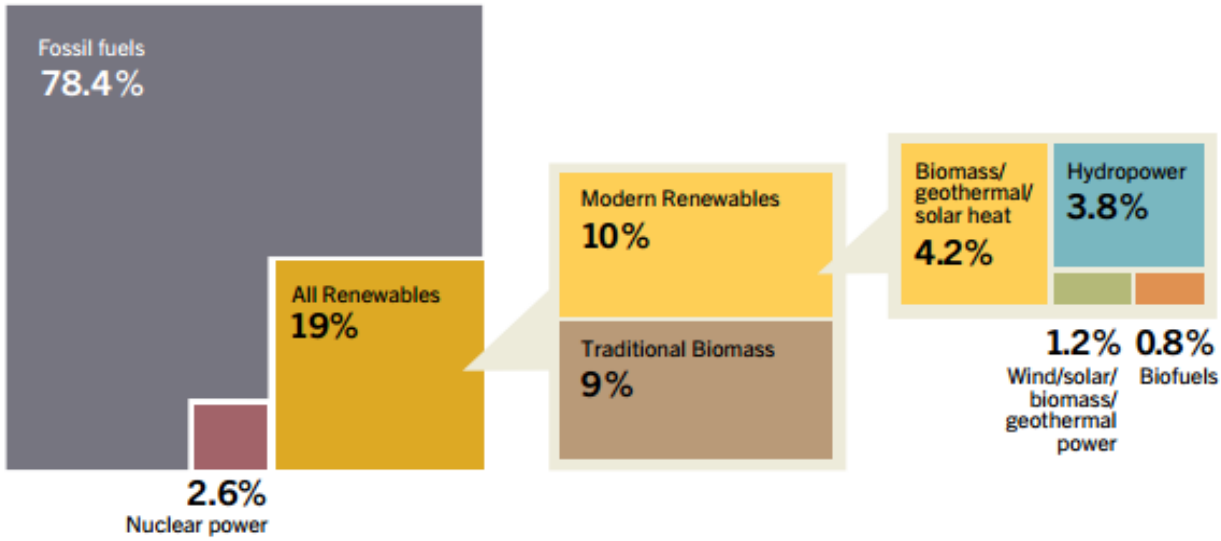


Figure 1-2: Shares of global energy consumption (Lins et al., 2014).

Like other clean energy resources, biomass is a viable alternative to fossil fuels. Sewage sludge can also be used for clean energy production. Various methods can be used to recover energy from sewage sludge; *e.g.*, anaerobic digestion (for biogas recovery), co-incineration and incineration with energy recovery, biofuel production (hydrogen, syngas, and bio-oil), pyrolysis, and gasification (Tyagi and Lo, 2013).

1.3 Research objective

Because sewage sludge contains organic matter, it may have utility as a renewable energy resource. In this study, the renewable energy potential of municipal WWTPs was investigated.

Dewatered sewage sludge (see Figure 1-1) was collected from municipal WWTPs throughout Japan in all seasons. Proximate and ultimate analyses of samples were conducted and their heating values were measured. The characteristics of sewage

sludge were compared to those reported previously (Murakami *et al*., 1988), and the effect of sewage developments was assessed. The relationship between heating value and the values determined by proximate and ultimate analyses was investigated and an equation for estimating heating value was derived.

Thermogravimetric and differential thermal analysis (TG-DTA) is a novel method of investigating the effect of heat on sample characteristics. In the second phase of the study, TG-DTA of sewage sludge was performed. Sample ignition loss during heating (to 650°C) was compared to the volatile matter content analyzed by standard methods. The relationship between sewage sludge treatment process and DTA peak shape was evaluated. The feasibility of heating value analysis of organic matter (such as sewage sludge) was assessed in this work.

Finally, energy recovery from primary sludge (PS) in primary sedimentation tanks (PST) (settling tank in Figure 1-1) was investigated. PS has a higher heating value than activated sludge. Suspended solids likely contain more heating than soluble. The particle size distribution of raw sewage is a key parameter for PST. Larger particles readily settle and can be removed by primary sedimentation; however, the heating values of particles of various sizes have not been determined due to insufficient sample quantities. Heating value analysis of each particle was performed using the DTA peak area, and the removal rates of particles of various sizes according to the PST surface area were calculated. Finally, the energy and mass balance of WWTPs was calculated in terms of the heating value and the rate of PS and WAS production.

1.4 Structure

This Ph.D. thesis is organized as follows (Figure 1-3):

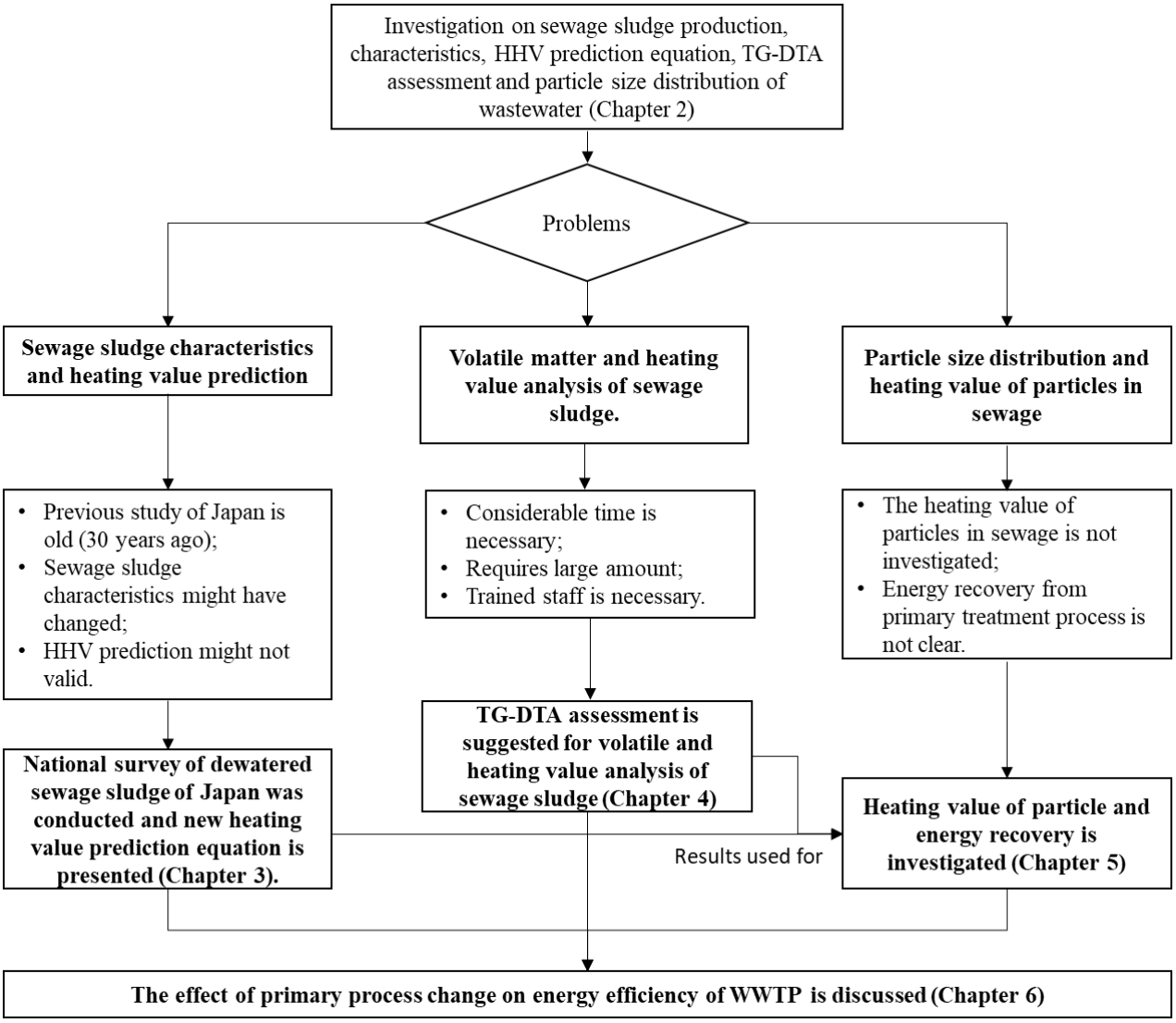


Figure 1-3: Research outline.

Chapter 1 is a general introduction to sewage sludge and sustainable energy consumption. The main purpose of this study; *i.e.*, the feasibility and potential of renewable energy production from municipal WWTPs, is explained in detail.

Chapter 2 is a review of the literature related to energy recovery from sewage sludge and WWTPs. Statistical information on sewage sludge production and disposal methods is presented for the European Union, the United States, China, and Japan. The characteristics of sewage sludge in terms of the moisture content (M); volatile

matter content (V); ash content (A); the percentage of carbon (C), hydrogen (H), nitrogen (N), sulfur (S), and oxygen (O); and the higher heating value (HHV) of sewage sludge are discussed. Finally, the literature on heating value prediction, TG-DTA, and particle size distribution is reviewed.

Chapter 3 describes proximate, ultimate, and heating value analyses of sewage sludge in Japan. The correlation of HHV with C, H, N, S, and O contents of sewage sludge is discussed and compared with previous report. Finally, an equation for predicting the heating value of sewage sludge is presented based on proximate and ultimate analyses.

In Chapter 4, the application of TG-DTA to sewage sludge is investigated. This method is assumed to be applicable to proximate analysis and heating value evaluation of sewage sludge. First, the reliability of TG for measurement of the volatile matter content of sewage sludge is evaluated. Next, the relationship between the DTA exothermic peak area and the lower heating value (LHV) of sewage sludge is assessed. Finally, the feasibility of TG-DTA for proximate and heating value analyses is examined based on the analysis results.

Chapter 5 discusses the heating value distribution of PST processes. First, the particle size distributions of PST influent and effluent and primary raw sludge are determined, and the effect of PST on the particle size distribution of wastewater is investigated. The HHV of several particle range has not been determined to date, and the small sample size precludes the use of bomb calorimetry. Thus, TG-DTA is used to analyze the heating values of particles. The role of particles of various sizes in energy extraction from WWTPs is also examined.

Chapter 6 combines the results obtained in Chapters 3, 4, and 5. First, the effect of changing the surface area of a PST on the removal of particles and suspended solids from the influent is investigated. The characteristics of PS and WAS are discussed. Finally, the heating value and daily energy content of WWTPs according to surface area is calculated.

Chapter 7 presents a summary of the main findings and makes recommendations for future research.

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Chapter 2

Literature Review

2.1 Sewage sludge production and management

Globally, million tons of sewage sludge is produced from municipal wastewater treatment plants (WWTPs) every year. The previous chapter provided a general introduction to sewage sludge and the necessity of energy recovery from sludge; in this chapter, a wide range of information on sewage sludge and suspended solids in wastewater is presented and discussed.

In detail, information on sewage sludge production and management in several countries is presented (where that information is available), and data on sewage sludge characteristics [such as volatile matter (V), percentages of carbon (C), hydrogen (H), nitrogen (N), sulfur (S), oxygen (O), and higher heating value (HHV)] from several countries are introduced.

HHV is an important parameter for energy recovery from sewage sludge. Thus, it is essential to be able to predict HHV from other parameters, to make HHV analysis faster and easier. Thus, articles that discuss HHV prediction from organic materials such as coal and biomass are also presented in this chapter.

Thermogravimetry and differential thermal analysis (TG-DTA) is a novel method for fast and accurate analysis of the ignition behavior of organic materials. Hence, studies on TG-DTA assessment of samples and the feasibility of this method for HHV analysis of organic materials are also discussed.

Finally, the particle size distribution (PSD) of wastewater is investigated to establish the effect of each part of the WWTP process. The aim of this part of the study is to

understand the role of each particle in the heating value distribution in wastewater. Thus, it is necessary to develop an understanding of PSD changes during the WWTP process.

2.1.1 Sewage sludge production and management in the EU, US, and China

It is estimated that more than 10 million tons of dry sludge (DS) is produced in the 27 EU member countries. A significant increase in sewage sludge production has been observed in the EU15 countries (those that were in the EU prior to 1st May, 2004). The total sewage sludge production of these countries increased from 6.5 million tons DS in 1992 to 7.1 million tons DS in 2005 and to 8 million tons DS in 2010 (approx. 20% increase). For the EU12 countries (new members since 2014) sewage sludge production increased from 1.1 million tons DS in 2005 to 1.3 million tons DS in 2013. It is estimated that sewage sludge production will increase further in the future and reach 10.5 million tons DS in the EU15 countries and 2.5 million tons in the EU12 members in 2020. Figure 2-1 shows sewage sludge production in some EU countries from 2005 to 2013. Germany is the biggest sewage sludge producer (approximately 2 million tons DS/year), followed by the United Kingdom, France, Italy, and Spain. About 75% of sewage sludge produced in the EU comes from these five countries (Eurostat, 2017; Milieu Ltd., 2010).

Figure 2-2 shows sewage sludge management methods in some EU countries in 2013, 2012, or 2010 (Eurostat, 2017). Agricultural use and incineration are the most widely used management methods in the EU. Approximately 40% of sewage sludge produced in the EU is managed through agricultural use. Trends in sewage sludge disposal differ by country. For some countries such as Portugal, the United Kingdom, Spain, and France, agricultural use was the most common sludge disposal

method in 2013 or 2012; however, landfill is more common in Italy, and Germany mostly disposes of sewage sludge through incineration.

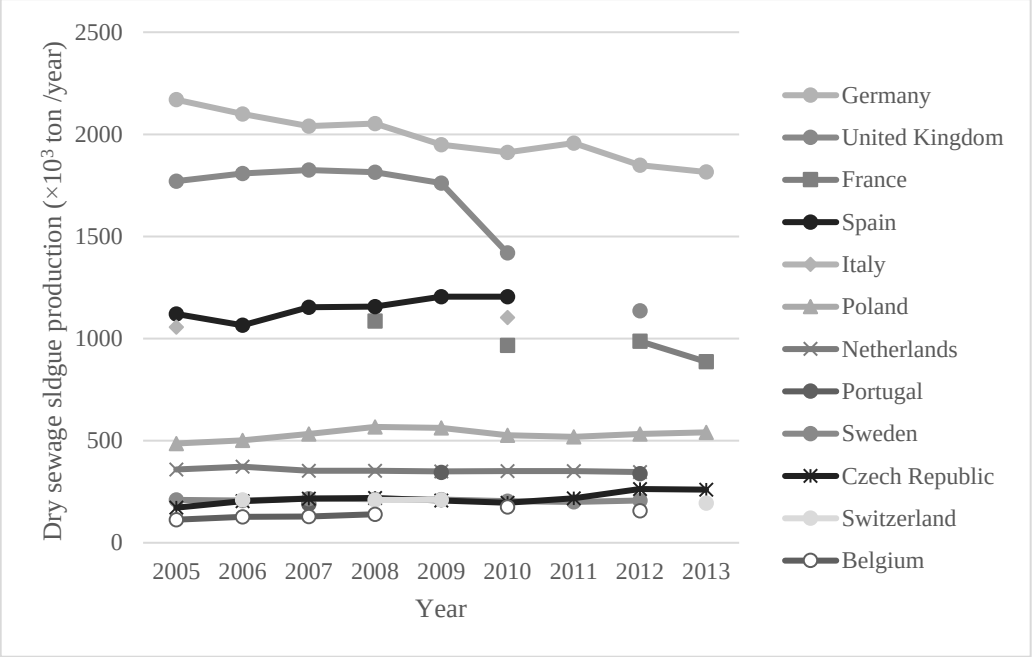
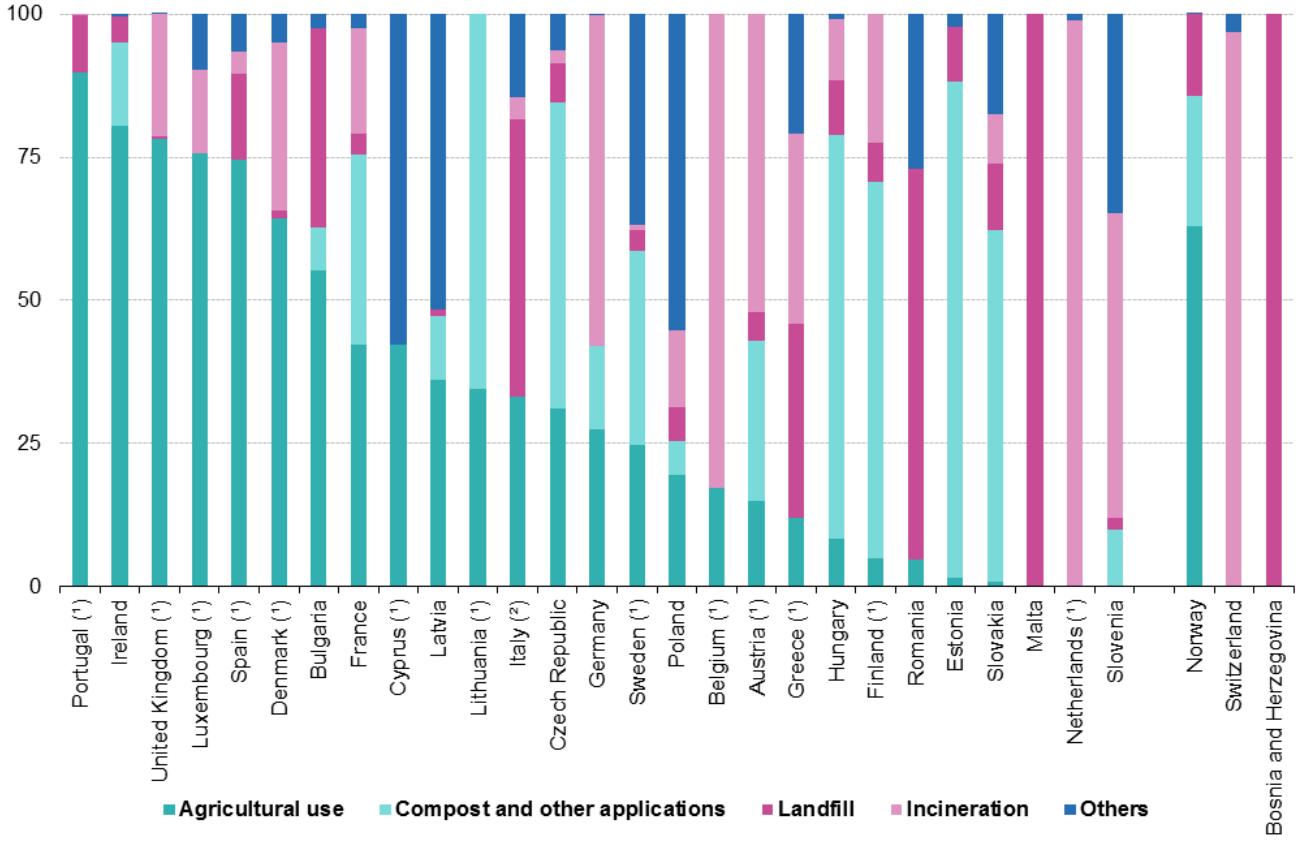


Figure 2-1: Sewage sludge production in some EU countries (Eurostat, 2017).

The most common methods of sewage sludge treatment processing are aerobic and anaerobic digestion, composting, sludge pasteurization, and lime stabilization (Burton *et al.*, 2003). In the EU, digestion is the most common sewage sludge treatment method, and most EU countries use digestion as a sewage sludge stabilization method (Kelessidis *et al*., 2012). Anaerobic digestion is the most cost-effective method because of the high energy recovery and low environmental impact (Mata-Alvarez *et al.*, 2000).

Although there are no accurate data on sewage sludge production in the United States, it is estimated that approximately 7.77 million tons DS was produced in 1988 (EPA, 1992). Figure 2-3 shows the spatial distribution of WWTPs with size in the USA, and Table 2-1 provides some general information on WWTPs. The

information indicates that nearly 240 million people are connected to a WWTP system and approximately 130×10^6 m^3 of wastewater is produced every day.



Note: Croatia: not available.
(1) 2012 data.
(2) 2010 data
Source: Eurostat (online data code: env_ww_spd)

Figure 2-2: Sewage sludge management methods in EU countries in 2013, 2012, or 2010 (Eurostat, 2017).

Figure 2-4 also shows the spatial distribution of sewage sludge production from WWTPs and Table 2-2 provides details on sewage sludge production in different regions. It can be estimated from this information that the total global sewage sludge production in united states is 12.56 million tons DS per year (Seiple *et al.*, 2017); therefore, sewage sludge production in the United States has increased during the past 30 years.

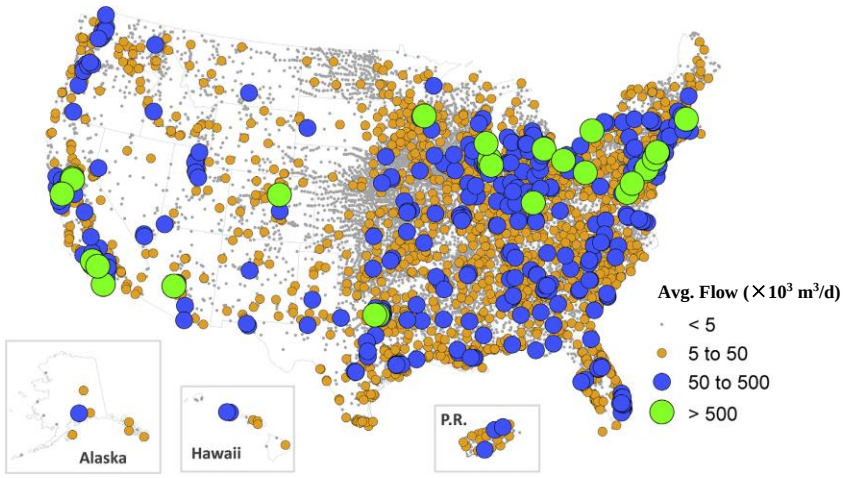


Figure 2-3: Spatial distribution of WWTPs distinguished by daily influent rate in the USA (Seiple et al.*, 2017).*

Table 2-1: Number and percentage of WWTPs, flow rate, and population covered in the USA (Seiple et al.*, 2017).*

Range ($\times 10^6$ m^3/d)	*No. of WWTPs*	*% of WWTPs*	*Total flow* $\times 10^6$ m^3/d	*% of total flow*	*Population served (million)*	*% of population*
≤0.5	6363	42	1.25	1	4.0	2
>0.5 to 5	5520	23	9.95	8	21.4	9
>5 to 50	2686	18	39.48	30	71.4	30
>50 to 500	414	3	51.45	39	94.1	40
>500	31	0.2	28.37	22	47.1	20
Total	**15014**	**100**	**130.5**	**100**	**238.0**	**100**

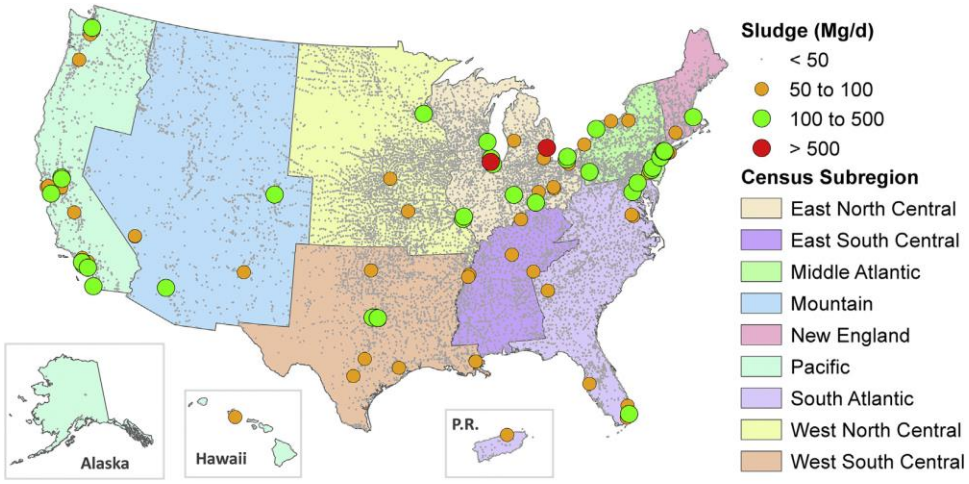


Figure 2-4: Spatial distribution of sewage sludge production in the USA (Seiple et al.*, 2017).*

Table 2-2: Estimated sewage sludge production in the USA by subregion.

Region	*No. of WWTPs*	*Dry sewage sludge production (million tons/y)*
East North Central	2773	2.61
East South Central	1122	0.67
Middle Atlantic	1569	2.03
Mountain	1120	0.67
New England	535	0.56
Pacific	978	1.76
South Atlantic	1914	2.06
West North Central	3126	0.84
West South Central	1877	1.35
Total	**15014**	**12.56**

In China, the number of WWTPs increased dramatically between 2007 and 2013. Figure 2-5 (a) shows that the daily sewage treatment capacity and the number of WWTPs increased from 70 million m^3/d to 150 million m^3/d and 1400 to 3500, respectively, from 2007 to 2013. Figure 2-5 (b) indicates that sewage sludge production increased significantly from approx. 3.5 million ton DS in 2007 to 6.25 million tons DS in 2013. Figure 2-6 shows sewage sludge production in different parts of China (Yang *et al.*, 2015). It can be concluded that the eastern region of China produces more sewage sludge than the Western Central region because of the greater population density, industrialization, and urbanization in the East (Yang *et al.*, 2014).

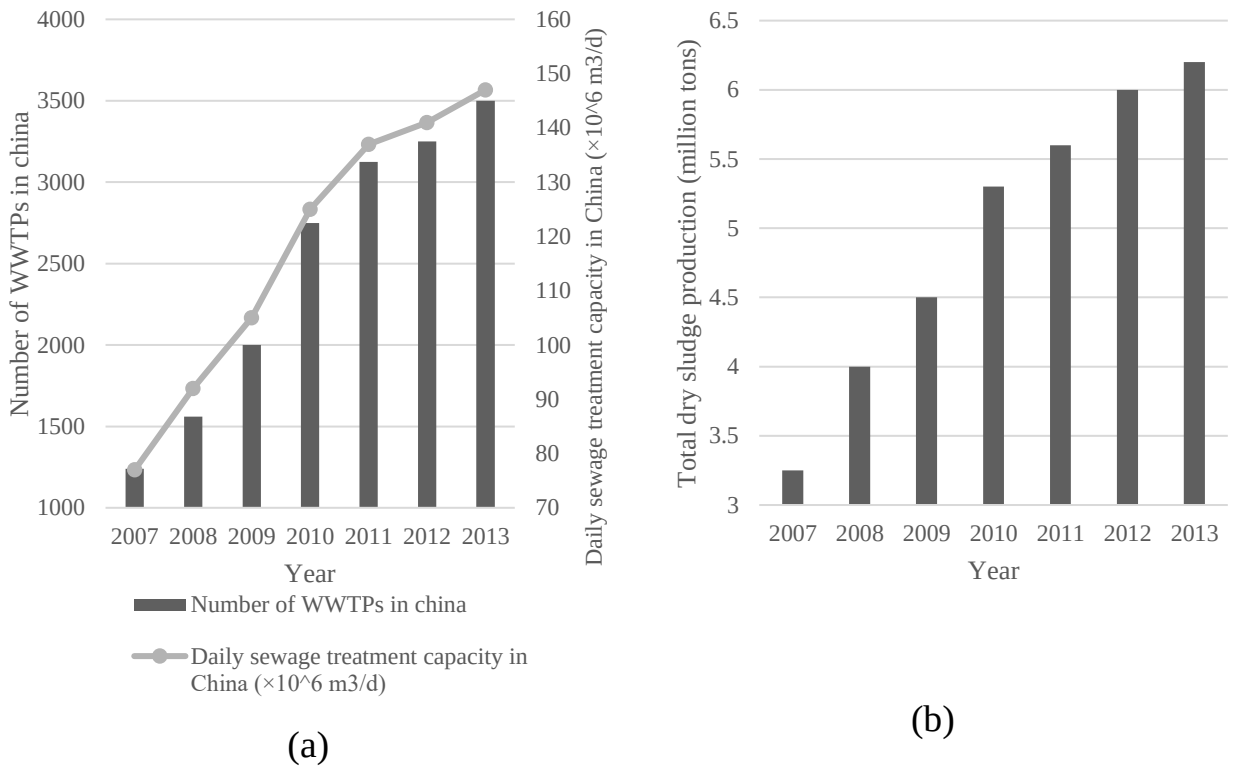


Figure 2-5: a) Daily wastewater treatment capacity and WWTP number, b) Total sewage sludge production in China (Yang et al., *2015).*

In China, thickening, conditioning, dewatering, stabilization, and drying are the most widely used sludge processing methods (Jin *et al.*, 2014). Figure 2-7 shows the percentage of sludge treated using each disposal method in China. More than 80% of sewage sludge is disposed of improperly, such as through improper dumping and sanitary landfill, because these methods are cheaper. After improper dumping, sanitary landfill, land applications, incineration, and building materials are the most widely used sewage sludge disposal methods (Yang *et al.*, 2015).

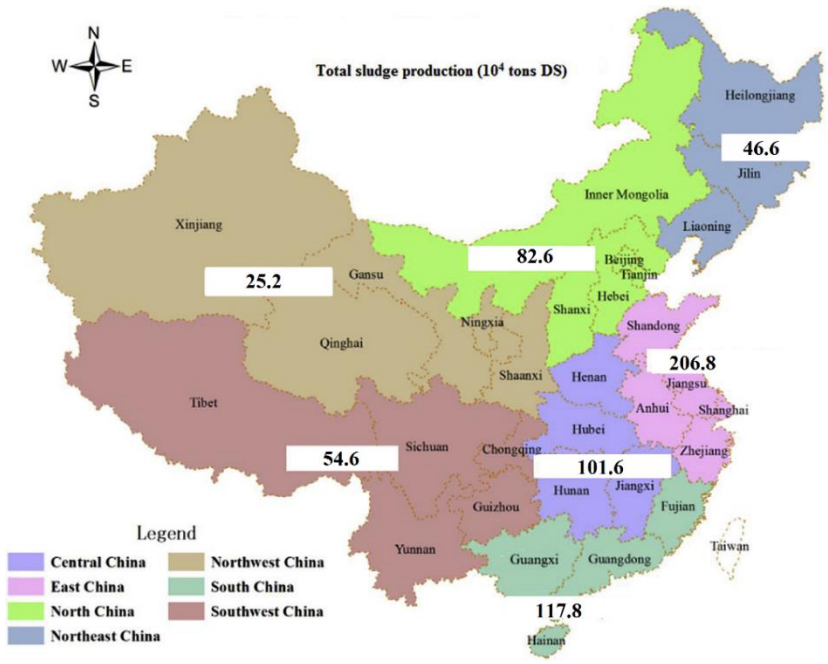


Figure 2-6: Sewage sludge production in different regions of China in 2013 (Yang et al., 2015).

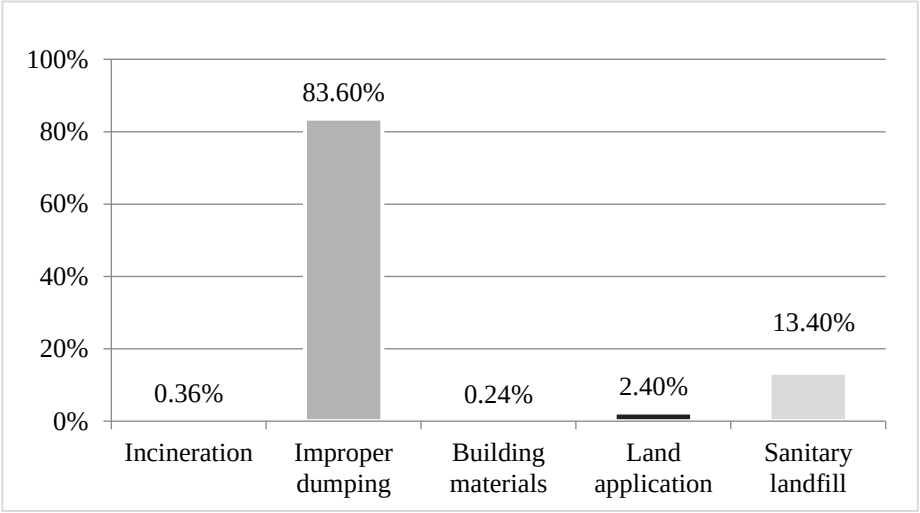


Figure 2-7: Sewage sludge disposal methods in China (Jin et al.*, 2014).*

In this section, sewage sludge production and management in several countries has been discussed. Sewage sludge production has a strong correlation with population and the number of inhabitants connected to WWTPs, and hence an increase in sewage sludge production is expected in developing countries such as China or the

EU12 members. However, developed countries such as the EU15 members, the US, and Japan have produced sludge at a constant level in recent years.

2.1.2 Sewage sludge production and management in Japan

More than 2 million tons of dry sewage sludge is produced in Japan annually. Figure 2-8 shows the annual sewage sludge production and the proportion that was recycled in Japan from 1988 to 2015 (MLIT, 2017). The proportion of sludge landfilled has decreased and the proportion recycled has increased over the past few decades.

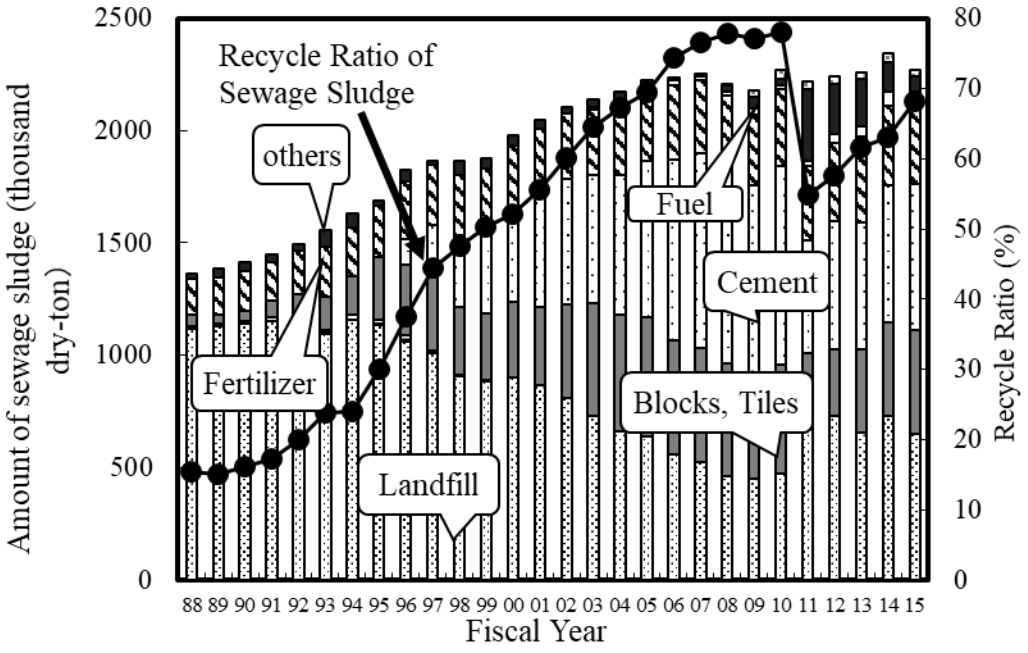


Figure 2-8: Annual sewage sludge production and its management in Japan (MLIT, 2LI7).

In 2010, the proportion of sludge landfilled reached approximately 15%. Utilization as building materials (such as cement, block, or tile) and agricultural land usage (as fertilizer) corresponded to 55% and 10% respectively. These disposal methods led to an increase in the proportion of sewage sludge that was recycled to approximately 80%. However, a smaller proportion of sewage sludge has been used as fuel

compared with other well-known disposal methods, indicating that sewage sludge has not been used as an energy resource in Japan in recent decades. After 2010, the sewage sludge recycling ratio decreased dramatically from approx. 80% to 55%. This was due to the contamination of sewage sludge with cesium discharged from the Fukushima Daiichi nuclear power plant disaster in 2011; as a result, landfill and onsite stock replaced other recycling methods.

Sewage sludge contains ample amounts of organic matter; it is thus expected that a higher proportion could be utilized as an energy resource. Figure 2-9 shows the energy usage of organic matter in sewage sludge in Japan in 2012. Most organic matter (approx. 85%) was not used as an energy resource, and only 16% was used. Assuming a higher heating value of dry sewage sludge of 18 MJ/kg and dry sludge production of 2.2 million tons/year, approximately 12,000 GWh of energy can be extracted from sewage sludge in Japan.

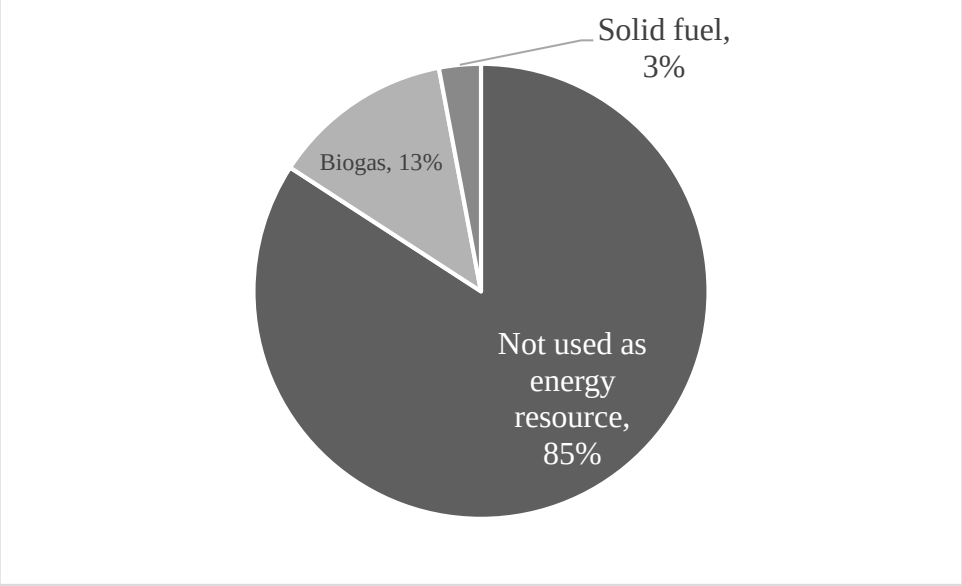


Figure 2-9: Energy usage proportion of organic matter in sewage sludge in Japan (MLIT, 2017).

2.2 Sewage sludge characteristics

Raw sewage sludge is a combination of primary and secondary sludge. The physical and chemical features of sludge must be known to treat and dispose of it in a safe and effective manner; however, sewage sludge characteristics depend on the source of the wastewater and the method used for wastewater treatment. Table 2-3 provides general information obtained from the literature on the physical and chemical characteristics of untreated and digested sewage sludge (Burton et al., 2003). These data indicate that sewage sludge contains a number of chemicals (such as organic compounds, nitrogen, and phosphorus) that are required for plant growth; thus, sewage sludge can be used as a conditioner or fertilizer for soil.

Proximate and ultimate analyses are important for studying sewage sludge. Proximate analysis covers parameters such as moisture content (M), volatile matter (V), fixed carbon (F), and ash content (A). Ultimate analysis provides percentages of substances such as carbon (C), hydrogen (H), nitrogen (N), sulfur (S), and oxygen (O).

Table 2-4 shows proximate and ultimate analysis results from the literatures for sewage sludge from Spain, Turkey, Thailand, the Netherlands, Germany, Denmark, the UK, and Switzerland (Thipkhunthod *et al.*, 2005; Inguanzo *et al.*, 2002; Dogru *et al.*, 2002; Otero *et al.*, 2002; Menendez *et al.*, 2002; Folgueras *et al.*, 2003; ECN, 2017).

Table 2-3: Typical composition and properties of sludge (Burton et al., 2003).

Item/sludge	Untreated primary		Digested primary		Untreated activated sludge
	Range	Typical	Range	Typical	Range
Total solids (TS) (%)	5-9	6	2-5	4	0.8-1.2
Volatile solids (% of TS)	60-80	65	30-60	40	59-88
Grease and fats (% of TS)					
Ether soluble	6-30	-	5-20	18	-
Ether extract	7-35	-	-	-	5-12
Protein (% of TS)	20-30	25	15-20	18	32-41
Nitrogen (% of TS)	1.5-4	2.5	1.6-4.0	2.5	2.8-11
Phosphorus (% of TS)	0.8-2.8	1.6	1.5-4.0	2.5	2.8-11
K_2O (% of TS)	0-1	0.4	0-3.0	1	0.5-0.7
Cellulose (% of TS)	8-15	10	8-15	10	-
Iron (% of TS)	2.0-4.0	2.5	3.0-8.0	4	-
Silica (SiO_2% of TS)	15-20	-	10-20	-	-
pH	5.0-8.0	6.0	6.5-7.5	7.0	6.5-8.0
Alkalinity (g/L as $CaCO_3$)	0.5-1.5	0.6	2.5-3.5	3	0.6-1.1
Organic acids (g/L as acetic acid)	0.2-2	0.5	0.1-0.6	0.2	1.1-1.7
Heating value (MJ/kg of TSS)	23-29	25	9-14	12	19-23

TSS: total suspended solids.

Table 2-4: Sewage sludge characteristics from the literatures.

No.	Proximate analysis (%wt.)				Ultimate analysis (%wt.)[a]					HHV (MJ/kg)	Country
	M	V[a]	A[a]	F[a]	C	H	N	S	O		
1	5.2	60.7	29.5	9.8	35.7	5.2	3.5	0.7	25.4	16.6	Spain
2	11.8	53.5	26.6	12.8	39.5	6.2	3.9	1.5	25.5	17.1	Turkey
3	4.3	59.3	31.0	9.7	38.1	5.2	4.5	0.9	20.3	16.8	Spain
4	3.9	58.5	30.8	10.7	38.3	5.1	3.7	0.7	21.4	16.6	Spain
5	8.5	50.8	43.3	5.9	30.1	4.1	3.8	0.9	17.8	13.3	Spain
6	78.1	60.7	36.9	2.4	37.3	5.8	5.5	0.8	13.7	16.6	Spain
7	-	55.9	40.3	3.8	29.0	4.4	3.2	0.5	22.6	12.8	Spain
8	-	49.6	44.0	6.4	25.5	3.7	2.4	0.6	23.8	12.6	Spain
9	-	71.0	21.2	7.8	40.0	6.0	7.0	0.7	25.1	18.4	Spain
10	6.1	53.0	38.4	8.6	31.1	4.2	3.3	1.1	24.3	13.9	Thailand
11	5.1	51.2	42.0	6.7	27.5	4.1	4.0	1.1	23.3	13.2	Thailand
12	5.4	50.0	43.0	7.0	26.4	4.1	4.3	0.9	23.7	12.6	Thailand
13	6.4	47.6	48.4	4.0	23.9	3.9	3.8	1.3	21.8	11	Thailand
14	3.7	42.2	51.8	6.0	20.9	3.4	3.3	0.9	21.7	10.1	Thailand
15	4.1	34.5	61.8	3.7	18.0	2.9	2.3	0.8	16.7	9.4	Thailand
16	3.4	39.0	56.0	5.0	19.5	3.2	3.1	0.8	19.4	8.7	Thailand

No.	Proximate analysis (%wt.)				Ultimate analysis (%wt.)[a]					HHV (MJ/kg)	Country
	M	V[a]	A[a]	F[a]	C	H	N	S	O		
17	3.9	33.3	63.5	3.2	14.5	2.6	2.6	1.2	18.1	6.9	Thailand
18	3.7	32.9	64.0	3.1	15.3	2.5	2.3	0.5	17.7	6.5	Thailand
19	3.2	30.6	67.6	1.8	12.7	2.0	1.8	0.6	17.5	5.7	Thailand
20	4.4	24.8	72.9	2.2	10.6	2.0	1.6	0.4	15.7	4.3	Thailand
21	8.9	23.4	74.2	2.4	9.0	2.2	1.5	1.6	18.2	3.5	Thailand
22	9.9	53.5	35.0	11.5	34.0	4.9	4.7	1.3	20.0	15.1	Netherlands
23	9.6	58.5	30.7	10.8	36.5	5.2	5.6	1.2	20.7	16.3	Netherlands
24	7.6	42.8	48.2	9.0	25.2	3.1	3.7	1.5	17.6	11.8	Netherlands
25	-	53.1	45.6	1.3	25.1	4.6	3.2	2.9	18.5	11.5	Netherlands
26	-	44.2	49.8	6.0	26.5	4.1	3.2	1.1	15.2	11.3	Germany
27	-	53.5	35.9	10.6	35.2	4.7	4.8	1.6	17.7	15.1	Netherlands
28	53.0	40.4	53.0	6.6	23.1	3.2	2.9	0.5	17.3	7.2	Netherlands
29	3.0	48.9	43.8	7.3	31.2	4.4	3.1	1.1	16.2	13.84	Netherlands
30	6.5	60.4	37.5	2.1	32.4	4.7	4.4	1.8	19.2	13.9	Netherlands
31	9.4	74.2	26.7	-0.9	40.8	5.5	6.2	1.4	19.3	18.2	Netherlands
32	20.0	48.0	45.0	7.0	27.0	4.2	5.7	1.0	17.0	13	Netherlands
33	15.0	-	37.5	-	32.8	4.5	4.4	1.7	19.0	12.7	Netherlands
34	7.6	42.8	48.2	9.0	25.2	3.1	3.7	1.5	18.2	11.8	Netherlands
35	81.6	52.2	36.4	11.4	33.7	5.2	5.4	0.7	18.5	15.33	Denmark
36	8.4	-	37.8	-	33.4	4.2	4.4	1.7	18.5	13.97	Netherlands
37	3.6	45.9	47.5	6.6	24.4	4.3	3.5	0.9	19.0	11.45	Germany
38	5.3	51.1	46.6	2.3	25.5	5.0	3.3	1.1	18.5	11.95	Netherlands
39	3.4	39.7	54.5	5.8	26.4	3.6	4.1	1.0	10.3	10.26	Netherlands
40	6.7	52.0	38.6	9.4	28.5	4.2	4.2	0.9	10.0	14.02	Netherlands
41	4.9	44.2	49.7	6.2	26.5	4.1	3.2	1.1	15.5	11.44	Netherlands
42	70.2	-	48.0	-	28.9	2.6	3.8	1.2	15.3	12	Germany
43	0.2	50.0	46.5	3.5	27.9	4.1	3.6	1.1	16.8	12.2	Switzerland
44	0.4	45.1	47.1	7.8	27.8	3.6	3.6	1.0	16.9	11.48	Germany
45	0.3	46.9	49.5	3.6	24.8	3.2	3.0	1.0	18.5	9.77	Germany
46	5.4	48.9	42.3	8.8	31.2	4.8	3.3	1.0	17.3	13.67	UK
47	5.0	-	42.0	-	-	-	6.0	-	-	15	Netherlands
48	5.0	-	52.5	-	-	-	5.8	-	-	-	Netherlands
49	78.0	-	50.0	-	26.7	3.7	3.8	1.2	15.0	11.6	Denmark
50	14.2	56.8	37.3	5.9	-	4.6	-	0.9	-	15.01	Denmark
51	15.0	-	37.5	-	32.8	4.5	4.4	1.7	18.9	12.77	Netherlands
52	5.7	54.5	39.9	5.6	31.9	4.4	4.6	1.7	17.5	14.65	Netherlands
53	5.1	46.9	48.4	4.7	29.2	3.7	2.8	0.8	15.1	12.1	Netherlands
54	77.6	-	38.4	-	31.7	4.7	4.7	1.9	18.8	12.86	Netherlands
55	72.0	-	37.7	-	29.6	4.7	1.4	1.0	25.6	16.27	Netherlands

No.	Proximate analysis (%wt.)				Ultimate analysis (%wt.)[a]					HHV (MJ/kg)	Country
	M	V[a]	A[a]	F[a]	C	H	N	S	O		
56	73.1	-	29.0	-	32.2	5.3	-	1.0	32.5	16.69	Netherlands
57	73.2	50.0	47.8	2.2	27.0	3.8	3.2	0.9	17.2	10.95	Netherlands
58	7.6	63.5	26.2	10.4	39.4	5.7	6.0	1.1	26.3	17.77	Netherlands
59	8.0	62.4	26.3	11.4	39.3	5.8	6.0	1.2	26.2	17.77	Netherlands
60	8.0	62.1	26.3	11.6	39.2	5.8	5.9	1.2	25.6	17.77	Netherlands
61	8.7	51.8	43.2	5.1	30.9	4.4	4.8	1.2	15.6	13.39	Spain
62	8.1	56.6	-	43.4	34.9	5.0	5.0	1.3	26.6	-	Netherlands
63	-	46.7	44.5	6.0	27.2	3.6	3.3	0.8	9.6	12.07	Switzerland
64	5.7	-	37.1	-	32.5	5.1	2.7	1.4	21.3	-	Netherlands

a) Dry basis.

The results indicate that the values of sewage sludge characteristics span wide ranges, such as 23.4%≤V≤74.2%, 21.2%≤A≤74.2%, −0.9%≤F≤43.4%, 9.0%≤C≤40.8%, 2.0%≤H≤6.2%, 1.4%≤N≤7.0%, 0.4%≤S≤2.9%, 9.6%≤O≤32.5%, and 3.5MJ/kg≤HHV≤18.4 MJ/kg.

Samples with a higher C percentage have a higher HHV; however, samples with a higher A percentage have a lower HHV. HHV also appears to have a strong and positive correlation with V. Figure 2-10 shows the correlation between HHV and the percentages of carbon and volatile matter [Figure 2-10 (a) and Figure 2-10 (b)].

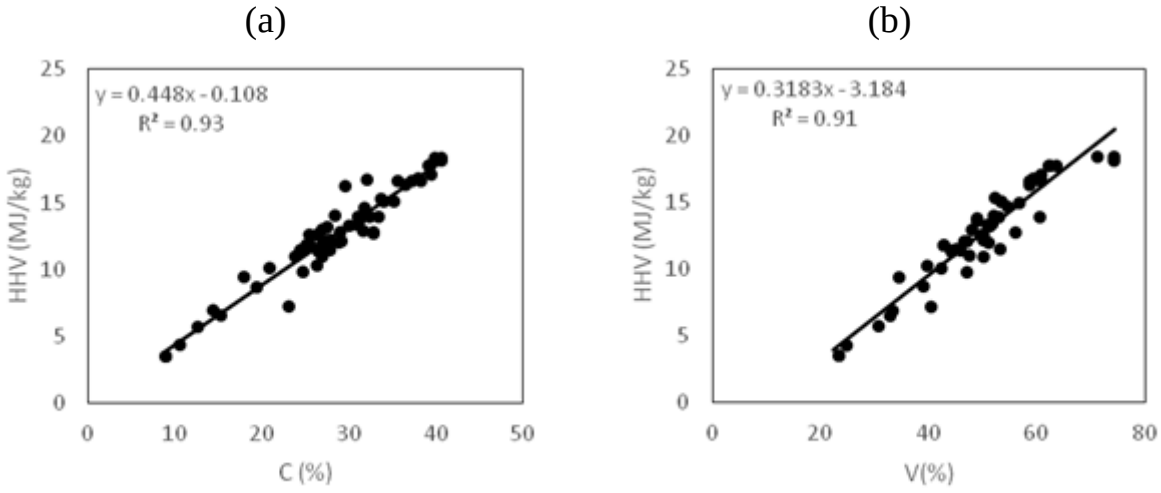


Figure 2-10: Correlation between HHV and a) C, b) V.

HHV has strong correlations with C and V, with R values of 0.96 and 0.95, respectively.

In this study, a similar survey is conducted for sewage sludge produced in Japan, including proximate and ultimate analyses and an investigation of correlations with HHV. An equation for prediction of the heating value is also investigated.

2.3 Heating value prediction

Obtaining renewable energy from waste is gaining significant attention. Energy recovery from waste such as municipal solid waste (MSW), plastics, agricultural waste, and sewage sludge can be achieved by bio-conversion, incineration, and thermochemical processes (Conesa *et al.*, 1998; Inguanzo *et al.*, 2002). The heating value of solid samples can be determined using an adiabatic bomb calorimeter; however, this device is not always available, especially in developing countries, and trained staff are necessary for accurate evaluation of HHV. A number of models have been proposed for heating value prediction that can provide estimations more quickly and simply than measurement using a calorimeter. A number of studies have discussed the relationship between HHV and the chemical composition of organic materials.

In Table 2-5 and Table 2-6, HHV prediction equations investigated by other researchers are presented based on ultimate and proximate analyses, respectively. These equations can be used for coal, lignite, MSW, biomass and sewage sludge (Channiwala *et al.*, 2002; Küçükbayrak *et al.*, 1991; Cordero *et al.*, 2001; Demirbaş, 1997; Jiménez *et al.*, 1991; Parikh *et al.*, 2005).

Table 2-5: HHV prediction equations from the literature (based on ultimate analysis).

No.	Study	Equation
1	Dulong-1880	HHV=0.3383C+1.443(H-O/8)+0.0942S
2	Strache and Lant-1924	HHV=0.3406C+1.4324H-0.1532O+0.1047S
3	Steuer-1926	HHV=0.3391(C-3/8O)+0.2386(3/8O)+1.444(H-1/16O)+0.1047S
4	Vondrecek-1927	HHV=(0.373-0.00026C)C+1.444(H-1/10O)0.1047S
5	D Huart-1930	HHV=0.3391C+1.4337H+0.0931S-0.1273O
6	Schuster-1931	HHV=$(1.0632+1.486\times10^{-3}O)[C/3+H-(O-S)/8]$
7	Grummel and Davis-1933	HHV=(0.0152H+0.9875)((C/3)+H-(O-S)/8)
8	Seyler-1938	HHV=$0.519C+1.625H+0.001O^2-17.81$
9	Gumz-1938	HHV=0.3403C+1.2432H+0.0628N+0.1909S-0.0984
10	Sümegi-1939	HHV=0.3391[C-0.75(O/2)]+1.444[H-0.125(O/2)]+0.1047S
11	Mott and Spooner	HHV=0.3361C+1.419H-0.1453O+0.0942S **for O≤15%** HHV=0.3361C+1.419H—(0.1532-0.0007O)O+0.0942S **for O>15%**
12	Boie-1953	HHV=0.3517C+1.1626H+0.1047S-0.111O
13	Dulong-Bertholt	HHV=0.3414C+1.4445H-(N+O-1)/8+0.093S
14	IGT-1978	HHV=0.341C+1.323H+0.0685-0.0153A-0.1194(O+N)
15	Tillman-1978	HHV=0.4373C-1.6701
16	Jenkins-1985	HHV=-0.763+0.301C+0.525H+0.064O
17	Grabosky and Bain-1981	HHV=0.328C+1.4306H-0.0237N+0.0929S-(1-A/100)(40.11H/C)+0.3466
18	Beckman et al.-1990	HHV=0.352C+0.944H+0.105(S-O)
19	Wilson-1972	HHV=0.352Co+1.507H-0.1384O-0.1485Ci+0.09263S+0.02419N
20	Chang-1979	HHV=35.8368+0.7523H-0.2674S-0.4654O-0.3814Cl-0.2802N
21	Khan and Abu Gharah-1991	HHV=0.0535[F+32.6CP]+0.3722PLR
22	Niessen-1995	HHV=0.2322C+0.7655H-0.072O-0.0419N+0.0698S+0.0262Cl+0.1814P

All equations give HHV in MJ/kg.
A) C, H, N, S, O, A, Cl, and P represent the percentages of carbon, hydrogen, nitrogen, sulfur, oxygen, ash, chlorine, and phosphorus, respectively.
B) Co and Ci indicate the percentages of organic and inorganic carbon, respectively.
C) F is the percentage of food.
D) CP is the percentage of cardboard and paper.
E) PLR is the percentage of plastic, rubber, and leather.
All parameters are expressed in % dry basis.

Table 2-6: HHV prediction equations from the literature (based on proximate analysis).

No.	Study	Equation
23	Küçükbayrak-1991	$HHV=076.56-1.3(V+A)+7.03\times10^{-3}(V+A)^2$
24	Cordero-2001	HHV=354.3F+170.8V
25	Demirbaş-1997	HHV=0.196F+14.119
26	Jiménez-1991	HHV=-10814.08+313.3T (T=F+V)
27	Parikh-2005	HHV=0.3536F+0.1559V-0.0078A

The Dulong equation (Eq. 1) assumes complete combustion and that all oxygen in a solid sample exists as H_2O; Eq. 2 is a modified version of the Dulong equation. These equations are applicable for coal or MSW. The Steuer equation (Eq. 3) assumes that oxygen in samples exists in equal amounts of H_2O and C-O, while Schuster (Eq. 6) assumes that all oxygen is present as C-O and Sümegi (Eq. 10) that oxygen presents as COOH. Moot and Spooner (Eq. 11) assume that two-thirds of oxygen is associated with hydrogen and the other third with carbon. It can be claimed that Eq. 14 is the most comprehensive HHV prediction equation for coal, as it is based on more than 700 data points. In 1978, Tillman demonstrated that HHV and carbon content have a strong correlation in biomass samples (Eq. 15). Jenkins investigated data from 57 biomass samples and presented Eq. 16 by multiple regression analysis, and Eq. 17 was suggested assuming reactions of C, H, S, and N to CO_2, H_2O, SO_2, and NO_2 for biomass. Eq. 18 was derived for biomass-derived oil. Eq. 19, 20, and 21 were presented for HHV prediction of waste (including MSW) and, for one of those, there is dependence on the type of carbon (organic or inorganic). Finally, Eq. 22 was suggested for HHV calculation of water sludge and Eq. 23-27 are derived based on proximate analysis of samples, for example, lignite and biomass.

The HHV for sewage sludge has a strong correlation with proximate and ultimate analysis (especially C) data. Furthermore, it has been demonstrated in this section

that the HHV of organic materials can be predicted by ultimate or proximate analysis. Thus, it should be possible to derive an equation to predict the HHV of sewage sludge from Japan. In this study, in addition to proximate and ultimate analyses of sewage sludge from Japan, correlations with those parameters are illustrated, and HHV prediction equation is developed.

2.4 Volatile matter and heating value analysis by TG-DTA

The chemical composition and HHV of sewage sludge are important parameters. It is also important to know the volatile matter content; to evaluate this, samples must be dried in an oven at 105-110 °C and then ignited at 600 ±25 °C for 1 hour. The weight losses indicate the moisture content and the volatile matter content (Japan Sewage Works Association, 2012). Although this method seems simple, it is a lengthy process, including a cooling period after heating; in addition, samples of approximately 1.0 g after drying are needed, which may require large initial samples for specimens with a large water content. TG-DTA measures the mass change and the temperature difference between the sample and a reference material simultaneously as a function of time and temperature, while changing or maintaining the temperature of the sample according to a certain program (Hatakeyama, 2006). The technique is widely used for the thermal analysis of polymer materials, organic materials, metals, ceramics, and fuels. Some researchers reported that volatile matter analysis results for coal samples (anthracite, bituminous coal, brown coal) (sample amount: 10–25 mg) can be determined by TG, which are compatible with volatile matter analysis results obtained by the American Society for Testing and Materials (ASTM) and the UK standard method (Rosenvold *et al.*, 1982; Cumming & McLaughlin, 1982). Gamel and Smothers showed that the exothermic peak area of the DTA curve and the calorific value of the sample are proportional to each other for three types of subbituminous coal (Gamel & Smothers, 1952). Muñoz-Guillena

et al. reported that the exothermic peak area of the DTA curve of eight types of Spanish coal (sample weight: about 3.0 mg) and the heating values of the samples were highly consistent (Muñoz-Guillena *et al.*, 1992). These studies indicate that it is possible to evaluate volatile matter and HHV of coal by TG-DTA with small sample sizes to a high level of accuracy.

Studies related to the use of TG-DTA for sewage sludge analysis are limited. The heat generation starting temperature for sludge fuel (carbonized or dried sludge) has been investigated, and Urabe analyzed the combustion reaction rate and combustion behavior of dry sludge. Other studies investigated combustion gas generation and characterization by combining TG-DTA and mass spectrometry (MS) (Urabe, 1979; Magdziarz & Wilk, 2013; Magdziarz & Werle, 2014).

2.5 Particle size distribution

Wastewater consists of numerous organic substances that affect the wastewater treatment process. Primary sludge (PS) and waste activated sludge (WAS) are produced in the process of removing these organic substances. PS is generated mainly by physical treatment processes such as primary sedimentation, and WAS is generated by biological processes. In a typical WWTP, more than 60% of the organic matter in wastewater is concentrated in sludge containing PS and WAS. Thus, 60% of the energy content of the wastewater is concentrated in the sludge and can be used for energy recovery (Garrido *et al.*, 2013). The heating value of sludge is a key factor controlling energy recovery, which is normally achieved via methane fermentation or using dried sludge as fuel. The heating value of PS is generally higher than that of WAS (Tyagi & Lo, 2013). The range of heating values of PS is 20–28 MJ/kg and that of WAS is 16–22 MJ/kg (Burton *et al.*, 2003).

PS is an aggregate of suspended solids (SS) removed by the primary sedimentation tank (PST). The particle size distribution (PSD) of SS in influent is a key parameter for determining the characteristics of PS. Several studies have investigated the PSD of wastewater and its characteristics and reported the effects of primary and secondary treatments on the PSD of wastewater. These studies show that particle sizes in treated wastewater are generally smaller after the primary treatment process (Levine *et al.*, 1991; García-*Mesa et al.*, 2010). However, the heating value of each particle size fraction in wastewater has not been investigated. It is important to evaluate the role of each wastewater particle size on heating value distribution, and estimate the amount of energy that can be extracted from each particle size in the wastewater collected as PS.

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Chapter 3

National Survey of Dewatered Sewage Sludge Composition in Japan: Trend of Organic Elements and Prediction of Higher Heating Value

3.1 Introduction

Japan depends on other countries for many resources including energy, and it is thus important that this is secured. It is necessary to increase the proportion of energy generated using renewables to reduce greenhouse gas (GHG) emissions due to a number of factors, including the Fukushima Dai-ichi nuclear power plant accident and the Paris Agreement. In addition, internationally, a special committee (ISO/TC275) related to "sludge recovery, recycling, treatment and disposal" was established in 2013, and a draft of international standards for related technologies is in development. The scope of this standard is the "standardization of sorting, classification, pretreatment, processing, recycling and management methods for sludge or biosolids and output from water treatment facilities"; from the perspective of characterization, current data on sewage sludge composition is required for Japan (Suzuki, et al., 2014).

Energy utilization is an effective disposal method for the organic matter contained in sludge. The elemental composition and the heating value are important parameters for sludge samples. The higher heating value (HHV) of sewage sludge can be obtained using a bomb calorimeter; however, because incineration has gained wider attention since the 1960s, there has been an ongoing demand for simple prediction methods for the HHV. Initially, the ignition loss was suggested as an explanatory parameter for HHV prediction (Honda, et al., 1963; Sano, 1965; Kondo et al, , 1965;

Omiya, 1968; Hiraoka et al., 1973). In 1983, the Ministry of Construction of Japan and the Japan Sewage Works Agency conducted a nationwide survey of dewatered sewage sludge. The chemical composition and HHV of samples was studied and the following equation proposed for HHV prediction, which is still used today (Ministry of Construction, Japan and Japan Sewage Works Agency, 1984, 1988; Ministry of Land, Infrastructure, Transport and Tourism (MLIT), Japan, 2003; National Institute for Land and Infrastructure Management, MLIT, Japan, 2015; Tasaki, 2011):

$$HHV = 4.186 \times (58.3V - 193) \quad 3\text{-}1$$

where the HHV is in kJ/kg-DS and V is the ignition loss (%DS).

Additional datas were collected in 1987, and a new equation presented, but with only a small change in coefficients and intercepts. Over 30 years have passed since these surveys, and there have been significant changes in environmental conditions. Furthermore, due to the changing properties of sewage (in response to changing lifestyles) and increasingly sophisticated wastewater treatment processes, dewatering and coagulants may have produced changes in dewatered sewage sludge characteristics.

In this study, a nationwide survey was conducted on the properties of dewatered sludge, and these were compared to past findings to evaluate changes in characteristics and key factors. A new prediction method for the HHV is proposed.

Specifically, various dewatered cakes were collected in different seasons, dewatering methods, sewage collection systems (combined or separated), coagulants (organic/inorganic), and sludge digestion. Samples were collected throughout the country and analyzed for moisture content; volatile matter, carbon (C), hydrogen

(H), nitrogen (N), and sulfur (S) content; and other major element constituents. The HHV was also measured. First, the effect of each factor on sewage sludge characteristics was established. Changes in factors since the previous study conducted in 1983 were then analyzed (Ministry of Construction, Japan and Japan Sewage Works Agency, 1984), for ignition loss, organic element composition, and HHV. Finally, a new prediction equation for HHV was developed using volatile matter and other parameters.

3.2 Material and methods

3.2.1 Samples

Wastewater treatment plants (WWTPs) for sampling were chosen based on statistical information (Japan Sewage Works Association, 2013). Several sampling parameters were considered, so that samples were fully representative of dewatered sewage sludge in Japan: sewage collection method (combined or separated), sludge type (digested or not digested), coagulants (polymer only, polymer+polyferric sulfate or polyaluminium chloride (PAC)), and dewatering method (centrifuge, belt press, screw press, multi disk or rotary press). These combinations cover WWTPs in Japan exhaustively, both in the ranges of these parameters and geographically. As shown in Table 3-1, 32 types of dewatered sludge were collected and sampling was conducted in each season. The sampling periods were: summer: 9–18 September 2015; fall: 30 October to 1 December 2015; winter: 29 February to 11 March 2016; and spring: 9–29 May 2016. However, due to site maintenance during some of these periods, not all 32 types of sludge were sampled in every period. A total of 120 samples was collected. Table 3-1 details the samples collected for this study and those from the 1983 survey. The 1983 survey consisted of 33 samples mainly collected from centrifugal and belt press dewatered sludge, the most common

dewatering methods at that time. This survey covers a wider range of sample and takes seasons and coagulants into consideration.

3.2.2 Analysis parameters and methods

After sampling, the dewatered sludge was placed in a refrigerator in the dark and maintained at 4°C or less; analysis was conducted as soon as possible after collection. Proximate analysis of samples, specifically volatile matter and ash content, was conducted according to the Japanese Wastewater Examination Method (Japan Sewage Works Associoation, 2012). Samples were dried at 105 ± 5°C overnight then ignited in a muffle at 600°C for 1 hour (h). The differences in the weights of the samples after heating indicate the moisture and ash contents, respectively.

Ultimate analysis was conducted using a CHN analyzer (JM-10, J-Science Lab Co. Ltd., Kyoto, Japan) and an X-ray fluorescence spectometer (XRF-1800, Shimadzu Co. Ltd., Kyoto, Japan). The CHN analyzer measures the percentage of C, H, and N on a dry basis. We assumed that these elements are included in volatile matter. XRF was used to measure the percentages of other elements, such as total sulfur (T-S), Al, Ca, Fe, K, Mg, P, and Si (on a dry basis). The HHV value was measured using a bomb calorimeter (CA-4AJ, Shimadzu Co. Ltd.).

Volatile matter consists of C, H, N, volatile S, and O; however, the CHN analyzer cannot measure the O percentage accurately; hence, Eq.3-2 was used to calculate the O percentage:

$$O = V\text{-}C\text{-}H\text{-}N\text{-}S_c \qquad 3\text{-}2$$

where Sc indicates combustible S, which was assumed to be 0.66 of total S, from the proportion of combustible and total sulfur observed in the results of the 1983 survey (Ministry of Construction, Japan and Japan Sewage Works Agency, 1984).

Table 3-1: Sample details for this study and the 1983 survey.

Dewatering	Sewage collection system	Sludge type	Previous study	This study				
				Sampling season				Sample number
			Sample number	Sum.	Aut.	Win.	Spr.	
Centrifuge	Combined	Undigested	2	1	1	1	1	1
		Digested	7	2	2	2	2	2(1)
	Separated	Undigested	4	2	2	2	2	2(1)
		Digested	2	2	1	1	1	2(1)
Screw press	Combined	Undigested	-	3	3	2	2	3(2)
	Separated	Undigested	1	2	2	2	2	2(1)
		Digested	-	2	2	2	2	2(1)
Multi disk	Combined	Digested	-	1	1	1	1	1
	Separated	Undigested	-	3	3	3	3	3(2)
		Digested	-	1	1	1	1	1
Belt press	Combined	Undigested	3	3	4	3	3	4(2)
		Digested	4	1	1	1	1	1
	Separated	Undigested	6	1	1	1	1	1
		Digested	4	2	2	2	2	1
Rotary press	Combined	Undigested	-	2	2	2	2	2(1)
	Separated	Undigested	-	2	2	2	2	2(1)
		Digested	-	1	1	1	1	1
Total			33	31	31	29	29	32(14)

(). polymer+polyferric sulfate or PAC.

The lower heating value (LHV) of dry and dewatered samples was calculated from the measured HHV values using Eqs. 3-3 and 3-4:

$$LHV_d = HHV - 4.186 \times 600 \times 9 \times H \quad (3\text{-}3)$$

$$LHV_W = (100-w)/100 \times LHV_d - 4.186 \times 600 \times (w/100) \quad (3\text{-}4)$$

where LHV_d and LHV_W represent the LHV of the dry and dewatered samples, respectively, and w is the moisture content of the dewatered sample.

3.3 Results and discussion

3.3.1 Sewage sludge characteristics

Table 3-2 shows the sewage sludge characteristics. The moisture content of the dewatered cake samples ranged from 72.1–85.7% (average value: 79.8%), the volatile matter from 64.6–88.7%DS (average value: 80.0%DS), the HHV from 14.7–20.1 MJ/kg-DS (average value: 18.1 MJ/kg-DS), the LHV of the wet samples ranged from 0.9–16.4% of the HHV (average value: 2.2% of HHV), and the C content was in the range 32.2–44.1%DS (average value: 39.8%DS) The Fe and Al concentrations in samples those two polymers were used for conditioning were generally higher, but not significantly above the average values of 1.29% for Fe and 2.15% for Al, and fell within the range of variation of Fe and Al concentrations in the sludge before conditioning. Eriksson (2001) conducted a survey and elemental analysis of 48 dewatered sewage sludge cake samples from WWTPs in Sweden, and found mean moisture content of 79% and volatile matter, Al, Ca, Fe, and Si values of 62, 4.0, 2.8, 4.9, and 4.5%DS, respectively (Eriksson, 2001). A lower volatile matter content and generally higher Al, Ca, Fe and Si concentrations were found in this study; this may be due to a higher proportion of combined sewage systems and different coagulants used in biological sewage treatment in Sweden compared with Japan.

Next, a preliminary study was conducted to evaluate the standard composition of dewatered sludge, as the properties of the dewatered cake are influenced by factors such as season, digestion, and the type of dewatering method. The degree of influence of these factors was investigated, and the standard sludge composition classified by factors with a higher influence rather than simply by the total average values found for the samples.

The influence of season, sewage collection system, use of digestion, conditioning material, and dewatering method on sludge characteristics was calculated using mathematical quantification theory class 1. Mathematical quantification theory class 1 is a method corresponding to multiple regression analysis when the objective variable is a quantitative variable and the explanatory variables are qualitative variables. In this study, the variables were as follows: season (four categories), sewage collection system (two categories), use of digestion (two categories), coagulant (two categories), and dewatering method (five categories). The objective variable was the volatile matter content of the 120 samples analyzed in this survey. JMP® (ver 12.0, SAS Institute Inc., Cary, NC, USA) and College Analysis (Programmed by Fukui, 2017) statistical analysis software were used for mathematical quantification theory class 1.

Table 3-2: Sewage sludge characteristics.

No.	Dewatering	Sewage	Digestion	Coagulants	M (%)	V(%-DS)	Heating value（MJ/kg)			Ultimate analysis of organic matter (% of DS)					Ultimate analysis of other element (% of DS)							
							HHV of DS	LHV of DS	LHV of wet	C	H	N	S	O	Total S	Al	Ca	Fe	K	Mg	P	Si
1	Centrifuge	Com.	Undigested	Polymer	79.4	82.2	18.10	16.70	1.40	40.7	6.5	3.6	0.6	30.7	0.9	0.8	1.2	2.3	0.3	0.4	1.8	2.6
2	Centrifuge	Com.	Digested	Polymer only	80.5	64.6	14.70	13.50	0.60	32.2	5.6	4.9	1.4	20.5	2.1	1.5	2.0	5.4	0.4	1.2	4.0	3.2
3	Centrifuge	Com.	Digested	Polymer+Poly Fe	80.4	65.2	15.10	13.80	0.70	32.2	5.5	4.6	1.2	21.6	1.8	2.0	2.0	4.8	0.4	0.7	3.9	3.7
4	Centrifuge	Sep.	Undigested	Polymer only	84.0	87.6	20.00	18.40	0.80	44.0	7.1	8.2	0.9	27.4	1.3	1.0	1.3	1.0	0.4	1.1	3.1	1.3
5	Centrifuge	Sep.	Undigested	Polymer+Poly Fe	80.1	84.4	19.60	18.10	1.60	42.9	6.9	6.8	0.7	27.1	1.0	0.7	0.8	1.7	0.4	0.5	2.8	1.3
6	Centrifuge	Sep.	Digested	Polymer only	85.5	74.3	17.50	16.00	0.20	38.0	6.3	5.5	1.2	23.3	1.8	1.9	2.3	1.2	0.2	0.4	3.2	2.8
7	Centrifuge	Sep.	Digested	Polymer+Poly Fe	79.2	73.8	16.30	14.90	1.10	35.4	6.1	6.2	0.9	25.2	1.4	1.2	2.0	2.1	0.3	1.2	3.7	1.8
8	Screw press	Com.	Undigested	Polymer only	79.6	78.6	17.70	16.30	1.30	39.5	6.2	4.7	0.6	27.5	1.0	2.2	1.1	1.6	0.4	0.7	2.8	2.9
9	Screw press	Com.	Undigested	Polymer+PAC	77.6	81.6	18.30	16.90	1.80	40.9	6.5	5.4	0.8	28.1	1.1	1.4	0.8	1.9	0.4	0.4	2.3	2.4
10	Screw press	Com.	Undigested	Polymer+Poly Fe	76.2	85.8	18.90	17.30	2.20	42.9	6.9	4.8	0.7	30.4	1.1	1.2	0.9	1.5	0.3	0.4	2.0	1.9
11	Screw press	Sep.	Undigested	Polymer only	78.4	88.7	19.70	18.00	1.90	44.1	7.1	5.5	0.5	31.4	0.8	0.8	1.1	0.6	0.3	0.5	2.2	1.4
12	Screw press	Sep.	Undigested	Polymer+Poly Fe	81.8	84.1	18.70	17.20	1.10	41.7	6.7	5.5	0.9	29.2	1.3	1.1	1.1	1.4	0.4	0.5	2.4	1.4
13	Screw press	Sep.	Digested	Polymer only	85.7	76.2	17.70	16.20	0.20	38.3	6.5	5.9	1.1	24.3	1.7	1.7	1.9	1.9	0.3	0.5	3.0	2.3
14	Screw press	Sep.	Digested	Polymer+Poly Fe	82.5	74.1	17.00	15.60	0.70	36.7	6.3	6.0	1.1	24.0	1.7	1.0	2.0	3.6	0.3	1.2	4.1	1.8
15	Multi desk	Com.	Digested	Polymer only	78.4	76.8	17.90	16.50	1.60	38.4	6.4	6.2	1.3	24.5	2.0	1.2	1.6	2.7	0.3	0.9	2.7	2.8
16	Multi desk	Sep.	Undigested	Polymer only	83.4	79.1	18.20	16.70	0.70	39.2	6.8	7.1	0.6	25.3	0.9	3.4	1.1	0.4	0.4	0.4	2.9	2.5
17	Multi desk	Sep.	Undigested	Polymer+Poly Fe	85.2	87.0	20.00	18.30	0.60	43.2	7.2	8.4	1.0	27.3	1.4	0.3	0.5	2.4	0.6	0.4	2.4	0.6
18	Multi desk	Sep.	Undigested	Polymer+Poly Fe	81.9	82.5	18.20	16.70	1.00	40.1	6.7	7.1	0.8	27.8	1.2	1.6	0.7	1.8	0.4	0.3	2.6	2.3
19	Multi desk	Sep.	Digested	Polymer only	79.2	68.0	15.20	13.90	0.90	33.4	5.7	5.0	0.9	23.0	1.3	1.7	1.3	5.5	0.3	0.6	3.6	3.1
20	Belt press	Com.	Undigested	Polymer only	77.5	86.8	19.80	18.20	2.10	43.7	6.9	5.7	0.6	29.9	1.0	0.6	0.8	1.3	0.3	0.5	2.4	1.0
21	Belt press	Com.	Undigested	Polymer only	79.0	87.4	19.10	17.50	1.70	42.9	6.9	4.9	0.5	32.1	0.8	0.8	1.2	0.5	0.5	0.5	1.9	1.8
22	Belt press	Com.	Undigested	Polymer+PAC	79.3	79.3	18.10	16.60	1.40	39.7	6.4	5.3	0.8	27.2	1.2	1.4	1.3	2.9	0.4	0.3	2.4	2.4
23	Belt press	Com.	Undigested	Polymer+Poly Fe	77.5	81.3	18.30	16.80	1.80	40.6	6.6	5.6	0.8	27.7	1.2	0.9	0.8	2.5	0.4	0.4	2.7	2.1
24	Belt press	Com.	Digested	Polymer only	84.3	73.3	17.20	15.80	0.30	37.2	6.1	6.0	1.1	23.0	1.6	1.7	2.1	2.1	0.4	1.0	3.3	3.5
25	Belt press	Sep.	Undigested	Polymer only	75.9	87.6	19.40	17.80	2.40	43.5	7.0	5.1	0.5	31.5	0.8	1.1	1.0	0.4	0.3	0.5	2.1	1.4

No.	Dewatering	Sewage	Digestion	Coagulants	M (%)	V(%-DS)	Heating value（MJ/kg)			Ultimate analysis of organic matter (% of DS)					Ultimate analysis of other element (% of DS)							
							HHV of DS	LHV of DS	LHV of wet	C	H	N	S	O	Total S	Al	Ca	Fe	K	Mg	P	Si
26	Belt press	Sep.	Digested	Polymer only	83.2	81.5	19.10	17.60	0.90	41.5	6.7	7.3	0.8	25.1	1.2	1.1	1.8	0.7	0.4	0.9	3.1	2.3
27	Belt press	Sep.	Digested	Polymer+Poly Fe	77.2	71.2	15.40	14.10	1.30	33.2	5.8	5.6	1.8	24.7	2.7	0.9	0.9	5.8	0.2	0.9	3.9	1.6
28	Rotary press	Com.	Undigested	Polymer only	73.2	78.8	17.80	16.40	2.50	40.0	6.4	4.3	0.6	27.5	0.9	1.1	1.7	1.5	0.4	0.6	1.9	3.4
29	Rotary press	Com.	Undigested	Polymer+Poly Fe	76.2	78.0	17.90	16.50	2.00	38.6	6.2	4.3	0.6	28.2	0.9	1.0	0.9	3.6	0.4	0.5	2.5	2.8
30	Rotary press	Sep.	Undigested	Polymer only	72.1	88.1	19.40	17.90	3.20	44.0	7.0	4.3	0.5	32.4	0.8	0.6	1.0	0.4	0.2	0.5	1.7	1.7
31	Rotary press	Sep.	Undigested	Polymer+Poly Fe	77.2	85.5	18.70	17.20	2.00	41.7	6.8	4.9	0.6	31.5	0.9	0.6	0.6	3.0	0.3	0.3	2.3	1.2
32	Rotary press	Sep.	Digested	Polymer only	82.5	85.0	20.10	18.40	1.20	43.4	7.1	6.6	0.9	27.0	1.3	0.9	1.9	0.5	0.3	0.5	2.2	1.9
Average					79.8	79.9	18100	16600	1300	39.8	6.5	5.7	0.9	27.1	1.3	1.2	1.3	2.2	0.4	0.6	2.8	2.2

Com.: combined. Sep.: separated.

The analysis yielded a multiple correlation coefficient of 0.813, a contribution rate of 0.629, and a contribution rate adjusted in terms of the degree of freedom of 0.629, indicating that the volatile matter content can be represented by the explanatory variables with relatively high correlation. Table 3-3 shows the category score (partial regression coefficient in the multiple regression analysis), the range of the explanatory variable (the difference between the minimum and maximum category score), and the partial correlation coefficients for each variable. An explanatory variable was judged to have a large influence if it had a low p-value, a wide category score range, and a high partial correlation coefficient. Use of digestion and sewage collection methods were found to have the greatest influence, with digestion having a strong negative influence on volatile matter, separated sewage collection systems having a positive influence, and combined sewage collection systems having a negative influence. In addition, the results were significant ($p < 0.01$) for coagulants, while the use of a single flocculent (polymer only) had a positive influence and the use of double flocculants (polymer+polyferric sulfate or PAC) had a negative influence. However, the range was small and the influence was limited. No significant effects were observed for season and dewatering method. Similarly, the results of the 1983 survey were analyzed by mathematical quantification theory class 1 using volatile matter as the objective variable. For the 1983 survey, the explanatory variables were sewage collection system (two categories), use of digestion (two categories) and dewatering method (two categories). A multiple correlation coefficient of 0.772, a contribution rate of 0.595, and a degree of freedom-adjusted contribution ratio of 0.552 was calculated, indicating that the volatile matter content was expressed by the explanatory variables with relatively high correlation.

Table 3-3: Mathematical quantification theory class 1 results for volatile matter in sludge.

Objective parameter	Item	Category	Category score	Standard error	t value	P value	Range	Correlation coefficient
This study	Season	Summer	-1.91	0.68	-2.79	0.0061	3.32	0.283
		Autumn	0.57	0.68	0.83	0.41		
		Winter	1.41	0.70	2.01	0.047		
		Spring	-0.07	0.70	-0.10	0.92		
	Sewage	Combined	-2.54	0.42	-6.04	<0.0001	5.09	0.511
		Separated	2.54	0.42	6.04	<0.0001		
	Digestion	Undigested	5.30	0.43	12.2	<0.0001	10.6	0.768
		Digested	-5.30	0.43	-12.2	<0.0001		
	Coagulant	Polymer only	1.13	0.41	2.75	0.0069	2.26	0.257
		Polymer+Other	-1.13	0.41	-2.75	0.0069		
	Dewatering method	Centrifuge	-1.64	0.80	-2.04	0.044	3.90	0.346
		Screw press	0.80	0.78	1.02	0.31		
		Multi desk	-2.12	0.88	-2.43	0.017		
		Belt press	1.78	0.76	2.33	0.022		
		Rotary press	1.19	0.87	1.36	0.18		
Previous survey	Sewage	Combined	-6.88	1.51	-4.55	<0.0001	13.8	0.653
		Separated	6.88	1.51	4.55	<0.0001		
	Digestion	Undigested	3.11	1.50	2.07	0.05	6.21	0.366
		Digested	-3.11	1.50	-2.07	0.05		
	Dewatering method	Centrifuge	2.24	1.45	1.54	0.13	4.48	0.286
		Belt press	-2.24	1.45	-1.54	0.13		

For the 1983 results, volatile matter was most influenced by the sewage collection system followed by the use of digestion, based on the p-values, the range of the category score, and the number of partial correlation coefficients. Dewatering method had no influence on volatile matter. Based on these findings, information on dewatered sludge composition was divided into four main categories: sewage collection method (combined or separated) and sludge type (digested or undigested); this information is provided in Table 3-4. The moisture content of undigested sludge tended to be lower than that of digested sludge; sewage sludge originating from combined sewage collection systems also tended to have a lower moisture content

than that derived from separated collection systems. On the other hand, undigested sludge from separate sewage collection systems had the highest volatile matter content, and the lowest volatile matter values were found for digested sludge from combined sewage systems. The same trends were observed for HHV (which is strongly correlated with volatile matter), C, H, and O (which are the main constituents of volatile matter). Digested sludge tended to have higher concentrations of S and other elements, as the overall solid content of sludge decreases with organic matter digestion, concentrating these elements as suspended solids. However, the concentration of K did not change significantly across the four categories. The reason for this is unclear; however, K is presumed to exist as a soluble component and may be transferred to the liquid phase during the dewatering process. Thus, it is not affected by digestion.

Table 3-4 a: Sewage sludge characteristics of combined/separated and digested/undigested samples (Part 1).

Sewage	Digestion	Value	M (%)	V (%)	Heating value (kJ/kg)			Ultimate analysis of organic matter (% of DS)				
					HHV of DS	LHV of DS	LHV of wet	C	H	N	S	O
Combined	Undigested	Average	77.6	81.6	18.3	16.8	1.82	40.7	6.5	4.8	0.7	28.9
		Standard deviation	3.15	4.47	0.97	0.91	0.56	2.1	0.4	0.8	0.1	2.4
Separated	Undigested	Average	80.0	85.5	19.2	17.6	1.52	42.4	6.9	6.3	0.7	29.1
		Standard deviation	4.13	3.36	0.81	0.76	0.85	1.9	0.3	1.4	0.2	2.6
Combined	Digested	Average	80.9	70.0	16.2	14.9	0.81	35.0	5.9	5.4	1.2	22.4
		Standard deviation	2.92	6.61	1.65	1.55	0.61	3.5	0.6	0.8	0.1	2.1
Separated	Digested	Average	81.5	75.6	17.3	15.8	0.853	37.4	6.3	6.1	1.1	24.7
		Standard deviation	3.10	5.68	1.72	1.62	0.44	3.7	0.6	0.7	0.3	2.1

Table 3-4 b: Sewage sludge characteristics of combined/separated and digested/undigested samples (Part 2).

Sewage	Digestion	Value	Ultimate analysis of other elements (% of DS)							
			Total S	Al	Ca	Fe	K	Mg	P	Si
Combined	Undigested	Average	1.01	1.15	1.12	2.02	0.382	0.484	2.26	2.44
		Standard deviation	0.214	0.608	0.329	1.07	0.0891	0.248	0.571	0.912
Separated	Undigested	Average	1.05	1.13	0.92	1.32	0.384	0.488	2.45	1.50
		Standard deviation	0.260	0.868	0.280	1.010	0.102	0.297	0.570	0.683
Combined	Digested	Average	1.88	1.61	1.95	3.73	0.362	0.939	3.48	3.32
		Standard deviation	0.225	0.405	0.266	1.53	0.0537	0.201	0.605	0.755
Separated	Digested	Average	1.65	1.24	1.72	2.79	0.295	0.831	3.37	2.14
		Standard deviation	0.485	0.418	0.515	2.13	0.0852	0.424	0.787	0.647

3.3.2 Comparison with previous study and changes in composition

In this section, the results obtained in this study are compared with those from the 1983 study. The mean values of measured parameters for both studies are shown in Figure 3-1. The amount of volatile material increased by 14%DS ($t(38) = -6.30$, $p < 0.01$) in this study compared with that of the 1983 study, whereas the concentrations of C, H, and Sc increased by 4.3%DS ($t(39) = -3.84$, $p < 0.01$), 2.0%DS ($t(36) = -9.91$, $p < 0.01$), and 0.20%DS ($t(151) = -3.35$, $p < 0.01$), respectively. In addition, the concentration of O also increased significantly by 7.0%DS ($t(40) = -7.32$, $p < 0.01$), and, as a result, the HHV increased significantly by 2.75 MJ/Kg-DS ($t(37) = -4.97$, $p < 0.01$). Figure 3-2 shows sewage sludge characteristics categorized by sewage collection system type and sludge type for both studies. A significant increase in parameters such as volatile matter, C, H, Sc, HHV, and O was observed in this study (particularly for samples collected from combined sewage collection system WWTPs). This is likely due to the separation of

rainwater and improvements in combined sewage systems (MLIT, 2008), which result in an increased organic matter content. On the other hand, the HHV per volatile matter decreased by 0.52 MJ/kg VTS ($t(38) = 2.20$, $p < 0.05$).

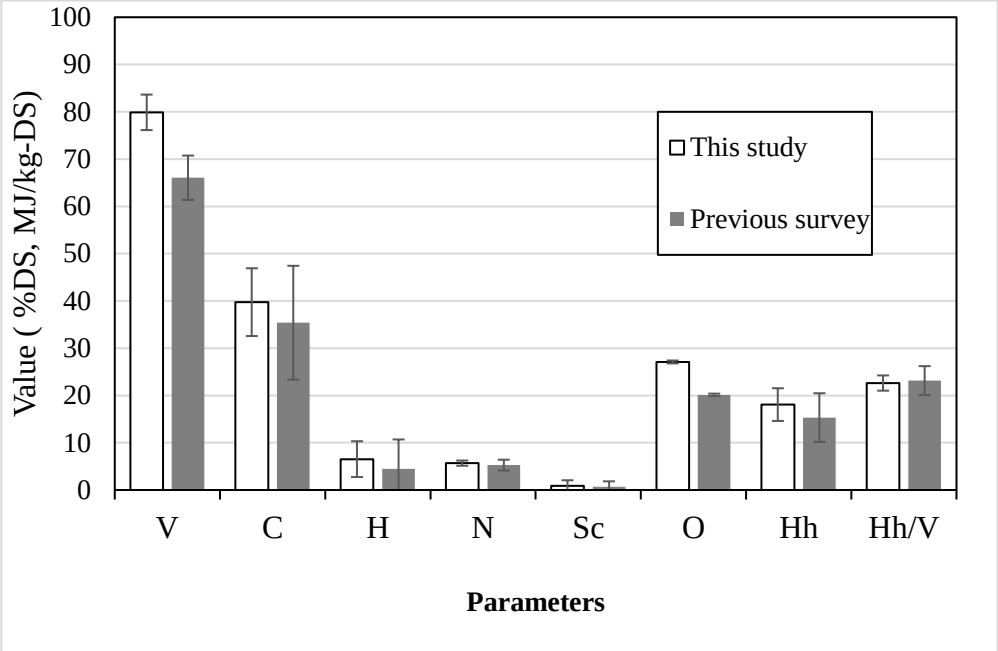

Figure 3-1: Sewage sludge characteristics measured in both studies (this study n = 120; previous survey: n = 33).

This indicates that the quality of organic matter in sludge for fuel has decreased, due to the increase in O content (which contributes to the reduction in HHV) and the increase in other parameters such as C, H and Sc (these contribute to an increase in HHV). The results for the four categories from both studies are plotted in a Van Krevelen diagram using C, H, and O values in Figure 3-3. In this diagram, the horizontal axis represents the O/C atomic ratio, while the vertical axis represents the H/C ratio (van Krevelen, 1950). This technique has been used recently for Fourier-transform ion cyclotron resonance/mass spectrometry (FTICR/MS) data for fossil fuel evaluation and interpretation of matter distributions in environmental samples (Wu *et al.*, 2004; Sleighter & Hatcher, 2008). In addition to the results of both

surveys, plots for a range of biomass and coal samples, with representative data obtained from the literature, are also shown

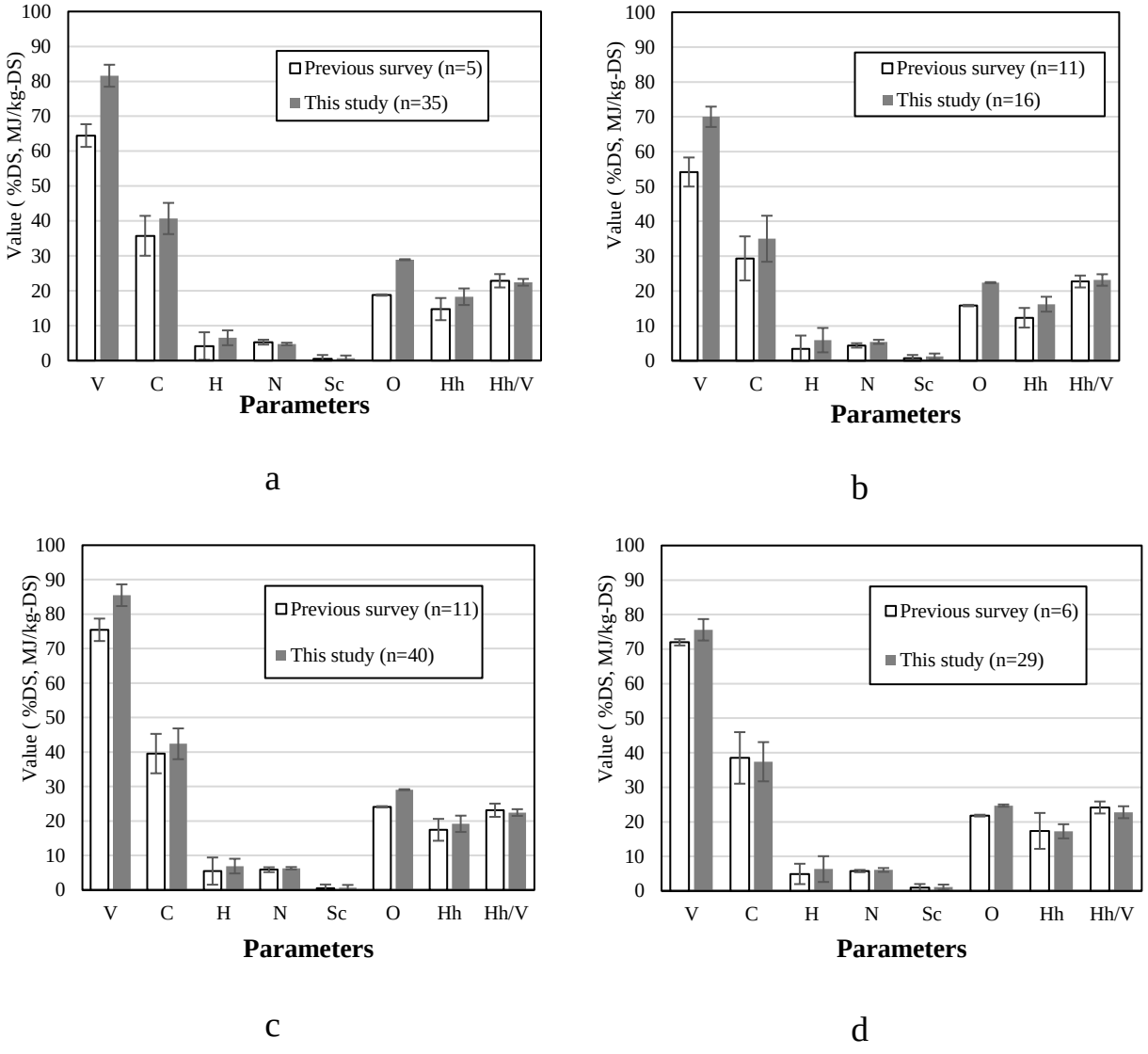

Figure 3-2: Sewage sludge characteristics based on origin and digestion (a. combined/undigested, b. combined/digested, c. separated/undigested and d. separated/digested).

for reference (Vassilev *et al.*, 2010). The blue line indicates dehydration, the red line shows decarbonation, and the green line is demethanation (in the direction of the arrow). The results of the 1983 study were distributed across a range of O/C ratios from 0.28–0.56 and H/C ratios from 1.00–1.90, indicating a similar composition to

peat and livestock manure. No influence from sewage collection method or sludge digestion was observed. In contrast, the results of this study show a shift in the O/C ratio to a range of 0.40–0.67 and in the H/C ratio of 1.80–2.25; these values are similar to those associated with refuse-derived fuel (RDF).

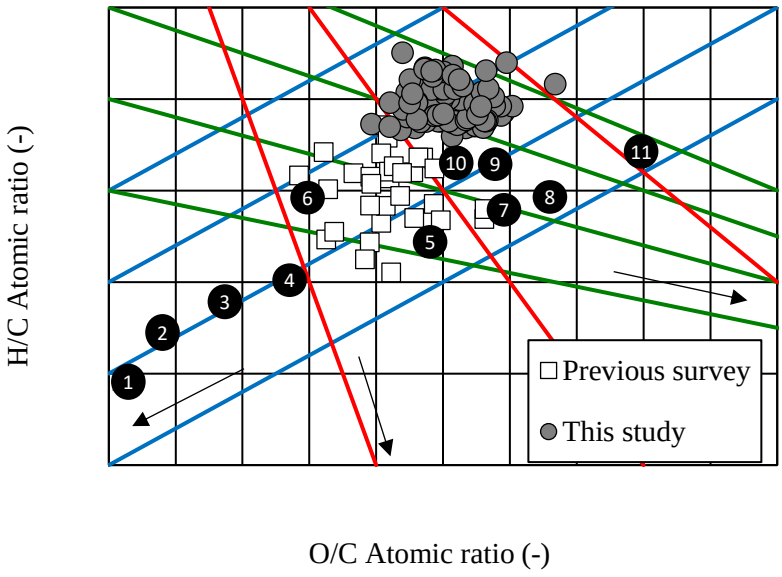

Figure 3-3: Van Krevelen diagram of sewage sludge from both studies and other reference materials: 1.Anthracite, 2.Bituminous coal, 3.Subbituminous coal, 4.Lignite, 5.Peat, 6.Manure, 7.Woody biomass, 8.Leaf and straws, 9.Paper, 10.RDF, 11.Algae (Sleighter and Hatcher, 2008).

Comparing the operation lines between the two studies, the plots shifted along the positive direction of the blue line (hydrolysis) and the negative direction of the green line (carbonation) between 1983 and the current study. The shift toward hydrolysis/carbonation is attributable to advances in biological treatment processes in WWTPs in recent years results in oxidation of organic matter. In 1983, the mean aeration time in WWTPs was 9.8 h with a median of 7.6 h, while in 2013 these values were 12.8 h and 10.3 h, respectively (Japan Sewage Works Association, 1985, 2013); the increase in these parameters was statistically significant ($t(1032) = 7.69$, $p < 0.01$). This change is due to the adoption of the oxidation ditch process in

activated sludge treatment result in changes in dewatered sewage sludge characteristics.

3.3.3 Higher heating value prediction

3.3.3.1 Prediction from volatile matter

Figure 3-4 shows the relationship between volatile matter and HHV for both studies. The trendlines indicate a strong correlation between these two parameters, with R values of 0.960 and 0.936 for the previous study and the current study, respectively. No difference in trendline was observed when the data for different sewage collection systems and the use of sludge digestion were separated. Equation 3-5 can be used to predict the HHV of sewage sludge from volatile matter.

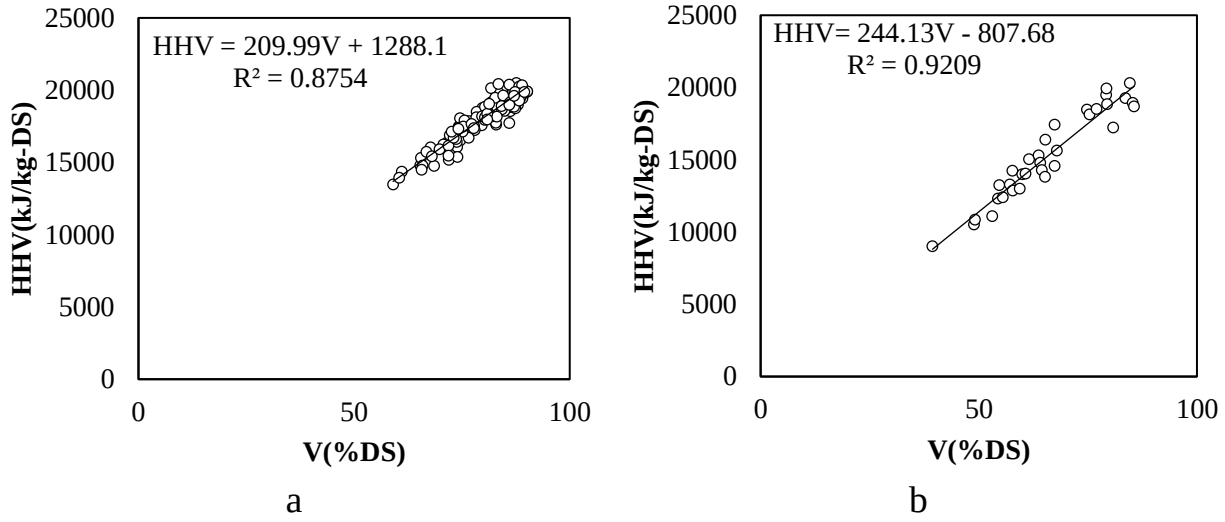

Figure 3-4: Correlation between volatile matter and HHV (a. this study and b. previous study).

$$Hh = 4.186 \times (50.2V + 308) \quad 3\text{-}5$$

Compared to Eq. 3-1, the equation developed has a slope value that is smaller by about 15% and an intercept value that is higher by about 2,100 kJ/kg-DS. The slope

value can be considered as HHV/V in the trendline. This result is in agreement with the discussion in the previous section suggesting that HHV/V has decreased significantly since 1983. Both equations were developed using linear regression analysis. However, before Eq. 3-5 can be suggested as a new HHV prediction equation, it is necessary to prove that there is a significant statistical difference between Eqs. 3-1 and 3-5. Analysis of covariance (ANCOVA) was conducted to confirm this.

First, a regression model of Eq. 3-6 was prepared using both the 1983 data and the data from this study, to test the parallelism of the two straight lines. In this model, a dummy variable, and an interaction term between volatile matter and the dummy variable are introduced. The 1983 data and the data from this study can be represented by Eqs. 3-7 and 3-8, respectively. Statistical analysis software JMP® (ver. 12.0, SAS Institute Inc.) was used for the regression analysis.

$$Hh = \alpha 1 + \alpha 2V + \alpha 3D + \alpha 4VD \quad (3\text{-}6)$$

$$Hh = (\alpha 1 + \alpha 3D) + (\alpha 2 + \alpha 4D)\ V \quad (3\text{-}7)$$

$$Hh = \alpha 1 + \alpha 2V \quad (3\text{-}8)$$

Here, α1–α4 are partial regression coefficients, and D is a dummy variable that was set to 1 for the previous study and 0 for this study.

The results of the regression analysis provided a multiple correlation coefficient of 0.960, a contribution rate of 0.923, and a contribution rate in terms of the degree of freedom of 0.921. Hence, it is possible to predict the HHV of sewage sludge with very high correlation. The details of each parameter are shown in Table 3-5. α1 and α2 represent the intercept and slope in Eq. 3-5, respectively. Similarly, (α1 + α3D) and (α2 + α4D) are the intercept and slope in Eq.3-1, respectively. The p-value of

α4 was less than 0.01, and the partial regression coefficient of α4 is, hence, non-negligible. Thus, it can be concluded that the slopes of Eqs. 3-1 and 3-5 are statistically different and that the two equations are statistically different.

Table 3-5: Regression analysis results of both studies.

	Partial regression coefficient	Standard error	T value	P value
α1	1290	662	1.94	0.0537
α2	210	8.26	25.4	<0.001
α3	-2100	919	-2.28	0.0239
α4	34.1	12.6	2.72	0.0074

The error of the HHV prediction equation is given by:

$$X = |Xobs - Xabs| / Xabs \quad 3\text{-}9$$

where X is the percentage error (%), Xobs is the predicted HHV, and Xabs is the HHV measured for the samples using a bomb calorimeter.

The averages and standard deviations of the errors for each equation are shown in Table 3-6, when the values of V from each of the studies were applied. From the table, when Eq. 3-1 is applied to the results of the 1983 survey, the average relative error is 4.49% with a standard deviation of 2.92%. However, when applying the value of V obtained in this study, the average relative error is 3.72% with a standard deviation of 2.68%. When the value of V from this study is applied to Eq.3-5, the average value and standard deviation of the relative error decrease to 2.47% and1.94%, respectively. Applying the value of V obtained from samples from the

previous study to Eq. 3-5 results in an increase in the error and the standard deviation of the error.

It can therefore be concluded that Eq. 3-5 developed in this study can predict the HHV of dry sludge accurately. Thus, this equation is proposed as an updated version of Eq. 3-1.

Table 3-6: Average and standard deviation for sample data and equations.

Date of sample	Prediction equation	Average error (%)	Standard deviation
1983	1983	4.49	2.92
	This study	5.27	3.31
This study	1983	3.72	2.68
	This study	2.47	1.94

However, when Eq. 3-5 is applied to sewage sludges from different countries introduced in the previous chapter, the average of the relative error is approximately 14%, much higher than when it is applied to sewage sludge from Japan. This difference arises due to differences in sewage sludge characteristics. For example, the average values of V and C for the samples discussed in the previous chapter were 49 and 28%, respectively, much lower than those measured for Japanese sewage sludge in this study. Equation 3-5 is therefore not suitable for HHV prediction for sewage sludge from outside of Japan.

3.3.3.2 Prediction by chemical composition

In this section, regression analysis of organic constituent elements, specifically C, H, N, Sc, and O, is used to develop a prediction expression for HHV. Specifically, a brute force method was used with these elements considered as explanatory

variables. The number of explanatory variables was set from 1 to 5 and HHV was considered as the object variable. Statistical analysis software JMP® (ver 12.0, SAS Institute Inc.) was used for regression analysis.

Here, the equations with the highest correlation coefficient for different numbers of explanatory variables are discussed and (Eqs. 3-10 to 3-14), and details of the correlation coefficients for these formulas are provided in Table 3-7.

$$Hh = 411C + 1751 \quad (3\text{-}10)$$
$$Hh = 396C + 172N + 1340 \quad (3\text{-}11)$$
$$Hh = 384C + 101H + 165N + 1210 \quad (3\text{-}12)$$
$$Hh = 392C + 104H + 152N + 89Sc + 828 \quad (3\text{-}13)$$
$$Hh = 389C + 101H + 157N + 90Sc + 4.36O + 810 \quad (3\text{-}14)$$

Table 3-7: Correlation analysis details.

Equation No.	Number of explanatory variables	Correlation coefficient (R)	Average error (%)	STDV (%)
3-10	1	0.966	1.79	1.49
3-11	2	0.974	1.43	1.49
3-12	3	0.974	1.41	1.50
3-13	4	0.974	1.40	1.51
3-14	5	0.975	1.44	1.49

Equation 3-10, which has C as the only explanatory variable, had the highest correlation coefficient R of 0.966. The average and standard deviation of the relative error were 1.79% and 1.49%, respectively. The accuracy of this equation was higher than that of Eq. 3-5; based on volatile matter. A previous study also found the highest correlation coefficient with C when one variable was used, and reported a correlation

coefficient R of 0.96, almost the same accuracy as in Equation 3-1 based on volatile matter (Ministry of Construction, Japan and Japan Sewage Works Agency, 1984). Solid combustion-type TOC meters that can also measure total C have become relatively popular in recent years; Eq. 3-10 is also a simple and accurate prediction formula. Furthermore, other cases where between two and five variables were used produced almost constant correlation coefficients, relative errors, and standard deviations of about 0.974, 1.41%, and 1.50%, respectively. This indicates that no greater accuracy is achieved with the use of two or three explanatory variables. Equation 3-11 is a prediction equation with C and N, and Eq. 3-12 is a prediction equation with C, H, and N as explanatory variables. If a CN or CHN automatic analyzer is available, Eqs. 3-11 and 3-12 are more effective as prediction expressions with higher accuracy.

It was also observed that LHV has very strong correlation with HHV. Figure 3-5 shows that LHV can be predicted by LHV=0.93HHV-255, R^2=0.998. The average of absolute error and standard deviation of mentioned equation is 0.4 and 0.28% respectively.

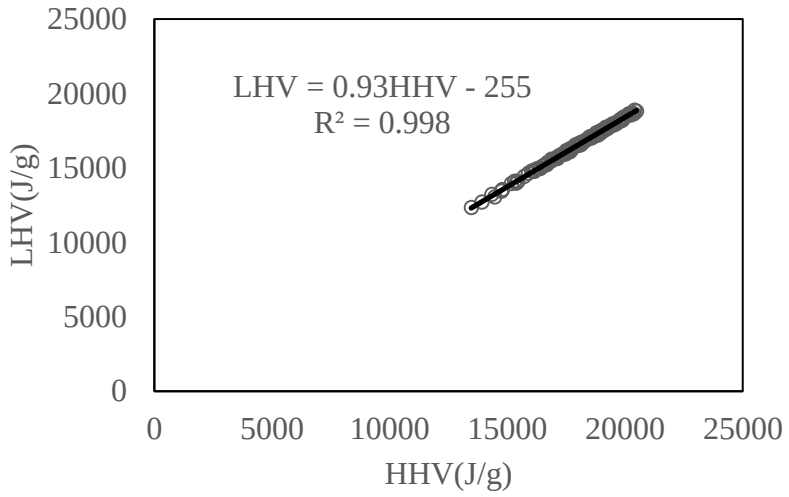

Figure 3-5: Correlation between LHV and HHV.

3.4 Conclusions and future trends

In this study, we investigated the characteristics of 32 kinds of dewatered sewage sludge nationwide and compared the results to those from a previous study conducted in 1983 to evaluate changes and key factors. A new prediction equation with explanatory variables such as volatile matter is proposed. The main conclusions and future works are:

1- Sewage collection system (combined/separated) and sludge type (digested/undigested) are the parameters that have the greatest effect on sewage sludge characteristics, based on results obtained from mathematical quantification theory class 1. Standard characteristics of four categorized sewage sludges were also discussed.
2- The volatile matter content, HHV, and organic matter content of sewage sludge in Japan has increased in the past 30 years. The quality of organic matter (HHV/V) has decreased due to increases in oxygen concentration resulting from increasing hydrolysis and carbonization. It is speculated that this has resulted from improvements in sewage collection systems (from separated to combined) and an increase in average aeration times (during biological processing in WWTPs).
3- An updated equation: HHV ＝ 4.186×(50.2V＋308) is proposed for the HHV prediction for sewage sludge. This equation is statistically different from that presented 30 years ago. This equation can predict the HHV of sewage sludge within an average relative error of 2.47%.
4- Slight improvement in the accuracy of ultimate analysis-based prediction was found for two or more variables. The average relative error can be reduced to within 1.41% when C, H, and N are used as variables.

5- The volatile matter content of sewage sludge can be measured simply without the use of any special chemicals using a muffle furnace. Therefore, the HHV prediction equation can be used effectively when a bomb calorimeter is not available, such as in developing countries. Similar attempts have been made in Thailand and elsewhere. In future work, it is suggested to conduct domestic and an international comparison of sewage sludge characteristics and a worldwide HHV prediction equation for sewage sludge developed. Differences in analytical methods between countries should also be considered.

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Chapter4

Prediction of the Ignition Loss and Heating Value of Sewage Sludge by Thermogravimetry and Differential Thermal Analysis

4.1 Introduction

Utilization of the energy contained in organic matter in sewage sludge is an important issue, and the organic matter (volatile matter; V) content of sludge and its heating value are important factors. The V content of sewage sludge is obtained from the difference between the weight of the residue heated and dried at 105 to 110°C for 2 h and the weight of the residue when ignited at 600 ± 25°C for 1 h. Although this method is simple, it requires a long time, including that needed for cooling after heating. The higher heating value (HHV) of sewage sludge can be determined by bomb calorimetry, but it is necessary to obtain a dry weight sample of about 1 g. A large amount of wastewater or sludge samples with a low solid concentration is required. Thermogravimetry and differential thermal analysis (TG-DTA) can measure the mass change and temperature difference between the measurement sample and the reference material simultaneously as a function of time and temperature, while changing and holding the temperature of the sample according to a predefined program (Hatakeyama, 2006). The V-value obtained by TG of coal samples has been compared with that determined using standard methods; the results suggested that TG can be used to evaluate the V content of organic materials (Rosenvold *et al.*, 1982; Cumming *et al.*, 1982). The DTA exothermic peak area can reportedly be used to determine the heating value of bituminous coal and coal samples (Gamel *et al.*, 1952; Muñoz-Guillena *et al.*, 1992).

The time required for V and heating value analyses can be considerably diminished by TG-DTA of very small quantities of samples. This would facilitate control of auxiliary fuel in sewage sludge incineration facilities.

As mentioned in Chapter 2, the objective of this research was to develop a method for estimating the V content and heating value of sewage sludge by TG-DTA. Specifically, we analyzed the basic properties of 29 types of dewatered sewage sludge cakes collected throughout Japan, and generated a TG-DTA curve. Next, we assessed the agreement between the V content obtained from the TG curve with that measured by the Wastewater Examination Method (Japan Sewage Works Association, 2012). The lower heating value (LHV) is the net heating value obtained by ignition of a unit mass without energy recovery from moisture evaporation. The LHV calculation was introduced in a previous chapter (Eq. 3-3). However, CHN analysis and bomb calorimetry are necessary for determining the LHV, and require trained staff and considerable time. Furthermore, regarding the mechanist of TG-DTA that doesn't recover evaporation energy, the DTA peak area reflects the LHV. The relationship between the exothermic peak area of the DTA curve and the origin and properties of the dewatered cake was examined, and a method for determining the LHV of sewage sludge from the exothermic peak area was studied. Finally, the agreement between the LHVs estimated from the DTA curve and by bomb calorimetry was evaluated, and the validity of the developed prediction method verified.

4.2 Materials and Methods

4.2.1 Sewage sludge samples

This study focused on dewatered sewage sludge. The samples used were as described in a previous chapter, and differed according to the sewage-collection method (combined or separated), sludge type (digested or indigested), coagulants (polymer only or polymer + polyferric sulfate or polyaluminum chloride [PAC]), and dewatering method (i.e., centrifuge, belt press, screw press, multi disk, or rotary press). Moreover, the wastewater treatment plants (WWTPs) were geographically dispersed. Twenty-nine types of dewatered sludge were collected (Table 3-1). Samples obtained in spring (period A) and winter (period B) were analyzed.

4.2.2 Sewage sludge characteristics and methods

The V percentage; percentages of carbon (C), hydrogen (H), nitrogen (N), sulfur (S or S_c), and oxygen (O); and the HHV and LHV of the samples were determined according to previous chapter method.

TG-DTA of dry dewatered sludge was performed using a differential thermal balance (Thermo Plus EVO 2, TG 8120; Rigaku). 10 mg –DS of the sample was heated from ambient temperature to 650°C at 10°C/min under 50 mL/min air flow (Air-zero A; Sumitomoseika). Thus, the analysis time was approximately 65 min. Measurements were carried out three times for samples gathered during period A, and once for those obtained in period B.

The DTA value may differ according to device condition and sample type. Therefore, referring to the method of Muñoz-Guillena *et al*., TG-DTA was conducted for metals with different melting points; *i.e.*, In, Pb, Sn, and Al under the

same conditions as the sludge sample. Energy calibration was conducted according to the ratio of the endothermic peak area for melting and the theoretical value of melting heat absorption for each metal, to convert the DTA value (μV) to the heat value (mW).

Using the obtained TG curve, the rate of weight decrease from room temperature to the temperature at which the weight reduction rate become almost zero was taken as the predicted V-value. In addition, the DTA curve area was calculated by integrating the exothermic peak area of sewage sludge obtained during heating. The integration boundaries were determined from the weight reduction start/end temperature and the peak shape of the DTA curve. The DTA curve of dried sludge has two peaks: near 300°C, and at 400– 500°C (Urabe, 1979). The areas of these peaks were named A_1 and A_2 (kJ). Figure 4-1 shows conceptual diagrams of TG-DTA of sewage sludge. Because generated steam is discharged to the outside of the TG-DTA system by air flow, the exothermic peak area reflects the LHV. The LHV of sewage sludge obtained by the exothermic peak area is calculated by Eq. 4-1. To compare the LHV of a sample with that obtained from the DTA peak area, the calibration coefficient (K) of samples obtained during period A was calculated by Eq. 4-2.

$$Hls_{DTA} = (A_1 + A_2) \div M \quad 4\text{-}1$$

$$K = Hls_{DTA} \div Hls \quad 4\text{-}2$$

where Hls_{DTA} is the LHV obtained from the peak area (kJ / kg − DS), M is the weight of the sample (mg), Hls is the LHV of the sample (kJ / kg − DS), and K is the calibration coefficient.

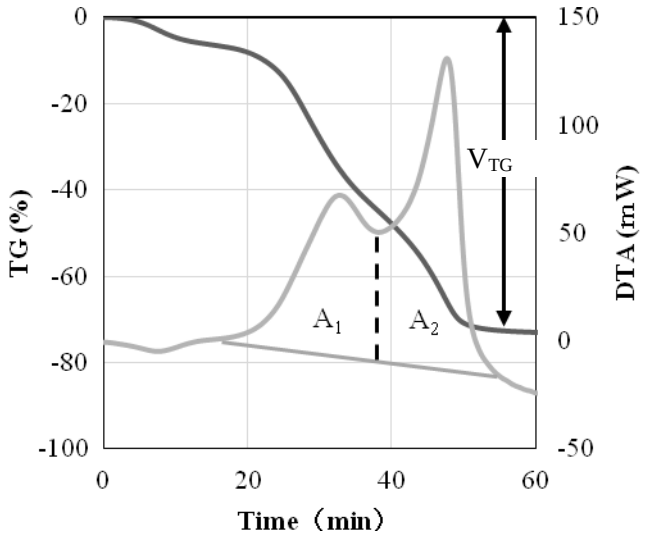


Figure 4-1: Thermogravimetry and differential thermal analysis (TG-DTA) curve of sewage sludge.

4.3 Results and Discussion

4.3.1 Volatile matter analysis by TG curve

Figure 4-2 shows the TG curve of sewage sludge collected in period A. The experiment was conducted in triplicate for each sample and the average TG values are shown. For all 29 samples, weight decreased with increasing temperature. In most cases, a primary weight loss occurred at 200–350°C and a secondary weight loss at 350–550°C. Because air was used, ignition was expected as the temperature increased. Easily and hardly combustible organic matter ignited during the primary and secondary weight loss, respectively. The TG curve was flat at around 600°C; therefore, this area reflects the ash content (A) of the sample.

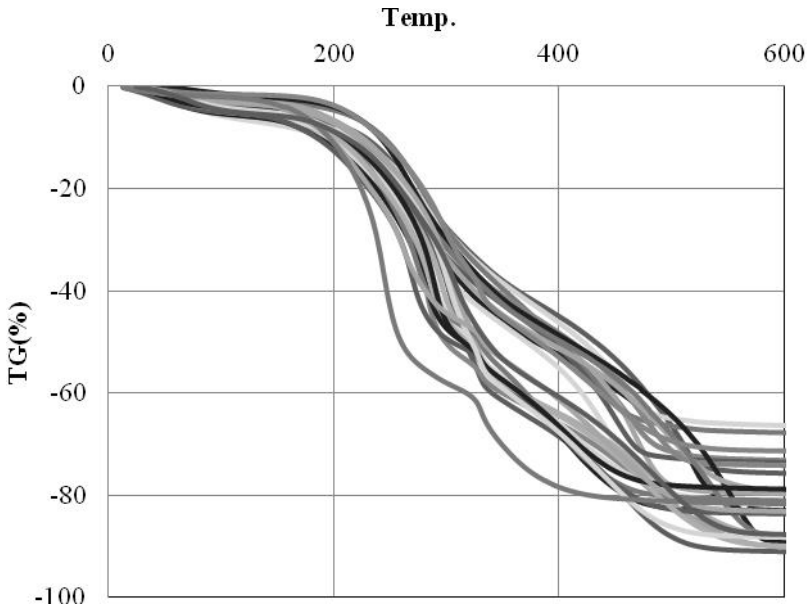


Figure 4-2: TG curve of sample A.

Figure 4-3 shows the V-values obtained by the Japan sewage testing method and that from the TG curve. The relationship between the V and V_{TG} (volatile matter measured by TG) values was approximately 1:1. Thus, TG can be used to assess the V content of sewage sludge. The relative error Ev (%) of the correspondence between the V and V_{TG} values was calculated as follows:

$$Ev = (|V_{TG} - V|/V) \times 100 \qquad 4\text{-}3$$

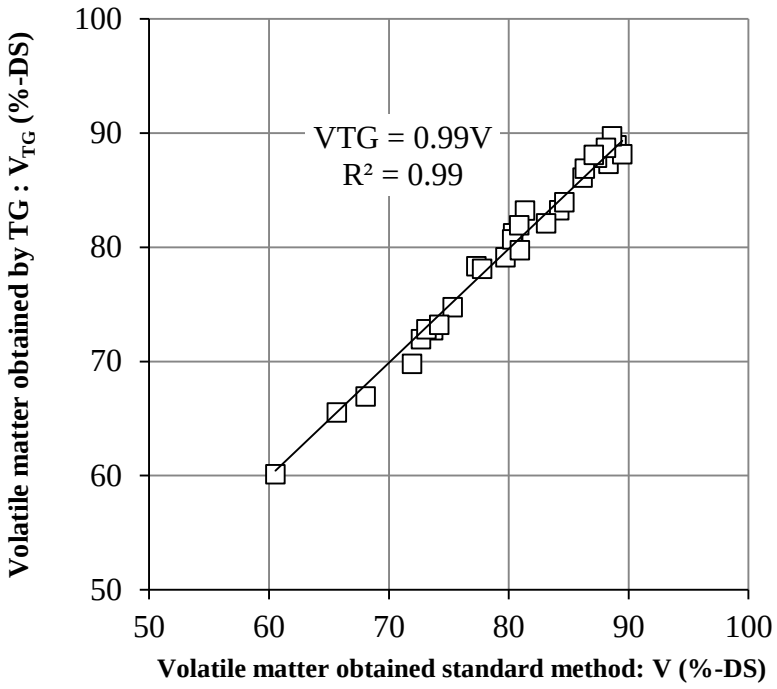


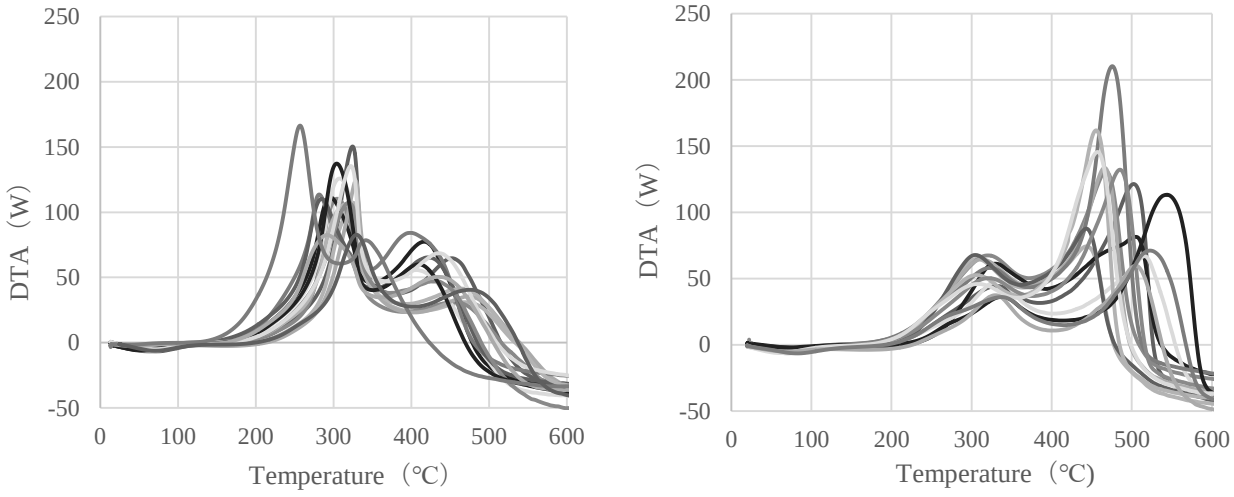
Figure 4-3: Correlation between volatile matter (V) values obtained by two methods.

Average of error and standard deviation of E for samples were 1.02% and 0.62%, respectively; thus, TG is capable of predicting the V content of sewage sludge.

4.3.2 DTA curve characteristics

As reported previously (Urabe, 1979), two peaks were observed as the temperature increased (Figure 4-4). Figure 4-4 a shows a DTA curve in which the area of the first peak is larger than that of the second peak ($A_1/A_2 > 1$), and vice versa in Figure 4-4 b ($A_1/A_2 < 1$). These peaks represent ignition of the samples because they lie within the 200–350°C and 350–550°C areas. Moreover, weight loss occurs in these regions. The DTA curve of lubricating oil also contains two peaks. The first peak represents simultaneous oxidation and pyrolysis of organic matter, and the second peak is oxidation and thermal decomposition of polymers and carbon residues (Yukawa,

1985). Similar phenomena likely occur during heating of sewage sludge. Next, we investigated the effect of various factors on the peak shapes of DTA curves.



a b

Figure 4-4: DTA curve of sewage sludge of season A for peak area ratio (a. $A_1/A_2>1$ [n=15] and b. $A_1/A_2<1$ [n = 14]).

4.3.2.1 Effects of factors related to sewage sludge origin

The effects of factors related to the origin of sewage sludge (sewage-collection method, sludge type, coagulants, and dewatering method) on the shape of the DTA curve were evaluated by mathematical quantification theory class 1 using JMP® statistical analysis software (ver. 12.0; SAS Institute Inc.).

The factors were season (four categories), sewage-collection system (two categories), digested or undigested (two categories), flocculation conditions (two categories), and dewatering method (five categories). The objective variable was the peak area ratio, A1/A2, as indicator of peak shape. Sludge type and dewatering method ($p < 0.01$) had the greatest influence on the shape of the DTA curve; however, dewatering method did not exert a significant effect ($p > 0.05$) (Table 4-1).

Fourteen of the fifteen samples in Figure 4-4 a were from undigested sludge and ten of the fourteen samples in Figure 4-4 b were from digested sludge. Undigested and digested sludge had positive and negative effects on A_1/A_2, respectively.

Table 4-1: Result of mathematical quantification theory class 1 of A_1/A_2.

Item	Category	Category score	STDV	t-value	P-value	Range
Sewage	Combined	−0.14	0.12	−1.2	0.23	0.28
	Separated	0.14	0.12	1.2	0.23	
Digestion	Undigested	0.40	0.12	3.40	0.0013	0.8
	Digested	−0.40	0.12	−3.4	0.0013	
Coagulant	Polymer only	0.17	0.12	1.4	0.16	0.34
	Polymer+other	−0.17	0.12	−1.4	0.16	
Dewatering method	Centrifuge	0.12	0.23	0.51	0.61	0.76
	Screw press	0.12	0.24	0.51	0.61	
	Multi desk	−0.51	0.26	−1.99	0.054	
	Belt press	0.28	0.22	1.27	0.21	
	Rotary press	−0.01	0.25	−0.05	0.96	

The shape of the DTA curve of dried sludge was significantly affected by digestion, as reported previously (Calvo et al., 2004; Otero et al., 2002). Organic matter corresponding to the lower temperature side peak is anaerobically digested by the methane fermentation results in existence of hardly digestible organic matters contributes in higher temperature peak for digested sludge.

4.3.2.2 Effects of factors related to sewage sludge characteristics

Next, correlation between the quantitative parameters (e.g., V, C, H, N, HHV, and LHV) and the A1/A2 value was evaluated (Table 4-2). Weakly positive correlations between C, H, and heating value with A_1/A_2 were observed. In contrast, N showed a negative correlation. The ammonia in sludge is likely volatilized during the drying process, and so the nitrogen content of sludge is likely mainly derived from protein, thermally denatured by the primary combustion, and then oxidized and decomposed by the secondary combustion.

Table 4-2: Regression analysis of A_1/A_2 with other parameters.

Parameter	R
V (%-DS)	0.39
C (%-DS)	0.38
H (%-DS)	0.14
N (%-DS)	−0.68
HHV(kJ/kg-DS)	0.30
LHV (kJ/kg-DS)	0.31

4.3.3 Prediction of LHV

4.3.3.1 Relationship between peak area and LHV

Table 4-3 shows the V_{TG}, LHVs derived from peak areas, peak area ratios (A_1/A_2), A_1+A_2, and K-values of samples obtained during period A. The K-values varied from 0.61 to 0.94, and all were less than 1.0. Thus, the LHV obtained from the DTA peak area was lower than that determined by the standard method. The K-value was

strongly negatively correlated with the V content (V_{TG}) (Figure 4-5). The linear regression equation (correlation coefficient [R] = 0.871) is as follows:

$$K = -0.0101V_{TG} + 1.482 \quad (4\text{-}4)$$

The underestimation of the heating value obtained from the peak area increases with increasing V content. The V content and the heating value are typically strongly positively correlated (Ministry of Construction, Japan and Japan Sewage Works Agency, 1984). Thus, the heat loss increases as the heating value of the sample increases. It can be because of the presence of low-temperature-volatizing materials.

4.3.3.2 Prediction of LHV from the peak area

The LHV was estimated using the peak area of the DTA curve. Specifically, heat flow derived from the DTA curve was calculated by Eq. 4-1. The LHV was estimated by Eq. 4-5, which includes Eq. 4-4.

Table 4-3: Estimation of LHV using the DTA peak area.

No.	V_{TG} (%DS)	A_1 (J/g)	A_2(J/g)	A_1/ A_2	DTA Peak Area (J/g)	LHV(J/g)	K
1	71.9	5200	7400	0.71	12600	15700	0.8
2	79.7	6300	6500	0.97	12700	16900	0.75
3	86.1	6000	4300	1.39	10400	17600	0.59
4	81.9	6200	5700	1.08	11900	16500	0.72
5	69.8	5000	6700	0.75	11800	14100	0.84
6	83.2	4600	6900	0.68	11500	17500	0.66
7	89.7	6100	4800	1.27	10800	17600	0.61
8	60.1	5400	6000	0.91	11400	12700	0.89
9	65.5	5300	7000	0.75	12300	13100	0.94
10	82.1	6900	5700	1.21	12600	16600	0.75
11	72.7	4100	7900	0.51	12000	15200	0.79
12	83.9	6100	5900	1.04	12000	18000	0.67
13	87.3	4100	8200	0.5	12300	18600	0.66
14	79.1	4600	8600	0.53	13200	16700	0.79
15	66.9	5500	6900	0.79	12400	14100	0.88
16	87.8	4500	8800	0.5	13300	18200	0.73
17	78.4	4400	6000	0.73	10400	16100	0.65
18	81.2	4200	8700	0.48	12900	16600	0.78
19	73.2	6100	5400	1.13	11500	15900	0.72
20	78.1	6000	5200	1.16	11200	15900	0.7
21	88.1	6100	5600	1.08	11700	18000	0.65
22	86.9	6300	5800	1.09	12200	17400	0.69
23	88.2	6000	4300	1.4	10300	18200	0.57
24	80.7	6200	5200	1.21	11400	16500	0.69
25	88.9	5700	5300	1.09	11000	18700	0.59
26	72.8	4700	7900	0.6	12600	15200	0.83
27	74.7	4800	8100	0.59	12900	15900	0.81
28	83.3	6100	6200	0.98	12400	17300	0.71
29	88.7	6400	5000	1.3	11400	18100	0.63

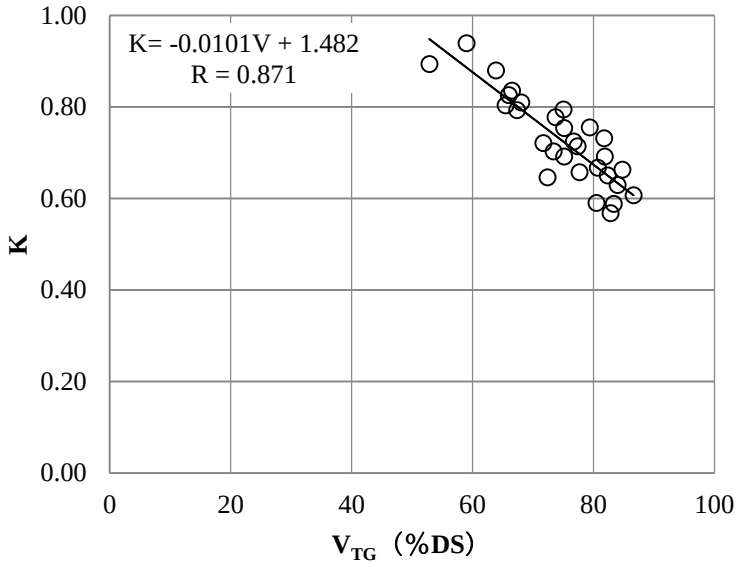


Figure 4-5: Calibration coefficient (K) and volatile matter measured by TG (V_{TG}) values of samples obtined during period A.

$$Hl_{DTA} = (Hls_{DTA}/(-0.0101V_{TG}+1.482)) \quad 4\text{-}5$$

where Hl_{DTA} and Hls_{DTA} are the LHVs obtained in this study and from the peak area, respectively (kJ/kg-DS).

The Hl_{DTA} values of samples obtained during periods A and B were compared with the LHVs measured by bomb calorimetry and CHN analysis (Hl) (Figure 4-6). The samples exhibited a correlation of 1:1. The errors of the estimated LHVs were calculated by Eq. 4-6.

$$H = |Hl_{DTA} - Hl|/Hl \times 100 \quad 4\text{-}6$$

The average correlation errors of the LHV calculated by Eq. 4-5 were $5.2 \pm 4.0\%$ and $5.17 \pm 4.07\%$ for samples obtained during periods A and B, respectively. Thus, the LHV of samples from periods A and B can be estimated with similar accuracy.

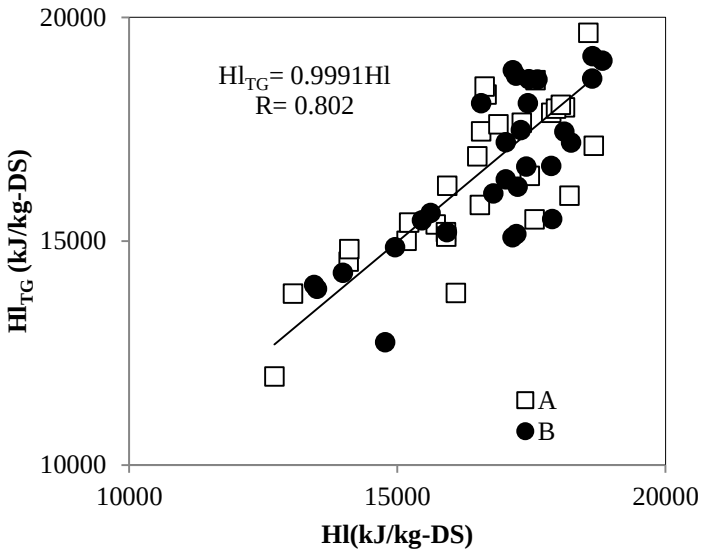


Figure 4-6: Relationship between the lower heating value (LHV) obtained by the standard method and that obtained by DTA.

4.4 Conclusion

In this study, the ability of TG-DTA to predict the V content and LHV of sewage sludge was investigated. The following conclusions were reached:

1- The two-stage weight loss is likely due to combustion of organic matter. The V contents measured by a typical sewage testing method and from the TG curve were strongly correlated (average relative error and standard deviation, 1.02 and 0.62%, respectively). Thus, the V content of sludge samples can be predicted by TG.

2- The DTA curve exhibited two peaks, at 200 to 350°C and 350 to 550°C. It was divided into two major groups with respect to the DTA peak area ratio. Mathematical quantification theory class 1 showed that digestion exerts the greatest effect on the area of the DTA curve.

3- The DTA peak area of sewage sludge samples obtained during period A was used to predict the LHV. First, the LHV obtained from the DTA peak area was lower than that calculated using the combination of HHV (measured by bomb calorimetry) and ultimate analysis. Furthermore, the calibration coefficient (K) was strongly negatively correlated with the V content; that is, as the V content increases, the heat loss of DTA increases.

4- The LHV calculated by Eq. 4-5 and that determined by the standard method exhibited an approximately 1:1 correlation. The average correlation error and standard error of period A and B samples were 5.20% and 4.00%, and 5.17% and 4.08%, respectively. Thus, this method can be applied to sewage sludge samples obtained during other seasons, or from other WWTPs.

Therefore, TG-DTA can be used to predict the V-percentage and LHV of sewage sludge. However, further investigation of the rate of temperature increase and the sample quantity required is necessary.

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Chapter 5
Heating Value Distribution of Suspended Solids in Wastewater

5.1 Introduction

Wastewater contains solids that can be utilized as a source of energy. Shizas and Bagley showed that the energy content of wastewater is 3.2 kJ/g of dry solids and that of the chemical oxygen demand (COD) is approximately 14.70 kJ/g. This value was similar for all stages of wastewater treatment plants (WWTPs); *e.g.,* primary and secondary sludge. Heidrich *et al.* reported that the energy contents of the COD of two WWTPs were 22.5 and 17.7 kJ/g. However, COD can be converted to methane (CH_4) by anaerobic digestion, the heating value of which is 55.53 kJ/g; because 1 kg of methane is equivalent to 4 kg of COD, the heating value of COD is 13.88 kJ/g (Shizas & Bagley, 2004; Heidrich *et al.*, 2010; Garrido *et al.*, 2013).

In a typical WWTP, more than 60% of the organic matter in wastewater is concentrated in sludge, which comprises primary sludge (PS) and waste activated sludge (WAS). Thus, 60% of the energy content of wastewater is in sludge and can be recovered (Garrido *et al.,* 2013).

PS is an aggregate of suspended solids (SS) removed by the primary sedimentation tank (PST). The particle size distribution (PSD) of SS in influent is key for determining the characteristics of PS. The PSD of wastewater and its characteristics, as well as the effects of primary and secondary treatment, have been investigated. The particle size in wastewater is reduced by the primary treatment process; *i.e.*, in the PST (Levine *et al.*,1991; García-*Mesa et al.*, 2010). However, the heating values of the various particle size ranges in wastewater are unclear.

In this study, we analyzed the PSD of the influent, primary treated wastewater (PTW), and PS of two WWTPs (A and B) in Japan. We also measured the carbon, hydrogen, and nitrogen (CHN) content and heating value of each particle size range of samples from both WWTPs to determine the relationship between the particle size and heating value, as well as the effects of particle size on the mass and heating value distributions. In addition, we determined the mass and energy recovery rates according to particle range to evaluate the energy-recovery efficiency of the two WWTPs.

5.2 Materials and Methods

5.2.1 Materials

Samples of influent, PS, and PTW were collected from two WWTPs (A and B) in Japan, which have combined and separate sewage-collection systems, respectively. The treatment capacity of WWTP A is 914,000 m^3/day, and that of WWTP B is 115,000 m^3/day. The secondary (biological) treatment process is conventional activated sludge process for WWTP A and anaerobic-anoxic-oxic process for WWTP B. Sludge dewatering is performed by screw press in both WWTPs. Spot sampling was performed for both WWTPs. Figure 5-1 shows the process flow of the two WWTPs and the sampling points used in this study.

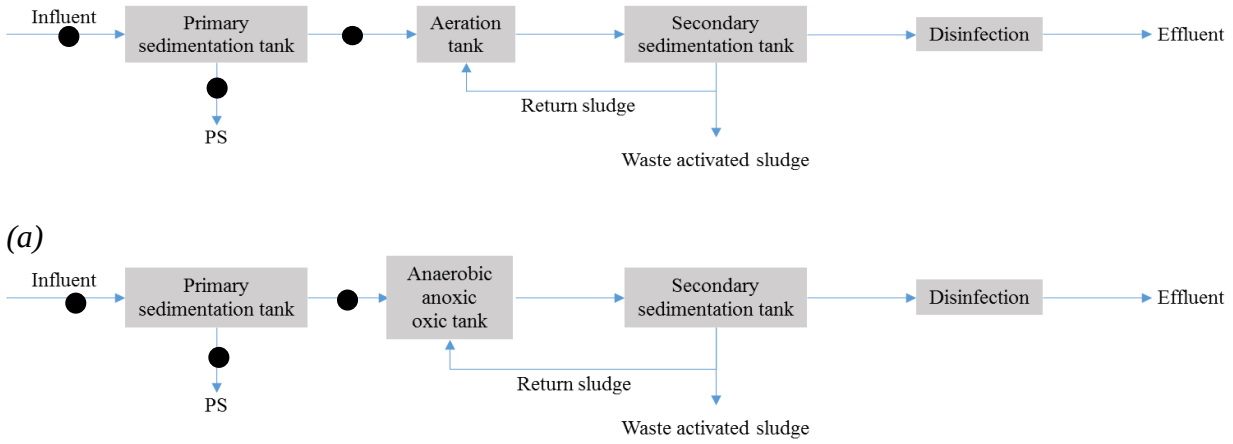


Figure 5-1: Flow chart of WWTPs A (a) and B (b).

5.2.2 Methods

5.2.2.1 Fundamental analysis

In this study, fundamental analysis of the influent, PTW, and PS of the two WWTPs was performed by determining the total solids (TS), SS, volatile solids (VS), and volatile suspended solids (VSS) contents. These parameters were measured following the Wastewater Examination Method of Japan (Japan Sewage Works Association, 2012). Chemical oxygen demand (COD_{Cr}) and soluble COD_{Cr} were analyzed by the US Environmental Protection Agency reactor digestion method by Hach Co. Ltd. (Hach, 2014). In this method, after blending and transferring 2 mL (for samples with a COD_{Cr} of 3–250 mg/lit) or 0.2 mL (COD_{Cr} of 250–1500 mg/lit) of the samples to vials (Hach Co., Ltd., Colorado, United States) and heating to 150–160°C for 2 h, COD_{Cr} was measured by colorimetry (DR 890, Hach Co., Ltd., CO). CHN levels were measured using a CHN analyzer (JM10, J-Science Lab Co., Ltd., Kyoto, Japan).

5.2.2.2 Particle size distribution

Particle size analysis was carried out by sieving and filtration. The samples were passed through 1,000-, 500-, 250-, 150-, 75-, 45-, and 20-μm sieves (JIS Z 8801, Tokyo Screen Co. Ltd., Tokyo, Japan), followed by vacuum filtering through a glass-fiber filter (GS-25; φ 47 mm, Advantec, Tokyo, Japan) to remove 1-μm particles. The particles remaining on the sieve were collected by back-washing using deionized water and filtering through a glass filter. The PSD of the samples was calculated by determining the mass of particles on each filter, and calculating the proportion of the total mass of particles.

5.2.2.3 TG-DTA and bomb calorimetry

Bomb calorimeter was used to measure the higher heating value (HHV) of samples available in sufficient quantity (*e.g.*, the SS or TS content of PS [0.5–1 g]). However, bomb calorimetry could not be used to analyze the heating values of all particles due to insufficient sample quantities. Thus, thermogravimetric and differential thermal analysis (TG-DTA) (TG8110; Thermoplus, Rigaku, Tokyo, Japan) was conducted to determine the HHVs. A previous study used the TG-DTA method to evaluate the HHV of coal using DTA data and the associated exothermic peak area, and compared these values with those calculated by ultimate analysis. The authors concluded that TG-DTA is suitable for analyzing the HHV (Muñoz-Guillena *et al.*, 1992). In the present study, 10 mg of each dried sample was placed in an alumina crucible and heated from ambient temperature to 650°C under air flow of 50 mL/min and a heating rate of 10°C/min. Figure 5-2 shows an example of a TG-DTA curve.

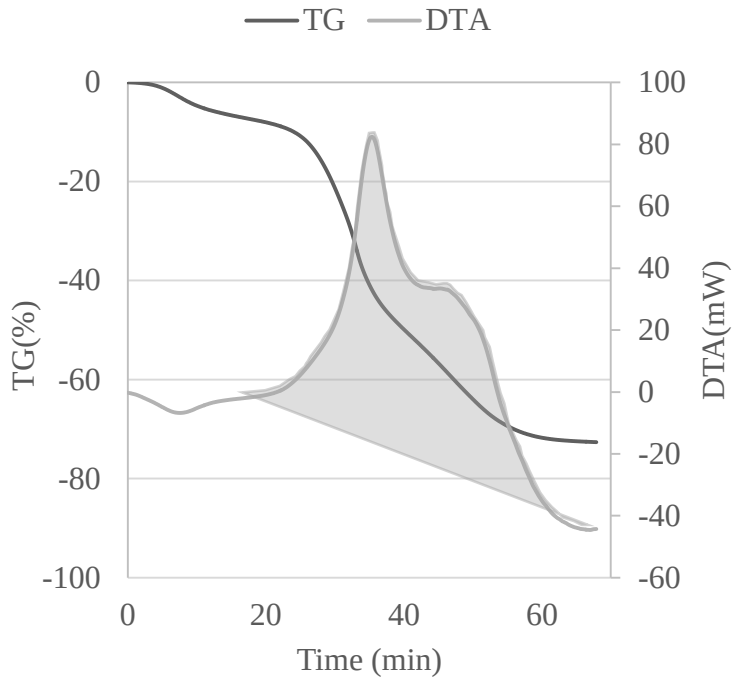


Figure 5-2: TG-DTA curve.

TG-DTA analysis was performed in triplicate for each particle size range. The lower heating value (LHV) of each particle size range was calculated by Eq. 5-1, or by Eq. 5-2 for samples available in sufficient quantity.

$$LHV=A/K\times10^{3} \quad (5\text{-}1)$$

$$LHV=HHV-9\times25\times H \quad (5\text{-}2)$$

where LHV is the lower heating value (J/g-DS), A is the area under the DTA curve (J/g), and K is the instrumental constant. HHV is the higher heating value (J/g-DS), and H is the hydrogen percentage (%-DS). The area under the DTA curve was determined (Figure 5-2) using software.

The value of K was negatively correlated with the VS content of sludge. About 300 mg of 250–1,000-μm and 1–45-μm particles were collected from PS and the

correlation of their VS and K values was determined for the purposes of calibration. Sufficient quantities of samples were available for determination of heating values by bomb calorimetry. The LHVs of these samples was calculated by Eq. 5-2. Samples with higher VS values had lower K values (Figure 5-3), Equation 5-3 (in Figure 5-3) was used to calculate the K value of each particle size range.

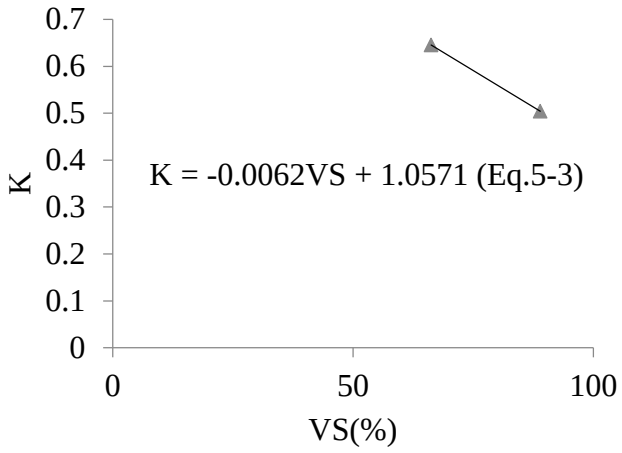


Figure 5-3: Correlation of the VS and K values of two particle size ranges.

5.3 Results and Discussion

5.3.1 Fundamental analysis

Table 5-1 shows the fundamental analysis, heating values, and CHN values of samples from the two WWTPs. The influent of WWTP B (which has a separate sewage-collection system) had higher TS, SS, VTS, VSS, and COD_{Cr} contents than that of WWTP A. Moreover, WWTP B had more organic matters than WWTP A regarding CHN result. Although the TS value of the influent of WWTP B was higher than that of WWTP A, their SS values were similar, indicating that the higher dissolved solids level of WWTP B and results in a reduced efficiency of total solid removal.

The carbon content of PS was higher than that of influent. Thus, the heating value of solids of PS was expected to be higher than that of influent. The COD decreased by almost 70 and 40% in the PST of WWTP A and B, respectively. The magnitude of the reduction in the COD of WWTP B was less than that of WWTP A due to its higher DS content, which is not removed in the PST. The carbon content and HHV of solids of the PS of WWTP B were higher than those of WWTP A, likely due to the different sewage-collection systems. In combined sewage systems, solids from other sources (such as rainfall, which contains soil and has a low organic compound content and heating value) reduces the heating value and COD. CHN analyses of each particle size range in influent, PTW, and PS were performed (Table 5-2). We were unable to measure the CHN content (%-DS) of 1,000–2,000-μm particles due to insufficient sample quantities. The hydrogen content was similar for all particle size ranges in the influent and PS of both WWTPs. However, the C content increased with increasing particle size in the influent and PS of WWTP A. The nitrogen content of small particles was higher than that of large particles.

Table 5-1: Fundamental analysis of WWTP samples.

WWTP	A			B		
	Influent	PTW	PS	Influent	PTW	PS
TS (mg/L)	350±40	220±20	8,070±70	530±10	370±10	6,970±130
SS (mg/L)	150±10	20±0	7,360±300	170±10	40±10	6,250±270
VTS (mg/L)	180±30	80±10	6,420±70	290±0	150±20	6,150±110
VSS (mg/L)	120±10	20±0	5,920±220	150±20	40±10	5,720±240
COD (mg/L)	240±40	70±0	9,550±370	630±90	380±60	10,030±370
S-COD(mg/L)	40±0	40±0	550±110	NA	NA	NA
C (% TS)	20.8±0.2	13.8±0.1	37.7±0.1	22.5[a]	18.7[a]	43.7[a]
H (% TS)	3.6±0.1	2.2±0.1	6.0±0.0	3.4[a]	3.0[a]	6.9[a]
N (% TS)	2.2±0.0	1.2±0.1	3.2±0.1	1.8[a]	1.5[a]	2.9[a]
HHV (J/g TS)	NA	NA	16,800[a]	NA	NA	19,200[a]

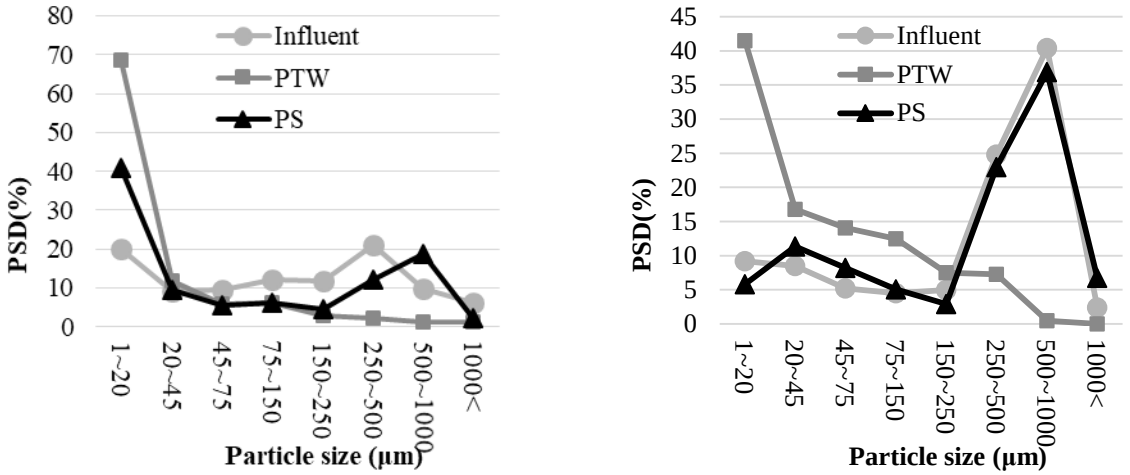
NA, not analyzed, [a] single determination

Table 5-2: CHN contents of particles according to size range.

WWTP	A						B					
	Influent			PS			Influent			Ps		
	C	H	N	C	H	N	C	H	N	C	H	N
Diameter (μm)	% SS	% SS	% SS	% SS	% SS	% SS	% SS	% SS	% SS	% SS	% SS	% SS
1–20	39.3	6.6	6.6	36.7	6.3	6.3	46.6	6.9	7.0	44.9	6.7	7.5
20–45	37.7	6.5	5.3	35.3	6.1	4.0	42.5	6.6	3.6	42.3	6.5	4.6
45–75	37.0	6.4	4.2	34.7	5.7	3.5	44.3	6.4	3.1	44.4	6.8	4.6
75–150	39.0	6.5	3.6	38.6	6.4	3.2	42.3	6.2	2.0	44.7	6.9	3.2
150–250	40.6	6.4	2.3	41.1	6.7	1.6	42.8	6.7	0.8	43.6	6.7	1.3
250–500	41.3	6.5	1.8	41.7	6.7	0.8	43.0	6.6	0.7	44.3	6.8	1.0
500–1,000	41.7	6.5	2.5	41.1	6.7	0.7	43.6	6.8	0.8	43.8	6.8	0.6

5.3.2 Particle size distribution

Figure 5-4 shows the PSD of the influent, PS, and PTW of WWTP A and B. In the influent of WWTP A, 1–20-μm and 250–500-μm particles predominated, comprising 20% of SS. Each of the other particle size ranges comprised ~10% of SS. However, in the influent of WWTP B, 250–500-μm and 500–1,000-μm particles predominated, comprising approximately 65% of the total particles. The PSD of the PTW showed a marked shift to smaller particles compared with the influent, as reported by Levine *et al*. (1991), suggested that the decrease was due to primary sedimentation. Moreover, Levine and co-workers showed that particles > 10 μm comprise approximately 60% of the SS of influent, compared with 40% after primary sedimentation. Zhang *et al*. (2006) stated that the proportion of large particles, particularly those > 10 μm, decreases in the PST. In this study, the primary sedimentation process decreased the > 10-μm particle content of the influent of WWTP A and B from 90 to 65% and 95 to 80%, respectively. These values are in good agreement with those reported by others (Levine *et al.*,1991, Zang *et al.*, 2006).



a b

Figure 5-4: PSD of (a) WWTP A and (b) WWTP B.

Table 5-3 shows the flow rate and daily mass content (DMC) of particles, as well as the mass recovery rates (M_R [%]) of WWTPs A and B. The influent and PTW flow rates of WWTP A were assumed to be equal. However, the flow rate of PTW in WWTP B was higher than that of the influent due to the return of supernatant from sewage sludge dewatering. DMC is calculated as in Eq. 5-4.

$$DMC = P \times SS \times F / 1{,}000 \quad (5\text{-}4)$$

where DMC is the daily mass content (kg/d), P is particle percentage (%), SS is the SS content (mg/L), and F is the flow rate (m^3/d).

Table 5-3: DMC and mass balance values of PST.

WWTP	A				B			
	Influent	PTW	PS	M_R	Influent	PTW	PS	M_R
Flow rate (m^3/day)	42,570	42,570	450		113,418	123,288	2,429	
	kg/day	kg/day	kg/day	%	kg/day	kg/day	kg/day	
Diameter (µm)								
1–20	1,270	690	1,350		1,730	1,890	880	
20–45	590	120	310		1,600	760	1,720	
45–75	600	60	180		980	640	1,250	
75–150	780	60	210		850	570	770	
150–250	750	30	150		930	340	440	
250–500	1,340	20	400		4,670	330	3,490	
500–1,000	620	10	630		7,610	20	5,600	
1,000–2,000	400	10	80		450	0	1,020	
Sum	6,360	1,000	3,310	68	18,810	4560	15,170	104

The M_R was calculated for the sum of all PSDs, which is the ratio of the DMC of PS + PTW to that of the influent (Eq. 5-5).

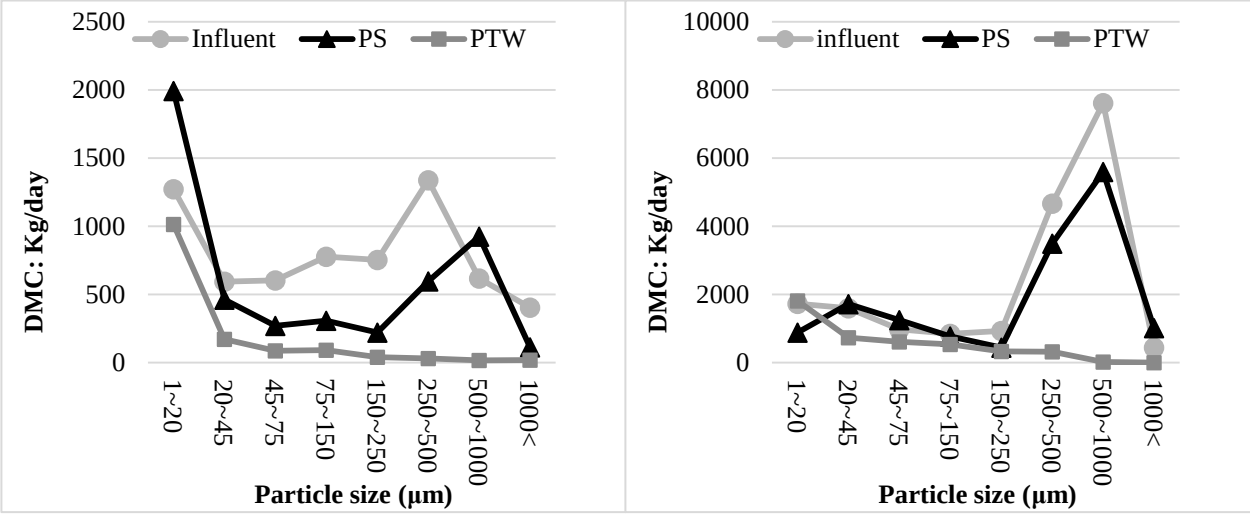
$$M_R = (M_{PS} + M_{PTW})/M_{INF} \times 100 \quad 5\text{-}5$$

where M_{PS}, M_{PTW}, and M_{INF} are the DMC values of PS, PTW, and influent, respectively (kg/d). The M_R values of both WWTPs were not 100% due to experimental error. However, low M_R of WWTP A can be due to sampling of influent which the effect of return waster (that contain mainly small particle) is not considered. In other words, the sample of INF of WWTP A in not the representing input of PST.

Thus, the DMC values of particles in PTW and PS were revised so that M_R equaled 100% (Table 5-4; Figure 5-5).

Table 5-4: Revised DMC and MR values of WWTPs.

WWTP	A			B		
	PTW	PS	M_R	PTW	PS	M_R
	kg/day	kg/day	%	kg/day	kg/day	%
Diameter (μm)						
1–20	1,010	1,990	236	1,810	840	153
20–45	170	460	107	730	1,640	148
45–75	90	270	59	610	1,190	184
75–150	90	310	51	540	730	150
150–250	40	220	35	330	420	80
250–500	30	600	47	320	3,330	78
500–1,000	20	920	153	20	5,340	70
1,000–2,000	20	110	32	0	970	218
total	1,470	4,890	100	4,350	14,470	100



a b

Figure 5-5: DMC values of particles in (a) WWTP A and (b) WWTP B.

The fractions of some particle size ranges changed during sedimentation, although the total mass recovery rate was 100%; this suggests changes in PSD in the PST. For example, in WWTP A, the mass recovery of 1,000–2,000-μm particles was 32%, compared with 236% for 1–20-μm particles. Thus, large particles crushed to smaller ones. Furthermore, crushed 1,000–2,000-μm particles in influent can be integrated

into the 500–1,000-μm range (because M_R > 100%). For 250–500, 150–250, 75–150, and 45–75-μm particles, the M_R values were less than 100%, but the M_R values of 1–20 and 20–45-μm particles were high. Therefore, 45–500-μm particles tend to integrate to 1–45-μm particles. However, this finding is not in agreement with PST basics. The high presence of small particle in PS of WWTP A is due to addition of return water before PST which is not considered in this study.

In WWTP B (which has a separate sewage-collection system), 500–1,000-μm particles in influent were integrated into 1,000–2,000-μm particles, because the mass recovery rate of this range was >100%. Moreover, a proportion of 500–1,000-μm particles fragmented to smaller particles in the PST. Furthermore, 150–500-μm particles fragmented and were integrated into 1–150-μm particles, which increased the mass recovery rates of 1–20, 20–45, 45–75, and 75–150-μm particles. However, the smallest particles predominated in the PTW of both WWTPs. Thus, the PSD of wastewater shifts to smaller particle sizes in the PST. This is in agreement with the results described above (Levine *et al.*,1991, Zang *et al.*, 2006). The difference in PSD and integration between WWTPs A and B is likely due to their different sewage-collection systems.

5.3.3 Heating value analysis

The HHVs for all particle size ranges in SS in the influent and PS of WWTPs A and B are shown in Table 5-5. HHV was calculated by Eqs. 5-1 and 5-2. However, the HHVs of 1,000–2,000-μm particles in PS and all particle size ranges in PTW could not be measured due to insufficient sample quantity. The K value for each particle size range was calculated by Eq. 5-3 using the VS determined by TG analysis. The HHV of particles in the influent of WWTP A and B was 17.0–20.9 and 16.8–21.1 MJ/kg, respectively. The HHV of particles from WWTP B tended to be higher than

that of WWTP A, which is in accordance with the HHV values of the SS in PS of both WWTPs (Table 5-1). However, the HHV of the SS in PS (calculated by Eqs. 5-1 and 5-2) was higher than that measured by bomb calorimetry; therefore, the HHV of each particle size range was also likely overestimated. The HHV of SS_{calc} is the weighted average of the HHV of particles (Table 5-5).

Table 5-5 HHVs of particles in the PS and influent of WWTP A and B.

WWTP	A			B		
	Influent	PS	PTW	Influent	PS	PTW
	J/g	J/g	J/g	J/g	J/g	J/g
Diameter (μm)						
1–20	20,900	19,300	-	21,100	20,000	-
20–45	18,700	18,000	-	21,100	19,600	-
45–75	16,700	16,700	-	18,800	20,700	-
75–150	18,000	18,100	-	18,000	20,500	-
150–250	17,300	18,100	-	17,100	19,100	-
250–500	17,300	16,000	-	17,900	18,400	-
500–1,000	18,700	15,400	-	16,800	16,900	-
1,000–2,000	17,000	-	-	17,900	18,400	-
SS	18,600	18,300	22,000	21,200	21,500	20,300
SS_{calc}	18,300	17,400	-	17,800	18,400	-

5.3.4 Energy balance

Using the HHV of each particle size range and the PSD for WWTP A, the contribution of each particle size range to the total heating value or daily energy content (DEC) can be determined.

$$DEC = M \times E \times 10^{-6} \quad 5\text{-}6$$

where *DEC* is the daily energy content of each particle size range (GJ/d), *M* is the DMC of each particle size range (kg/d), and *E* is the HHV of each particle size range (J/g).

Table 5-6 and Figure 5-6 show the DEC values of particles in the influent and PS, and those of the SS in the influent, PS, and PTW, of WWTPs A and B. The DEC values of the SS in influent, PS, and PTW differ from those calculated by summing the DEC values of all particle size ranges. However, the error was samll in most cases. The DEC value of the SS of PS of WWTP B was considerably different from that calculated by summing the DEC values of all particle size ranges. This may be because of K value calculated by Eq. 5-3, which is evaluated for analysis of particles of WWTP A (which has a different wastewater-collection system than WWTP B). The HHV of particles in PTW could not be measured due to insufficient sample quantities, but for calculating the DEC value, the HHV of each particle size range in PTW was assumed to be identical to that in PS. The total energy losses (E_L; Eq. 5-7) and recovery rates (E_R; Eq. 5-8) were approximately 23% and 96% for WWTP A and 26% and 105% for WWTP B, respectively.

$$E_L = E_{PTW}/E_{INF} \times 100 \quad (5\text{-}7)$$

$$E_R = (E_{PTW}+E_{PS})/E_{INF} \times 100 \quad (5\text{-}8)$$

where E_{PTW}, E_{INF}, and E_{PS} are the *DEC* values (GJ/d) of PTW, influent, and PS, respectively.

Table 5-6: DEC values.

WWTP	A					B				
	Influent	PS	PTW	E_L	E_R	Influent	PS	PTW	E_L	E_R
	GJ/d	GJ/d	GJ/d	%	%	GJ/d	GJ/d	GJ/d	%	%
Diameter (μm)										
1–20	27	38	20	74	215	36	17	36	99	145
20–45	11	8	3	27	100	34	32	14	43	138
45–75	10	5	1	10	60	18	25	13	68	202
75–150	14	6	2	14	57	15	15	11	73	171
150–250	13	4	1	8	38	16	8	6	39	89
250–500	23	10	0	0	43	79	61	6	7	85
500–1,000	12	14	0	0	117	128	90	0	0	71
1,000–2,000	7	0	0	0	0	8	18	0	0	224
SUM	117	85	27	23	96	335	266	87	26	105
SS	119	89	32	27	102	399	311	88	22	100

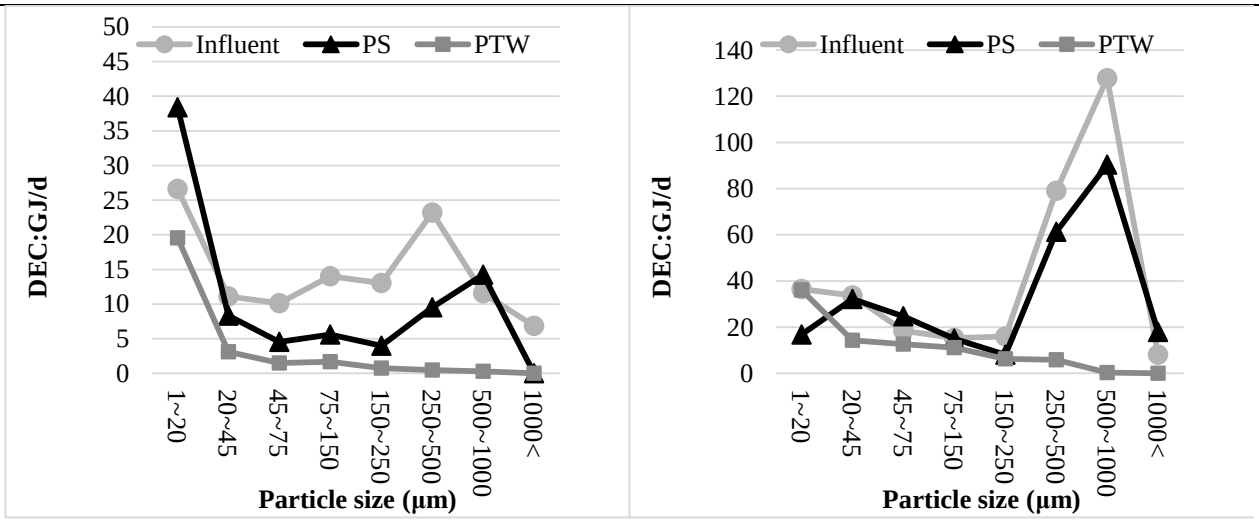


Figure 5-6: DEC values of particles of (a) WWTP A and (b) WWTP B.

For WWTP A, the DEC value of influent was 119GJ/d, and 32 GJ/d was released to PTW from the PST. Thus, approximately 25% of the energy in SS of influent is not extracted by the PST; the remaining energy is extracted as SS in PS. As expected, the high DMC values of 1–20 and 250–500-μm particles in influent resulted in an increased DEC value of these particles. Most of the energy in >20-μm particles was

extracted from influent; the DEC of these particles in PTW was negligible. Due to the aggregation of 45–500-μm particles in the range of 1–20 μm (as in the PSD experiment), the E_R value of 45–500-μm particles was <100%. However, particle integration resulted in an E_R value of 1–20-μm particles of 215%.

Approximately 70% of the DEC of PTW is from 1–20-μm particles. Notably, the change in DMC structure in the PST resulted in a shift in the DEC value of 20–500-μm particles of influent to 1–20 μm particles in PS.

For WWTP B, the DEC value of SS in influent was 399 GJ/d, which is markedly higher than that of WWTP A (mainly because of the higher DMC of SS). Approximately 80% of the DEC of influent can be extracted in the PST. This recovery rate is significantly higher than that of WWTP A due to the high DMC of large particles; *e.g.*, 250–1,000-μm particles. Integration of some 500–1,000-μm particles in 1,000–2,000-μm particles resulted in an E_R value of 224%. However, the DEC of 1,000–2,000-μm particles in PS was only 10 GJ/d higher than that of influent. As in the PSD experiment, the aggregation of 150–1,000-μm particles in smaller ones resulted in an E_R value of <100%. In contrast, the increased DMC value of 1–150-μm particles conferred an E_R value > 100%.

Similarly, 1–20-μm particles were important determinants of the DEC of PTW in WWTP B; the DMC of those particles was not fully recovered in the PST. Indeed, 40% of the DEC value of SS in PTW was from 1–20-μm particles. Due to the high DMC value of large particles; *e.g.*, 250–1,000-μm particles, and their integration into 1–150-μm particles in the PST, the DEC value of PS fell within this range. Moreover, because they settle very slowly, large particles in PTW had a high DEC value.

5.3.5 PSD removal scenarios and energy recovery

Mass and energy recovery from wastewater according to PST surface area are discussed in this section. Stoke's law was applied to calculate the sedimentation velocity of each particle size range (Metcalf & Eddy, 2003; Qasim *et al.*, 2000) (for WWTP A, Eqs. 5-9 ,5-10, and 5-11).

$$V_{Sc} = \sqrt{4/3 \times \frac{dg}{C_d} \times (S_g - 1)} \quad 5\text{-}9$$

$$V_{Sc} = \frac{g d^2 (S_{g-1})}{18v} \quad 5\text{-}10$$

$$E = \frac{V_{SC}}{OR} \quad 5\text{-}11$$

where V_{SC} is the settling velocity (m/s), d is the particle diameter (m), v is the kinematic viscosity (assumed to be 1.003×10^{-6} m^2/s), S_g is specific gravity (assumed to be 1.60 g/cm^2), and OR is the overflow rate. These assumptions are based on previous studies (Metcalf & Eddy, 2003; O'Kelly, 2005). In this study, PST depth was assumed to be 3.15 m and the OR was 2 h. The characteristics of PSTs according to surface area (A) are shown in Table 5-7.

Table 5-7: Characteristics of the PST of WWTP A according to surface area.

WWTP	A	
	HRT (h)	OR (m/h)
A=0%	2	1.575
Scenario 1: A=25%	0.5	6.3
Scenario 2: A=50%	1	3.15
Scenario 3: A=150%	3	1.05
Scenario 4: A=200%	4	0.7875

HRT, hydrolic retention time

The mass recovery rate of each particle size range is shown in Table 5-8; the mass recovery rate decreased with decreasing surface area. However, in most cases, the

mass recovery rate of >45-µm particles was 100%. The total mass recovery rate was 71% for scenario 1 and 83.8% for scenario 4. The total energy recovery rate was calculated by Eq. 5-12 and is shown in table 5-9.

$$E_E = \frac{\sum DMC \times HHV}{DEC_{SS}} \qquad 5\text{-}12$$

where E_E is the total energy recovery rate of suspended solids (%), DMC and HHV are the daily mass content and higher heating value, respectively, and DEC_{SS} is the daily energy content of SS. Table 5-9 shows the energy recovery rates of SS.

Table 5-8: Mass recovery rates of SS (total).

Diameter (µm)	A=0%	Scenario 1	Scenario 2	Scenario 3	Scenario 4
1–20	9.5%	2%	5%	14%	19%
20–45	91.5%	23%	46%	100%	100%
45–75	100.0%	78%	100%	100%	100%
75–150	100.0%	100%	100%	100%	100%
150–250	100.0%	100%	100%	100%	100%
250–500	100.0%	100%	100%	100%	100%
500–1,000	100.0%	100%	100%	100%	100%
1,000–2,000	100.0%	100%	100%	100%	100%
Total	81%	71.2%	75.9%	82.9%	83.8%

Table 5-9: Energy recovery rates of SS.

	A=0%	Scenario 1	Scenario 2	Scenario 3	Scenario 4
E_E	79%	68%	73%	82.9%	83.8%

The energy recovery rate increased with increasing PST surface area due to the increased mass recovery rate. The energy recovery rate decreased by approximately 10% when the PST surface area was reduced by 75%, but the hydrolic retention time decreased to 30 min. However, doubling A increased the energy recovery rate by only ~5%.

5.4 Conclusion

We analyzed the PSD and heating values of them in influent, PS, and PTW of two WWTPs in Japan. The particle size of wastewater decreased during the primary sedimentation process. The PSD influenced the heating value distribution of the wastewater. Small particles exerted the greatest effect on heating values characteristic of the wastewater SS, because they predominated over other particle sizes in the PTW. The PSD has more effect compared to heating value of particles on DEC because HHV of particles are almost equal.

Approximately 75% of the energy in SS in influent can be recovered in the PST; the remainder is lost as SS in the PTW of both WWTPs. However, the energy recovery rate decreased with decreasing particle size, indicating that removal efficiency decreased with decreasing particle size, and the energy within those particles was lost. Removal of small particles could be enhanced by increasing the efficiency of the primary treatment process, which would also increase the amount of energy recovered. This could be achieved by adding other low-energy-input primary treatment processes, such as filtration, or by application of chemical processes, such as flocculation and coagulation, prior to primary treatment.

Finally, changes in PST surface area has a negligible effect on mass and energy recovery. Changing surface area for WWTPs those particle is easy to sediment, seems not to be effective. This subject would be discussed in next chapter.

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Chapter 6
Daily Energy Content of Municipal Wastewater Treatment Plant and the Effect of Physical Recovery Efficiency

6.1 Introduction

Wastewater is composed of more than 99% water and less than 1% solid. The wastewater treatment process transforms the solid component into CO_2, N_2, and sewage sludge. Most wastewater treatment plants (WWTPs) recover solids and dissolved materials by applying physical sedimentation and aerobic biological processes, which produce primary and secondary sludge, respectively. Every wastewater treatment step consumes energy. According to previous studies, the most energy-intensive step is aeration, which is part of the biological treatment process. The energy consumption due to aeration in WWTPs that use conventional activated sludge (CAS), sludge treatment, and secondary sedimentation constitutes 50–60%, 15–25%, and 15%, respectively, of the total energy used by WWTPs (Mamais *et al.*, 2015). Figure 6-1 shows the proportion of energy consumed by each part of the WWTP according to Gu *et al.* (2017).

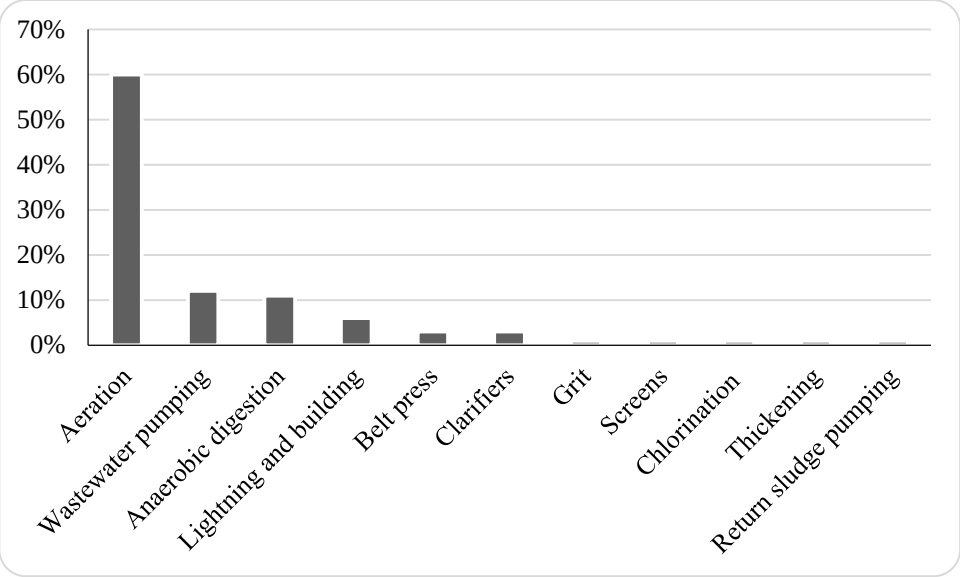


Figure 6-1: Energy consumption rate at each stage of a WWTP (Gu et al.*, 2017).*

In WWTPs that implement aeration, pumping, and digestion, these stages will have the highest energy requirements. These three parts consume approximately 80% of the total energy consumed by the WWTP.

The energy use intensity (EUI), which is proportional to the energy consumption and influent flow (kWh/m^3), varies widely between WWTPs from different countries. Table 6-1 shows results from previous studies, where typical EUIs were determined for WWTPs in a selection of countries.

Table 6-1: The energy use intensity in a variety of countries.

County	EUI (kWh/m^3)	Reference
USA	0.78	Garrido *et al.*, 2013
Canada	0.35	Fenu *et al.*, 2011
Germany	0.4-0.43	Wang *et al.*,2016
Austria	0.30	Jonasson, 2016
Sweden	0.47	Jonasson, 2016
South Africa	0.18-0.47	Wang *et al.*,2016

The organic matter contained in the solid component of the wastewater can be converted into energy thermodynamically. The heating value of suspended solids of wastewater was addressed in the previous chapter. Shizas & Bagley showed that wastewater contains 3.2 kJ/g of dry solids and assessed the energy content by calculating the chemical oxygen demand (COD), which was equal to 14.70 kJ/g-COD. However, the heating value of COD of primary sludge (PS), secondary sludge, and aerobically digested sludge are 11.12, 12.05, and 11.68 kJ/g-COD, respectively. Experimental studies have confirmed that this value is almost similar for all stages of WWTP *e.g.* primary of secondary sludge. However, Heidrich *et al.* assessed the energy contents of two WWTPs with HHV of CODs equal to 22.5 and 17.7 kJ/g-COD. There is a notable difference between the heating values of the organic matter from the two WWTPs. This suggests that we should develop an alternative method for evaluating the heating value of organic matter in wastewater.

However, anaerobic digestion can convert COD into methane (CH_4), which has a heating value of 55.53 kJ/g. Given that 1 kg of methane is equivalent to 4 kg of COD, the heating value of COD is equal to 13.88 kJ/g of COD (Shizas & Bagley, 2004; Heidrich *et al.*, 2010; Garrido *et al.*, 2013).

Hence, considerable energy is required to remove organic matter from the wastewater process. However, we can convert WWTPs from energy consumers to energy providers by recovering more organic matter from wastewater and using it as fuel for heat or electricity. Increasing the efficiency of the primary sedimentation tank (PST) has been shown to improve the energy recovery from WWTPs (Garrido *et al.*, 2013).

In this study, we investigated the effect of varying the solid recovery from the primary sedimentation process on the energy recovery of a Japanese WWTP. In the

experimental part of the study, we varied the surface area of the primary sedimentation tank. Our theoretical calculations were based on sedimentation theory, particle size distributon, and data described in Chapter 5.

6.2 Material and methods

6.2.1 Material

We spot-sampled influent (INF), PS, and primary treated wastewater (PTW) from the Toba WWTP. The flow rates of the INF, PS and PTW were 42600, 500, and 42100 m^3/d, respectively. The overflow rate (OR) was 40 m^3/m^2d and activated sludge was used in the biological treatment. The samples were stored at 4°C and analyzed shortly after collection. The characteristics of the influent from the Toba WWTP are summarized in Table 6-2.

Table 6-2: Influent characteristics.

Item	Symbol	Concentration (mg/L)
Total solids	TS	290
Suspended solids	SS	90
Volatile suspended solids.	VSS	80
Volatile dissolved solids	VDS	80
Fixed suspended solids	FSS	20
Fixed dissolved solids	FDS	120
Particulate organic carbon	POC	40
Dissolved organic carbon	DOC	30
Biological oxygen demand	BOD	90

We calculated the particulate organic carbon (POC) and dissolved organic carbon (DOC) to be 40 and 30 mg/L, respectively. We obtained these values measuring the C content of the total and suspended solids of influent those are 20 and 40%-DS respectively, assuming all of the C to be organic in both cases.

We considered the primary sedimentation process (PSP), aeration tank, and secondary sedimentation tank (SST) of the WWTP and that the treatment processes of the WWTP proceeded according to the flowchart presented in the previous chapter; the return flow factor was omitted from mass and energy recovery analyses. According to a previous study, the return flow only increases the influent flow by 6% and the SS_{INF} flow by 3% (Nakamura, 2013). Thus, we assumed that its effect on the characteristics of the sewage and sludge production would be negligible. Primary and waste-activated sludge (WAS) is produced during PSP and SST, respectively. In this study, we investigated the relationships between the surface area (A) of the PST and the PS, WAS production, and SS heating value.

6.2.2 Methods

In this study, we investigated the effect of increasing/decreasing the OR by changing the A of the PSP on the recovery of mass and energy from a WWTP. As previously mentioned, the OR was 39.4 $m^3/m^2.d$. The details of the variations in overflow considered in this study are summarized in Table 6-3.

Table 6-3: Overflow rate and surface areas used in this study.

OR decrease/increase (%)	0%	+30%	+100%	-20%	-30%	-50%
OR($m^3/m^2/d$)	40	52	80	32	28	20
A(m^2)	1080	830	540	1350	1550	2160
A decrease/increase (%)	0%	-23%	-50%	+25%	+43%	+100%

6.2.2.1 Primary sedimentation process

The removal ratio (E-%) of each factor in the PSP is defined as

$$E=100-M_{PTW}/M_{INF} \quad (6\text{-}1)$$

where M_{PTW} and M_{INF} are the mass flow rates of each factor of the PTW and INF, respectively. One of our main objectives was to calculate the particle removal ratio from the PST. We used the results from the PSD in the previous chapter in this chapter.

Figure 6-2 shows the sedimentation in the PST by the discrete settling mechanism. The settling equation obeys Newton's law and Stoke's law can be applied under linear conditions (Eq. 5-9 and 5-10) (Metcalf and Eddy, 2003; Qasim *et al.*, 2000).

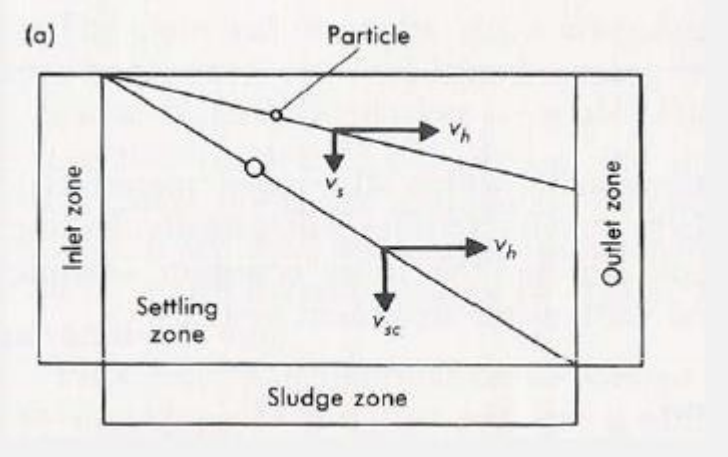


Figure 6-2: Discrete particle settling.

$$V_{Sc} = \sqrt{4/3 \times \frac{dg}{C_d} \times (S_g - 1)} \quad (6\text{-}2)$$

$$V_{Sc} = \frac{gd^2(S_g - 1)}{18v} \quad (6\text{-}3)$$

$$E = \frac{V_{SC}}{OR} \quad (6\text{-}4)$$

where V_{SC} is the settling velocity (m/s); d is the diameter of the particle (m); *v* is the kinematic viscosity, assumed to be 1.003×10^{-6} m^2/s; and *Sg* is the specific gravity, which we assumed to be 1.60 g/cm^2. These assumptions are based on values reported in previous publications (Metcalf & Eddy, 2003; O'Kelly, 2005).

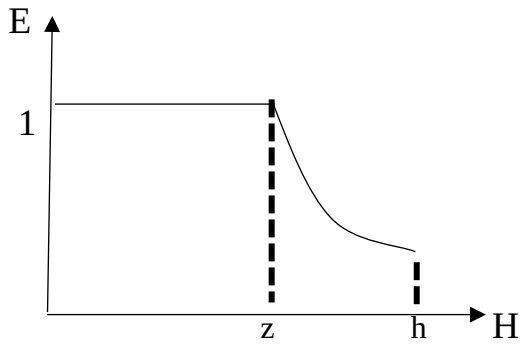
However, this method is applicable when all of the particles have begun to settle from the free surface of the PST. The Toba WWTP used a rectifier plate for PSP. Thus, all of the particles were considered to spread across the inlet zone. The sedimentation equation is as follows (h is distance from bottom of PST):

$$Vsc \times t = h \left(t = \frac{Volume}{Q}\right) \rightarrow Vsc = \frac{h \times Q}{Volume}$$

$$E = \frac{V}{Vsc} = \frac{V \times Volume}{h \times Q} = \frac{Z}{h}$$

$$For\ h \leq Z \rightarrow E = 1$$

$$For\ h > Z \rightarrow E = \frac{Z}{h}$$



Finally, the removal ratio can be calculated as follows:

$$E = 1/H \times (1 \times Z + \int_{Z}^{H} \frac{Z}{H} dh) = \frac{Z}{H}(1 + (LnH - LnZ))$$
$$= \frac{V_{SC}}{W_0}(1 + Ln\frac{W_0}{V_{SC}}) \quad 6\text{-}5$$

In this chapter, we use Eq. 6-5 to calculate the removal ratio of each particle. Table 6-4 shows the settling velocities of the particles described in Eq.6-3. The assumed diameter was defined as the average of the minimum and maximum diameter of each range. The Reynolds number (N_R) was also calculated as follows to check the settling region:

$$N_R = \frac{\emptyset V_{SC} d}{v} * 10^6 \quad 6\text{-}6$$

where $\emptyset$ is shape factor (assumed 1).

Table 6-4: Settling velocity of each particle when the overflow rate is 39.4 $m^3/m^2.d$.

Diameter (μm)	Assumed diameter (μm)	Settling velocity (m/s)	N_R
1~20	10.5	0.00003	0.00003
20~45	33	0.0003	0.01
45~75	60	0.0012	0.07
75~150	113	0.0041	0.46
150~250	200	0.0130	2.60
250~500	375	0.0458	17.14
500~1000	750	0.1834	137.13
1000~2000	1500	0.3260	325.05

Particles those Reynolds numbers is less than 1.0 assumed to be in laminar region which viscosity is the predominant force governing the settling process and settling velocity is considered same as described in Table 6-4. For particles with N_R bigger than 1.0, settling is in the transition region and its equation would be different.

However, the calculation showed that those particles are 100% removable because of high settling velocity in the WWTP.

The SS removal ratio is calculated as follows:

$$E_{SS}=\frac{\sum E\times DMC_{INF}}{\sum DMC_{INF}} \quad 6\text{-}7$$

where E_{SS} is the removal ratio of suspended solids (%) and DMC is the daily mass content of each particle in the INF (ton/d).

The removal ratio of the suspended parameters in the PSP are assumed to be the same as the removal ratio of the SS and that of the dissolved particles was assumed to be equal to the flow removal ratio (B). Figure 6-3 shows the removal ratio of each parameter of the PSP. However, the BOD removal ratio is of particular importance, especially with respect to biological processes. The BOD removal ratio is calculated as follows:

$$BOD_{Removal}=\frac{t}{a+bt} \quad 6\text{-}8$$

where *a* and *b* are constants, which we assumed to be 0.018 and 0.02, respectively, and *t* is the hydraulic retention time (h) (Metcalf & Eddy, 2003).

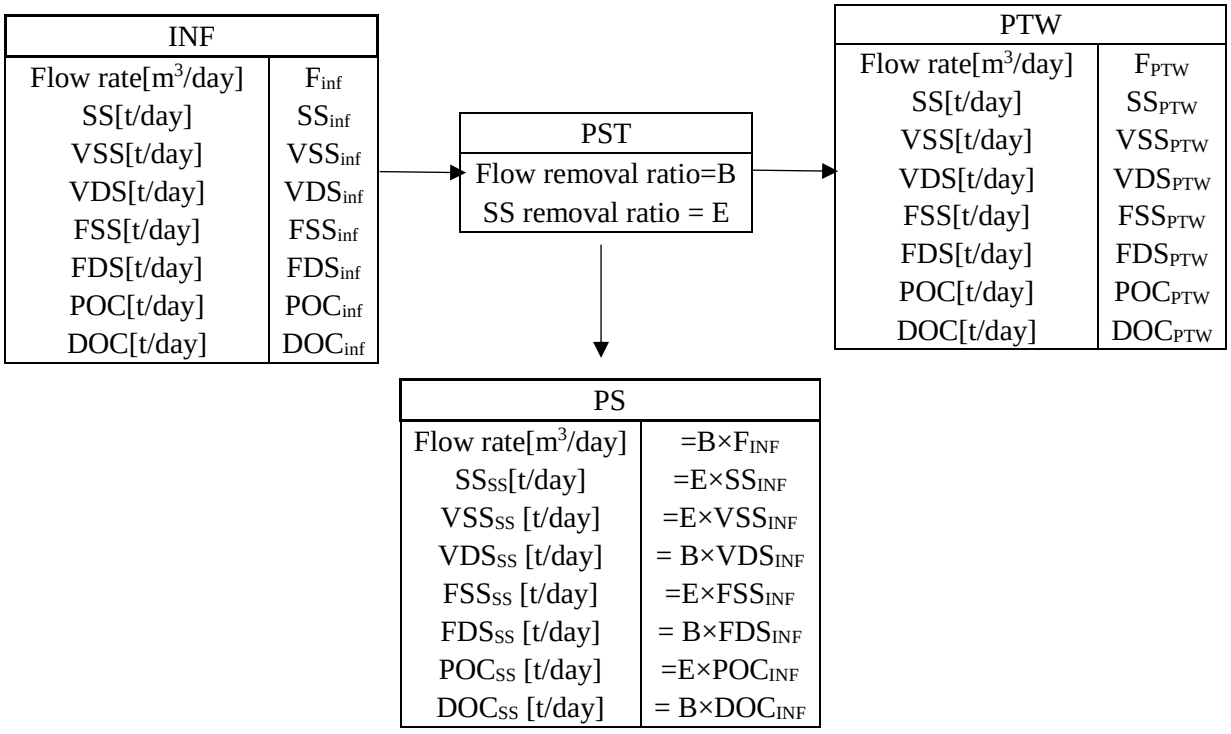


Figure 6-3: Removal of suspended and dissolved particles from the PSP.

6.2.2.2 Aeration and secondary sedimentation tank

As mentioned previously, Toba WWTP uses activated sludge to perform the biological treatment. The VSS of PTW increases during the aeration process, which emits CO_2 as a byproduct. In this study, we assumed that the MLSS remained constant at 1700 mg/L.

Figure 6-4 and Figure 6-5 show the effects of aeration and SST on each of the parameters. The assumed removal ratio of suspended and dissolved particles from the SST were 94% and 1.6%, respectively; these removal ratios are consistent with the rates reported by previous study (Nakamura, 2013).

The secondary sludge concentration (Cr) is given by:

$$Cr=((1+R) \times MLSS-SS)/R \quad (6\text{-}9)$$

where MLSS is the MLSS concentration (1700 mg/L) and R is the WAS return ratio (assumed to be 47%).

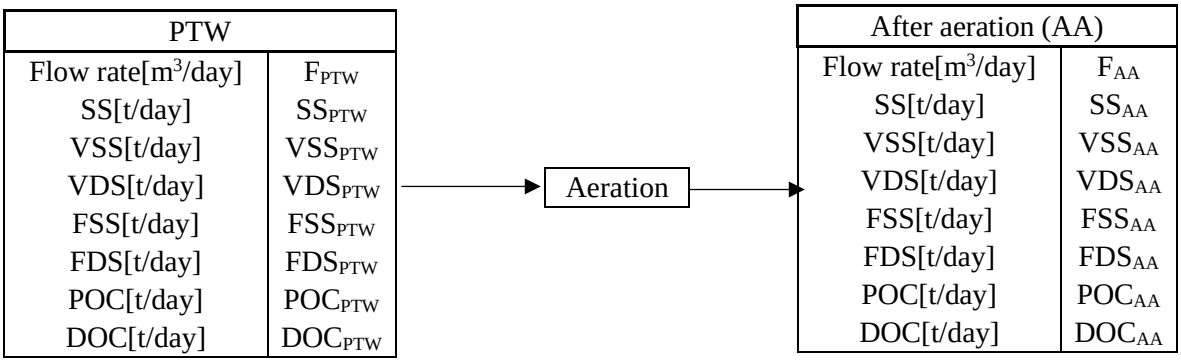


Figure 6-4: parameters change following aeration.

The AA sewage characteristics are calculated using the following:

$$F_{AA}= F_{PTW} \quad (6\text{-}10)$$
$$VSS_{AA}=a\times s\text{-}BOD_{PTW}+b\times VSS_{PTW} \quad (6\text{-}11)$$
$$VDS_{AA}=VDS_{PTW}-(VSS_{AA}-VSS_{PTW}) \quad (6\text{-}12)$$
$$FSS_{AA=} FSS_{PTW} \quad (6\text{-}13)$$
$$FDS_{AA=} FDS_{PTW} \quad (6\text{-}14)$$
$$POC_{AA=}a\times R_B\times DOC_{PTW}+b\times POC_{PTW} \quad (6\text{-}15)$$
$$DOC_{AA=}(1- R_B) \times DOC_{PTW} \quad (6\text{-}16)$$

where a is the VSS production rate from S-BOD (assumed to be 0.55 g-MLSS/g-s-BOD), b is the VSS rate remaining after aeration (assumed to be 0.95 g-MLSS/g-VSS), and S-BOD is 0.83×DOC×32/12. All of these constants and equations were presented in previous work (Nakamura, 2013).

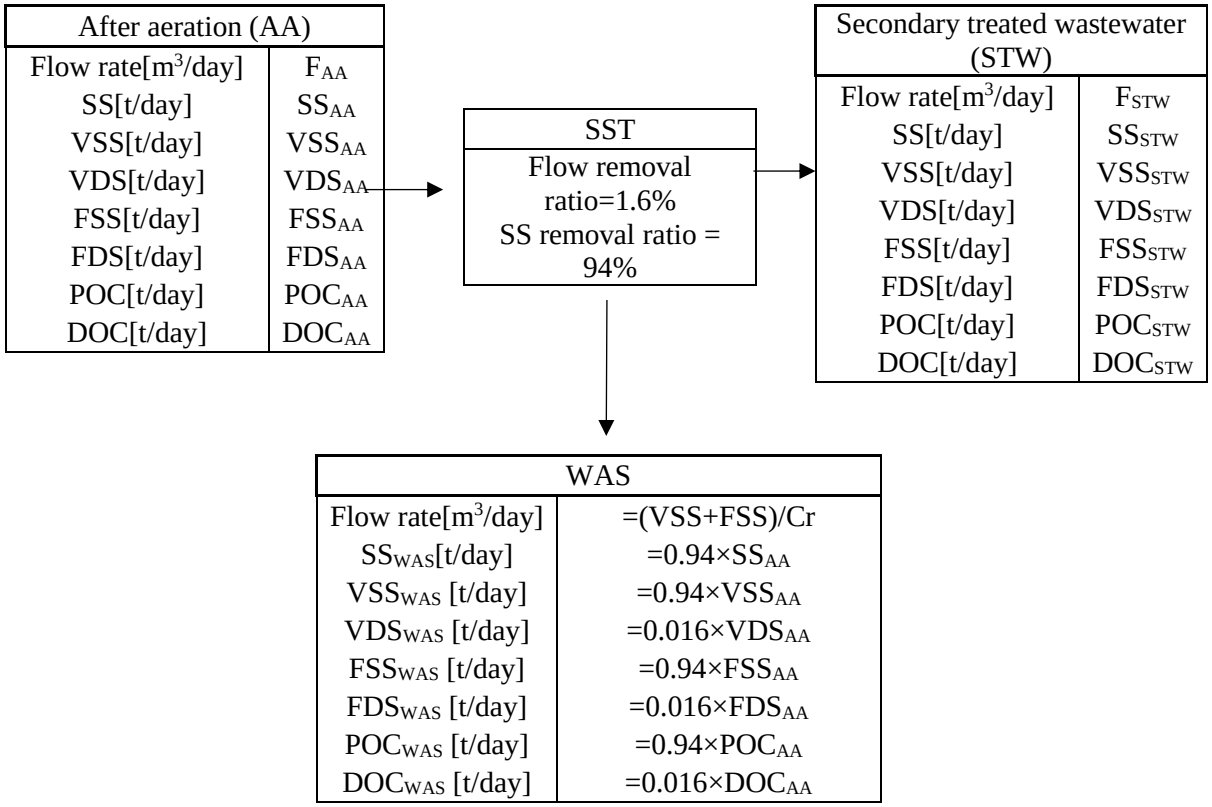


Figure 6-5: Suspended and dissolved parameters removal in SST.

6.2.2.3 Energy consumption and methane fermentation

The aeration power related to biological treatment was obtained using the model of Eckenfelder and O'Connor. The required amount of oxygen in the aeration tank is expressed as follows [all equations and assumptions are based on study conducted by Nakamura (2013)]:

$$AOR = D_B + D_E + D_O \qquad 6\text{-}17$$

Where AOR is the total oxygen demand (kg/day), and DB, DE, and DO are the amounts of oxygen (kg/day) required for organic matter oxidation, endogenous respiration, and maintaining dissolved oxygen, respectively. They can be calculated as follows:

$$D_B = a_{BOD} \times (TB) \times 10^3 \quad \text{6-18}$$
$$D_E = b_{MLVS} \times V_{MLVSS} \times V/10^3 \quad \text{6-19}$$
$$D_O = C_O \times (1 + R) \times F_{AA}/10^3 \quad \text{6-20}$$

where a_{BOD} is oxygen required per kg of BOD removed (assumed to be 0.5 kg-O_2/kg-BOD), TB is the BOD of PTW, b_{MLVS} is oxygen required per kg of MLVSS (assumed to be 0.1 kg-O_2/kg-MLVSS), V_{MLVSS} is ignition loss of MLVSS, V is the volume of aeration tank, C_O is the oxygen concentration in aeration tank and R is WAS return ratio (assumed to be 0.5 mg/L).

The amount of air used for aeration (Q_S) is calculated by Eq. 6-21:

$$Q_S = AOR/(\tau \times O_W \times E_A \times 24 \times 60) \quad \text{6-21}$$

Where τ is the specific mass of air (assumed to be 1.198 kg/m^3), Q_W is the oxygen concentration in air (assumed to be 0.229 kg-O_2/kg-air), and E_A is the oxygen transfer efficiency (assumed to be 0.075).

Finally, the daily electricity required for blower L (kW) is provided by Eq. 6-22.

$$L = \frac{1}{\xi} \times \frac{k}{k-1} \times \frac{Pa_1 \times Q_S}{6120} \times \left\{ (Pa_2/Pa_1)^{\frac{k-1}{k}} - 1 \right\} \quad \text{6-22}$$

where ξ is the adiabatic efficiency (assumed to be 0.650), k is the heat capacity ratio of air (equal to 1.4) and Pa_1 and Pa_2 are the pressures at inlet and exhaust, respectively (assumed to be 10,130 and 16,330 mmaq, respectively).

The methane fermentation ratio ζ_g was 0.611 and 0.500 L/g-VTS-digested for PS and WAS, respectively (Noike *et al.*, 2009). It was also assumed that 61% (B) of volatile

matter would be distracted (Garrido *et al.*, 2013). Therefore, following equation can be used to calculate the methane fermentation V_g (NL/d):

$$V_g = \zeta_g \times B \times VTS \qquad 6\text{-}23$$

6.3 Results and discussion

We now discuss the effect of changing the surface area of the PST on the mass and energy balance of Toba WWTP. We adjusted the surface area by -50, -23, 0, +25, +43 and +100% (double).

6.3.1 Effect of surface area on characteristics of the PSP and PS

We used Eq. 6-5 and 6-7 to calculate, respectively, the removal ratios of the particles and suspended solids in PSP. Table 6-5 shows E for each particle and the SS, HRT, and BOD removal ratios.

Table 6-5: Particle, SS and BOD removal ratio and HRT for different A of PSP.

Diameter (μm)	Change of A (%)					
	-50	-23	0	+25	+43	+100
1~20	15.48%	21.47%	26.01%	31.58%	31.58%	42.11%
20~45	74.53%	89.67%	96.72%	100%	100%	100%
45~75	96.23%	100%	100%	100%	100%	100%
75~150	100%	100%	100%	100%	100%	100%
150~250	100%	100%	100%	100%	100%	100%
250~500	100%	100%	100%	100%	100%	100%
500~1000	100%	100%	100%	100%	100%	100%
1000~2000	100%	100%	100%	100%	100%	100%
E_{SS}	80.36%	83.33%	84.90%	86.32%	86.71%	88.42%
HRT (h)	0.96	1.48	1.92	2.40	2.74	3.84
BOD removal	25.80%	31.07%	34.04%	36.36%	37.65%	40.50%

The removal ratio of particles larger than 45 μm was almost 100% for all cases and that of 1–45 μm was increased with A. Removal ratio of 20–45-μm particles was 100 for A larger than +25% and that of 1–20-μm particles was 15% for the smallest case and 42% for the largest case. E of SS increased with A; however, the difference was small. For the case of A of -50%, E_{SS} decreased to 80 from 85% and increased to 88 when A doubled. Thus, it can be concluded that surface area change has a negligible effect on the E_{SS} of the Toba WWTP.

Table 6-6 shows characteristics of PS regarding surface area change. Equations 6-24 and 6-25 are used for the calculation of volatile solids (%DS) and C (%DS) of PS.

$$V = \frac{VTS(ton/d)}{TS(ton/d)} \quad 6\text{-}24$$

$$C = \frac{TOC(ton/d)}{TS(ton/d)} \quad 6\text{-}25$$

Table 6-6: Effect of varying A on the production of TS, TOC, VTS, and characteristics of PS.

Change of A (%)	TS(ton/d)	TOC(ton/d)	VTS(ton/d)	C(%-DS)	V(%-DS)
-50	3.16	1.19	2.59	38	82
-23	3.28	1.23	2.69	38	82
0	3.33	1.25	2.73	38	82
+25	3.39	1.28	2.78	38	82
+43	3.41	1.28	2.80	38	82
+100	3.47	1.31	2.85	38	82

Although we expected the TS, TOC, and VTS removal ratio, and thus E_{SS}, to be affected by variations in the surface area, the effect was negligible. For example, in the case where A decreased by 50%, the TS production of PS was only reduced by 0.23 ton/d (-6.8%). On the other hand, we observed negligible increase of TS production by 0.14 ton/d (+2.4%) by increasing to +100%. Thus, increasing/decreasing the surface area did not affect TS, TOC, or VTS production of PS. Moreover, according to our calculations, the percentages of C and V were not affected. Finally, we concluded that the characteristics of the PS, including the heating value, were not affected by variations in the surface area.

6.3.2 Effect of the surface area on the characteristics of the PTW, AA, and WAS

The PS is affected by variations in the surface area and, thus, the characteristics of the PTW. Although they seem small, these effects warrant discussion. The recovery of solids from PSP improves when we increase the surface area, thus reducing the load induced by solids on aeration and SST. We expected this to decrease the WAS

production rate. Table 6-7 shows the SS and TOC flow rates (ton/d) of both PTW and AA for each surface area change and Table 6-8 shows the production of TS, TOC, VTS (ton/d) and characteristics of WAS.

Table 6-7: SS and TOC production of PTW and AA for each A.

	PTW		AA	
Change of A (%)	SS (ton/d)	TOC(ton/d)	SS(ton/d)	TOC(ton/d)
-50	0.75	1.36	2.03	0.95
-23	0.64	1.32	1.92	0.9
0	0.59	1.30	1.87	0.88
+25	0.52	1.27	1.81	0.86
+43	0.51	1.27	1.79	0.86
+100	0.44	1.24	1.73	0.83

Table 6-8: Effect of changing A on production of TS, TOC, VTS, and characteristics of WAS.

Change of A (%)	TS(ton/d)	TOC(ton/d)	VTS(ton/d)	C(%DS)	V (%DS)
-50	2.01	0.72	1.81	36	90
-23	1.91	0.68	1.72	36	90
0	1.86	0.66	1.68	36	91
+25	1.81	0.64	1.64	36	91
+43	1.79	0.63	1.62	36	91
+100	1.73	0.61	1.58	35	91

When A changes by 0%, the SS and TOC flows of PTW are 0.59 and 1.30 ton/d, respectively. Low amounts of SS and high amounts of TOC indicate that the DOC is the dominant parameter determining the TOC. These parameters change to 1.87 and 0.88 ton/d, respectively, after aeration. This indicates that the biological treatment process decreases the dissolved parameters and increases the suspended parameters.

The SS and TOC flows also depend on the surface area. For example, the SS flow of PTW increases by approximately 30% when the surface area decreases by 50%, but decreases by approximately 25% when it is doubled.

Varying A had little effect on TS, TOC, and VTS production from WAS. Halving the surface area increased the TS flow of WAS by only 7.5%. On the other hand, doubling it decreased the TS flow by 7%. However, the characteristics of WAS (C and V) do not depend on A. We observed the same phenomena in the cases of TOC and VTS flows. Thus, WAS production depends weakly on the surface area, but its characteristics do not.

6.3.3 Effect on daily energy content, energy utilization, and methane fermentation

We calculated the percentage of carbon and volatile matter in PS and WAS for each case. Equation 3-10, HHV (MJ/ton) = 411C (%-DS) +1751, was used to calculate the higher heating values (HHV) of PS and WAS in the dry basis. The daily energy contents (DECs) are calculated as follows:

$$DEC=HHV\times TS/1000 \qquad 6\text{-}26$$

where the units of DEC are GJ/d, HHV is the HHV of the dry TS (J/g), and the units of TS are (ton/d). Table 6-9 shows the DEC of the TS of PS, WAS, and the total DEC for each surface area.

Our results indicate that decreasing or increasing the surface area decreases or increases the DEC of PS, respectively. The total DEC is 88 GJ/d for all cases. Thus, variations in the surface area have a negligible effect on the DEC of PS and WAS.

This may be because, during PSP, the PSD of Toba WWTP does not vary with the surface area.

Table 6-9: DEC of TS of PS and WAS.

	DEC (GJ/d)		
Change of A (%)	PS	WAS	Total
-50	54	33	88
-23	56	31	88
0	57	31	88
+25	58	30	88
+46	59	29	88
+100	60	28	88

Energy is required to oxidize organic matter in aeration tanks. It is obvious that the WWTP consumes more energy when the organic load on the aeration tank increases. We calculated the energy required for aeration using the method developed in a previous study by Nakamura. The results for all cases are shown in Table 6-10 (Nakamura, 2013)

Table 6-10: Energy required for aeration by each case.

Change of A (%)	Aeration energy demand (kwh/d)
-50	4730
-23	4650
0	4600
+25	4560
+46	4540
+100	4500

As expected, the effect of varying the surface area on the energy consumption is negligible. This may be because almost the same mass was recovered in each case. The aeration energy only increases by 3% when the surface area is halved. Thus, decreasing the surface area of the PST by 50% provides an energy efficient strategy for properly removing suspended solids.

Garrido *et al.* illustrated that, from a stoichiometric perspective, 4 kg of COD can be converted into 1 kg of methane (Garrido *et al.*, 2013). It worth noting that methane fermentation is higher in PS than in WAS because the COD of PS is higher than that of WAS. Table 6-11 shows the methane fermentation potential from PS and WAS for all cases. The fermentation ratios were set to 0.611 and 0.500 L/g-VTS for PS and WAS, respectively (Noike *et al.*, 2009). We also assumed that 46% of the volatile matter would be digested (Garrido *et al.*, 2013).

Table 6-11: Methane fermentation of PS and WAS for each case.

	PS		WAS		Total ($L/d \times 10^6$)
Change of A (%)	VTS(ton/d)	Methane fermentation ($L/d \times 10^6$)	VTS(ton/d)	Methane fermentation ($L/d \times 10^6$)	
-50	2.59	0.73	1.81	0.42	45.37
-23	2.69	0.76	1.72	0.40	45.66
0	2.73	0.77	1.68	0.39	45.74
+25	2.78	0.78	1.64	0.38	45.93
+43	2.8	0.79	1.62	0.37	45.98
+100	2.85	0.80	1.58	0.36	46.17

Methane fermentation increases with A, as the PS can be digested easier than WAS. As with our previous results, the differences are small. The methane fermentation

potential of current WWTPs is 45.74 ML/d, but it decreases by 0.8% when A is varied by -50%. The 0.9% increase in methane fermentation when A varies by +100% is negligible.

6.4 Conclusion

In this study, we investigated the effects of varying the surface area on WWTPs, including on the DEC of sludge produced by WWTPs.

We observed that bigger particles were removed more easily in PSP. For each surface area, all particles larger than 45 μm can be removed, and the removal ratio of smaller particles increases as the surface area increases. The removal ratio of suspended solids increases with the surface area. However, the SS removal ratio varied little due to the PSD of the wastewater.

We did not observe variations in surface area to have any specific effects, especially in the case of the DEC of PS and WAS. Energy recovery potential (DEC) of influent is predicted to be 67 GJ/d and approximately 85% of it can be recovered as PS. Recovery ratio doesn't change significantly as surface area changes. This may be because the PSD does not change during each PSP. The DEC only deviated by ±7% when we doubled the surface area from -50% to 100%. Thus, the mass and energy recovery of the Toba WWTP will not be affected by halving the surface area of its PST.

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Chapter 7
Summary and Future Perspective

7.1 General conclusions

Millions of tons of sewage sludge is produced from municipal wastewater treatment plants (MWWTPs) globally each year. In Japan, approximately 2.2 million tons of sewage sludge dry base was produced in 2015. Previous studies have shown that a high proportion of organic matter in sewage sludge, such as that from MWWTPs, enhances heating value. Thus, there is increasing interest in sewage sludge as a viable energy resource.

In this study, a heating value analysis of sewage sludge and suspended solids from MWWTPs was conducted. The analysis indicated that sewage sludge can be utilized as a renewable energy resource. Furthermore, utilization of TG-DTA for volatile matter and heating value analysis of sludge and suspended solids was introduced. This method is especially suitable when limited quantities of samples are available. The main conclusions from each chapter are presented below.

Chapter 2

It is expected that global sewage sludge production will increase in the future, especially in developing countries such as China. Furthermore, sewage sludge contains organic matter that can be utilized as an energy resource. Several studies have indicated that higher quantities of volatile matter or carbon in sewage sludge lead to an increase in the HHV. To date, research has focused mostly on the development of an equation for HHV prediction for coal and MSW. However, the current HHV prediction equations are outdated for application to sewage sludge

from Japan. Previous studies indicated that TG-DTA is a suitable method for evaluation of the heating value and analysis of other parameters for organic materials such as coal. The particle size distribution (PSD), the heating value of each particle range, and the role of particles in the energy balance of wastewater were also identified as important parameters.

Chapter 3

Volatile matter content, organic matter content, and HHV of sewage sludge from Japan have increased over the last 30 years. However, the quality of organic matter (HHV/V) has decreased due to increasing oxygen concentrations. The equation Hh ＝4.186×(50.2V＋308) was proposed for HHV prediction of sewage sludge. This equation is statistically different from that presented 30 years ago. HHV prediction can be achieved by proximate within 2.47%. We conducted multiple regression analysis to predict higher heating value by ultimate analysis. Based on this research, a prediction equation Hh=384×C+101×H+165×N+1210 is suggested for higher heating value prediction that can predict is within 1.41%.

Chapter 4

In this chapter, the feasibility of TG-DTA for estimating the volatile matter content and heating value of sewage sludge was investigated. It was shown that TG-DTA is an appropriate method for volatile matter analysis of sewage sludge. On application, the volatile matter content measured using the sewage testing method and that obtained from the TG curve were in agreement (approximately a 1:1 ratio). The average of the relative error and the standard deviation were 1.02 and 0.62%, respectively. For the DTA curve, two peaks were observed in the temperature ranges of 200–350°C and 350–550°C, demonstrating that digestion affects the peak area

ratio significantly. Finally, the equation $Hl_{DTA}=(Hls_{DTA}/(-0.0101V_{TG}+1.482))$ was proposed for LHV prediction from the DTA peak area. The mean and standard deviation of the correlation error for this equation were 5.2 and 4.07%, respectively, indicating that TG-DTA is a suitable method for the prediction of volatile matter content and LHV.

Chapter 5

The PSD and heating value distribution of the influent, PS, and PTW from two WWTPs in Japan were investigated. The particle size of wastewater decreased during the primary sedimentation process. The PSD played a key role in the heating value distribution in the wastewater. Small particles have the greatest effect on the heating value of suspended solids due to their high abundance in the influent, PS, and PTW compared with other particles. Additionally, the results showed that the PSD has a greater effect than the heating value distribution, because heating values of particles are not as distributed as PSD.

From this, we concluded that approximately 75% of the energy of suspended solids of influent can be recovered by PST, and the remaining is lost as SS of PTW for both WWTPs.

Chapter 6

In this chapter, we investigated the effect of a change in overflow in the primary sedimentation tank on mass recovery and daily energy content of a WWTP. The overflow change was measured from the increase/decrease in surface area. The model results indicated that a change in surface area has no significant effect on the mass and energy balance. For example, it was illustrated that particles bigger than 45 μm can be fully removed, resulting in almost the same mass recovery ratio for all

cases. These results were almost identical for all energy recovery ratios. Results obtained from this and the previous chapter indicate that PSD is the most important parameter for mass and energy balance. Finally, it was concluded that for a WWTP in which the PSD can be recovered easily, the surface area can be considerably diminished without any significant effects on the mass and energy recovery ratio.

7.2 Future perspective

The heating value of sewage sludge and suspended solids from WWTPs was discussed in this study. Further investigations should be developed as follows:

1- Proximate and ultimate analysis of sewage sludge from Japan has been comprehensively conducted. It is strongly recommended that a similar investigation be carried out for other countries, especially developing countries, to evaluate the energy recovery potential of sludge not as adverse waste, but as a renewable energy resource.
2- The PSD of wastewater is a dominant parameter in the primary sedimentation process. It is suggested that the dependence of primary sedimentation efficiency on the PSD is investigated much more comprehensively with further samples under a range of conditions.
3- Finally, energy recovery from a WWTP has a strong correlation with PSD. It is necessary to come up with a new paradigm in which WWTPs are considered not as an energy consumer but as an energy producer. This scenario is possible if an appropriate amount of sludge is produced by physical treatment. Further investigations should evaluate the effect of physical treatments on energy recovery from WWTPs, with a broader consideration of new technologies for physical treatment.

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