



ULTRASONIC PRE-TREATMENT OF WASTEWATER SLUDGE FROM A MEAT PROCESSING INDUSTRY

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Abstract - This investigation deals with ultrasonic disintegration of sludge taken from a meat processing wastewater treatment plant. Different specific energy inputs ranging between 0 and 100 MJ/kg Total Solids (TS) were used in the study. The effect of ultrasonic pre-treatment on anaerobic biodegradability and filterability characteristics of sludge were evaluated using lab-scale experiments. 30 MJ/kg TS of supplied energy is efficient for cell lyses. Ultrasonic pre-treatment significantly enhanced the biodegradability of sludge. Biochemical methane potential test results also supported this result. For 30 MJ/kg TS, 2.4 times higher methane production was achieved compared to raw sludge. Sludge's supernatant characteristics were also affected by the ultrasonic pre-treatment. For 30 MJ/kg TS, oil content, total nitrogen and total phosphorus in the sludge supernatant increased by 3.8 times, 58 times, and 12 times, respectively. The other result is that ultrasonic treatment adversely affected filterability characteristics of sludge even for very low specific energy levels.

Keywords: Anaerobic biodegradability; Biochemical methane potential; Filterability; Floc disintegration; Meat processing wastewater.

INTRODUCTION

Meat industry wastes have a very complex composition and, to optimize the biological treatment conditions, it is necessary to make a thorough analysis of these organic wastes in terms of their different biodegradable fractions and degradation kinetics. Many of these wastes are solids and either not biodegradable or very slow to degrade in biological processes (Rico et al. (2007)). Sludge produced in the treatment units is also a big problem due to the non-bio-degradable fractions and lipid-rich materials contained. Lipid-rich materials have high methane production potential, but their degradation products,

long chain fatty acids (LCFAs), can be inhibitive in high concentrations. LCFA inhibition was long believed to be irreversible, but recent studies have shown the contrary, though recovery takes a long time. Pre-treating organic materials prior to anaerobic digestion aims at enhanced hydrolysis and thus more complete degradation, as bacterial cells are only able to uptake small molecules (Luste et al., 2009). Current technologies for oily sludge treatment, such as solidification, chemical heat treatment, and extraction, are either costly or ineffective. Therefore, oily sludge is not being disposed of properly (Ning et al., 2009).

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Disintegration was developed as pre-treatment of organic materials prior to anaerobic digestion of sludge to enhance hydrolysis and to complete degradation. Ultrasonic energy can be applied as pre-treatment to disintegrate sludge flocks and disrupt bacterial cell walls, and the hydrolysis can be improved, so that the rate of sludge digestion and methane production is improved (Wang et al., 2005). Ultrasound treatment for sludge disintegration results in increase of chemical oxygen demand in the sludge supernatant and size reduction of sludge solids (Tiehm et al., 1997). The ultrasonic process leads to cavitation bubble formation in the liquid phase. These bubbles grow and then violently collapse when they reach a critical size. Cavitation collapse produces intense local heating and high pressure at the liquid-gas interface, turbulence and high shearing phenomena in the liquid phase. Because of the extreme local conditions, $\text{OH}\cdot$, $\text{HO}_2\cdot$, $\text{H}\cdot$ radicals and hydrogen peroxide can be formed. Thus, three mechanisms (hydro-chemical shear forces, thermal decomposition of volatile hydrophobic substances in the sludge, and oxidizing effects of free radicals produced under the ultrasonic radiation) are responsible for the sludge disintegration (Wang, 2005; Riesz et al., 1985; Bougrier et al., 2005). Although the effects of ultrasonic pre-treatment (Show et al., 2007); Pham et al. (2009); Xie et al. (2009); Huan et al. (2009); Erden and Filibeli (2010); u et al., 2011; Anteneh et al., 2013; Zhou et al., 2016; Zielewicz, 2016) and its combinations with other methods (Sahinkaya, 2015; Xinbo et al., 2015; Yeneneh et al., 2015; Kavitha et al., 2016; Yeneneh et al., 2017) on activated sludge disintegration were investigated by several researchers, there are very limited studies on ultrasonic pre-treatment of meat processing sludge (Luste et al., 2009; Ning et al., 2009). The results of previous studies showed that ultrasonic pre-treatment is an effective method for waste activated

sludge processing. To our knowledge, ultrasonic pre-treatment has not been applied to sludge taken from a meat processing wastewater treatment plant.

Even though ultrasonic pretreatments for sludge minimization and biogas production have been studied widely, no previous research has been conducted to test the effectiveness of ultrasonic pretreatment for meat processing wastewater sludge. Therefore, the main objective of this study was to investigate the effect of ultrasonic pretreatment as a physical method for improving anaerobic digestion of meat processing wastewater sludge. Besides, the effect of ultrasonic pre-treatment on filterability characteristics of sludge was evaluated using lab-scale experiments.

MATERIALS AND METHODS

Sludge

Meat processing sludge was sampled from a local meat processing plant in Izmir, Turkey. The samples were taken from the sludge collection basin of the secondary clarifier. The properties of meat processing sludge are given in Table 1. All analysis was done according to procedures given in Standard Methods (APHA 2005) and was repeated three times. The values represent the mean of the measurements.

Ultrasonic pre-treatment

The ultrasonic apparatus was a Sonopuls ultrasonic homogenizer (Bandelin- Sonopuls HD 2200). This apparatus was equipped with a VS 70 T probe with an operating frequency of 20 kHz and a supplied power of 200 W. For each experiment, 500 mL of sludge were filled in a glass beaker without temperature adjustment (no cooling) and the ultrasonic probe was submerged into the sludge-containing beaker to the depth of 2 cm

Table 1. Properties of meat processing sludge.

Parameters	Average Value
pH	7.29
TS (Total Solids Content), %	1.1
VS (Volatile Solids Content), %	76
SS (Suspended Solids Content), mg/L	10520
VSS (Volatile Suspended Solids Content), mg/L	9100
Oil content in the sludge supernatant, mg/L	19478
SCOD (Soluble Chemical Oxygen Demand), mg/L	880
TN (Total Nitrogen in supernatant), mg/L	9
TP (Total Phosphorus in supernatant), mg/L	4.8
Protein, mg/L	2530.6
Capillary Suction Time, s	14.5

above the bottom of the beaker. Specific energy was considered as a main variable parameter for evaluation of the disintegration performance of sludge. Ultrasonic time varied from 1 to 60 min. SE was determined by using ultrasonic power (P), ultrasonic time (t), sample volume (V) and initial total solid concentration (TS_0) according to the following equation (Bougrier et al., 2006):

$$SE \text{ (kJ/kg TS)} = P \text{ (W)} \times t \text{ (s)} / V \text{ (L)} \times TS_0 \text{ (g/L)} \quad (1)$$

Biochemical methane potential (BMP) assay

The effect of ultrasonic pre-treatment on anaerobic biodegradability of meat processing sludge was evaluated based on the BMP assay (Owen et al, 1979). The BMP test was applied to both raw and sonicated samples (S) for comparison purposes. Anaerobic stock culture was used as inoculums (I). The culture was obtained from an upflow anaerobic sludge blanket bioreactor (UASB) unit of Efes Brewery Industry in Izmir, Turkey. The stock basal medium (BM) was prepared in distilled water (Speece, 1996). BMP tests were carried out in 150 mL serum bottles with 60 mL reaction volume. The ratio of basal medium/inoculums/sludge in the BMP test was BM/I/S=0.5/1/1 (v/v). All bottles were purged with 75 % N_2 and 25 % CO_2 containing gas mixture for 3-4 min to obtain anaerobic conditions. Rubber stoppers and screw caps were used to avoid gas leakage from the bottles. The serum bottles were placed in an incubator at constant temperature of 37 ± 2 °C. Methane gas productions were measured daily by the liquid displacement method using 3 % NaOH (w/v) containing distilled water (Razo-Flores et al., 1997). Anaerobic digestion performances of the sludges were evaluated from the methane productions in the bottles. High methane productions in the bottles indicated high anaerobic digestion performances of the sludge samples.

Analytical procedures

The disintegration degree (Muller, 2000) parameter based on soluble COD calculations was considered as the main parameter for evaluation of sludge disintegration. The soluble part of sludge was obtained by centrifugation carried out at 15 000 rpm and 4°C for 20 min. TS and VS were measured according to the procedure given in Standard Methods (APHA, 2005). Total nitrogen (TN) and total phosphorus (TP) in the

sludge supernatant were measured using spectroquant Merck kits numbered 14537, and 00616, respectively, in a NOVA 60 photometer. Particle size distributions of sludge were monitored using a Malvern Mastersizer 2000QM analyzer. Capillary Suction Time (CST) values were analyzed with a Triton A-304 M CST-meter. Volatile fatty acid (VFA) measurements were done using an Agilent 1100 HPLC with C18 column. Extracellular polymeric substances (EPS) were extracted from the samples using the heat extraction technique (Goodwin and Forster, 1985; Frolund et al., 1996).

RESULTS AND DISCUSSION

The effects of ultrasonic treatment on disintegration degree of sludge

The disintegration degree (DD) permits evaluation of the maximum level of sludge solubilization. Increase of DD is determined as the substance that can be readily used to produce methane in the anaerobic digestion (Wang et al., 2005). DD of sonicated sludge increased with increasing specific energy in each experiment (Fig. 1). For specific energy under 10 MJ/kg TS, the disintegration degree was low (3.5 % for 5 MJ/kg TS). Maximum disintegration degree of 47.4 % was obtained for a specific energy of 30 MJ/kg TS. Ultrasonic time was 40 min in this application, so 40 min ultrasonic duration and 30 MJ/kg TS specific energy were considered as optimum for meat processing sludge. Sludge taken from the meat processing wastewater treatment plant required higher SE levels than biological sludge to reach maximum DD value (Bougrier et al., 2005; Erden and Filibeli, 2010). The lower solubilization levels in our study can be attributed to the sludge composition. Sludge taken from the meat processing wastewater treatment plant contains non bio-degradable fractions and lipid-rich materials different from the biological sludge. For specific energies above 30 MJ/kg TS, DD decreased.

Decrease of the DD may be explained by the high oxidation effects of radicals. Up to 30 MJ/kg TS, hydroxyl radicals preferentially attack the organic substances and destroy the activated sludge microorganism's cell walls in biomass and oxidize them to dissolve organic substances and these substances are released to the liquid phase and increase the DD. For specific energies above 30 MJ/kg TS, as SE increases, more soluble organics were mineralized and DD decreased.

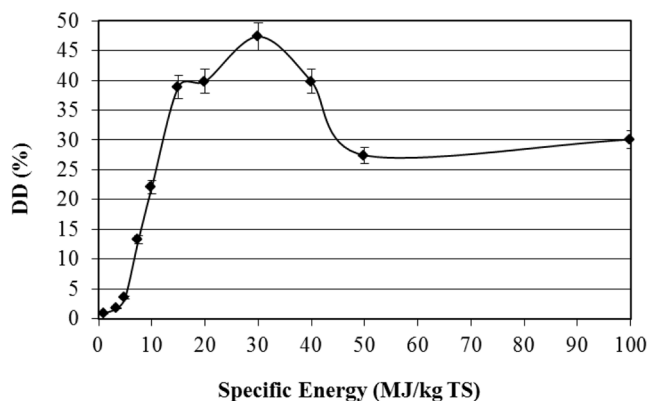


Figure 1. Variation of disintegration degree with specific energy

Changes in physico-chemical characteristics of sludge

Ultrasonic treatment led to a change of the physico-chemical characteristics of sludge. For instance, temperature increased almost linearly with increasing specific energy (Fig. 2). Temperature increased from 20°C in raw sludge to 84°C for ultrasonic pre-treated sludge with maximum specific energy input of 100 MJ/kg TS. The rise of temperature helps ultrasonic disintegration. On the other hand, the high increase in temperature leads to higher saturated vapor pressures, which makes it harder for vapor bubbles to collapse and thus decreases the intensity of cavitation (Huan et al., 2009). One of the reasons for the decreasing DD above 30 MJ/kg TS may be high temperatures. In contrast, pH decreased during ultrasonic pre-treatment (Fig. 2). It decreased from 7.3 in raw sludge to 6.3 for ultrasonic pre-treated sludge at 100 MJ/kg TS application. The reason for the decrease in pH can be explained by the release of fatty acids from biological materials in the sludge after hydrolysis by sonication (Fig. 8). This decrease in pH is within the range that anaerobic organisms can tolerate. Therefore, it can be concluded that sonication will not adversely affect the physico-chemical properties of wastewater; it could even provide improvements which help the operation of anaerobic processes.

Cavitation collapse produces turbulence and high shearing phenomena in the liquid phase. These events disrupt particles and reduce the size during sonication (Gibson et al., 2009). When particles of material in a liquid suspension are subjected to sonication, large particles are exposed to surface erosion or particle size reduction occurs (Kuldiloke et al., 2002) The reduction in particle size generally eases the hydrolysis of solids, since available particle surface area increases for hydrolysis. The result is an accelerated and enhanced degradation of the organic fraction of the solid phase

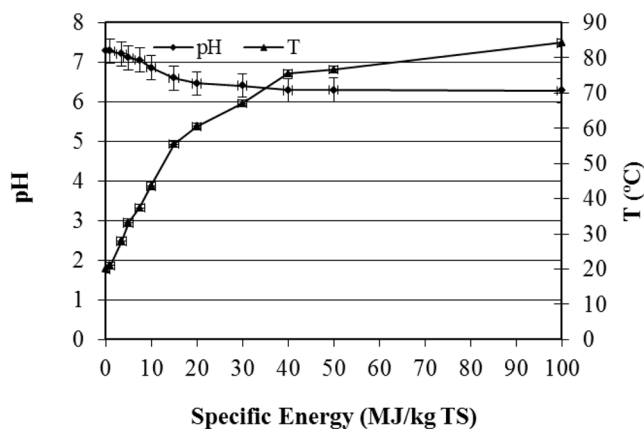


Figure 2. The variation of temperature and pH of sludge with specific energy

Table 2. Particle size changes for different specific energies.

Specific Energy (MJ/kg TS)	Particle Size (µm)				
	Surface weighted mean D[3,2]	Volume Weighted mean D[4,3]	d (0.1)	d (0.5)	d (0.9)
0	71.53	148.58	53.23	118.10	255.03
3.5	10.32	44.62	5.24	22.18	96.14
7.5	8.31	46.35	4.19	17.26	102.62
15	6.05	44.88	2.73	14.48	119.99
30	6.06	46.12	2.73	14.54	122.54
50	6.14	79.30	2.59	22.24	250.52
100	6.04	63.83	2.52	24.31	155.20

(Muller, 2003). Although particle size distribution shows the sludge disintegration, this parameter is not efficient for process optimization (Muller et al., 2004). Table 2 shows the particle size changes for different specific energies, demonstrating that 10 %, 50 %, and 90 % of particles (in volume) having a diameter lower or equal to d (0.1), d (0.5), and d (0.9), respectively. Particle size in sludge was reduced due to ultrasonic pre-treatment and higher reductions were obtained in ultrasonic pre-treated sludge with high specific energies comparing to that in raw sludge.

For 30 MJ/kg TS, a 70 % particle size reduction was recorded based on the volume weighted mean (D[4,3]). Ultrasonic treatment caused formation of many fine particles, which is not beneficial for sludge conditioning; the high CST values were recorded with increasing specific energy up to 15 MJ/kg TS. Then CST values decreased above 15 MJ/kg TS (Fig. 8). High temperature may cause a thermal conditioning effect for sludge. The lower particle size reduction above 30 MJ/kg TS supported this result.

The effects of ultrasonic pre-treatment on supernatant characteristics of sludge

Disintegration causes disruption of microbial cells in the sludge, thereby destroying the cell walls (Vranitzky et al. (2005)). The destruction of floc structure and disruption of cells results in the release of organic sludge components into the liquid phase. These components exist in a dissolved state, e.g., components of intracellular water, or can be liquified (Muller et al., 2004). Ultrasonic pre-treatment was very effective and contributed to the rapid initial increase of nitrogen and phosphorus. As shown in Figure 3, phosphorus and nitrogen were released to the sludge supernatant in a few minutes due to ultrasonic pre-treatment.

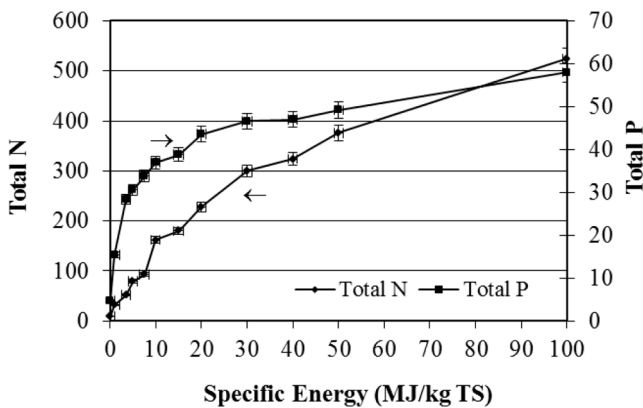


Figure 3. Variation of total N and total P in the sludge supernatant with specific energy

Most of the phosphorus in the supernatant existed in the form of organic products. Nitrogen concentration in the supernatant increased with increasing specific energy. Nitrogen and phosphorus concentrations in the sludge supernatant was increased from 9 mg/L to 524 mg/L and 4.8 mg/L to 58 mg/L for the highest specific energy of 100 MJ/kg TS, respectively.

Oil in the sludge is an important factor which interferes with the anaerobic digestion performance. Ultrasonic treatment may help demulsification of oil and then remove the adverse effect of oil on the anaerobic bio-process (Stack et al., 2005). Therefore, the effect of ultrasonic pre-treatment on oil concentration in the sludge supernatant was investigated. The variations of oil concentrations in the sludge supernatant with SE are depicted in Figure 4. There was a substantial increase in oil concentration when the specific energy was increased up to 40 MJ/kg TS. For higher specific supplied energies, oil removals were nearly constant. Increasing oil concentration in the sludge supernatant may be explained by increasing the sludge solids solubilization with ultrasonic pre-treatment. Oil which is bound to the sludge solids was released to the liquid phase due to solubilization of sludge solids with ultrasonic pre-treatment.

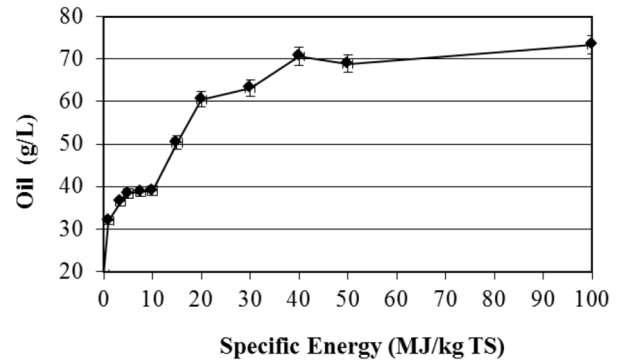


Figure 4. Variation of oil in the sludge supernatant with specific energy

The effects of ultrasonic pre-treatment on anaerobic sludge bio-processing

The anaerobic digestion process is achieved through several stages: hydrolysis, acidogenesis, and methanogenesis. Hydrolysis is the rate-limiting stage for waste activated sludge degradation (Apples et al., 2008). In order to improve the rate of hydrolysis and anaerobic digestion performance, disintegration was developed as the pre-treatment process of sludge to accelerate the anaerobic digestion and to increase the degree of stabilization (Bougrier et al., 2005). Increase of the stabilization degree of sludge with the disintegration process provides less sludge production, more stable sludge, and more biogas production compared to the classical anaerobic digestion. A BMP assay was carried out to assess the feasibility of using ultrasonic pre-treatment in order to improve the anaerobic biological degradation of biological sludge. The BMP assay, in which cumulative methane production was monitored, was applied to both raw meat processing sludge and pre-treated sludge at the optimum condition based on the DD parameter (30 MJ/kg TS) and at the highest SE application (100 MJ/kg TS) for comparison purposes. Cumulative methane productions in serum bottles were monitored for 40 days (continued until the gas production ceased) and the results show that ultrasonic pre-treatment of meat processing sludge may be a good alternative for improving the anaerobic degradation (Fig. 5). The methane production volume was the highest for 30 MJ/kg TS and it was the lowest for raw sludge. While the methane volume for raw sludge was only 121 mL, the methane volume for sonicated sludge with the specific energy of 30 MJ/kg TS was 287 mL at the end of the 40 days of incubation. 2.4 times higher methane production in sonicated sludge was obtained compared to the raw sludge in this application. For the

highest SE of 100 MJ/kg TS, 2.0 times higher methane production was obtained. Since the disintegration degree obtained at 100 MJ/kg TS is lower than that of 30 MJ/kg TS (Fig. 1), it is acceptable to obtain lower methane production at the highest SE.

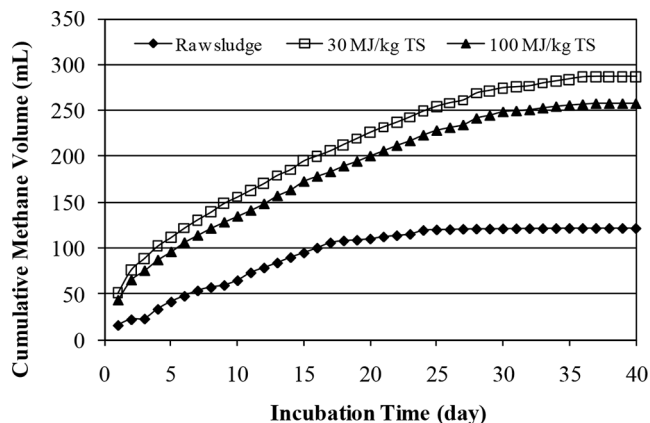


Figure 5. Results of the BMP test

Cell lysis transferring cell content into the medium is the first stage and breakdown of the extracellular polymeric substance (EPS) fraction in the sludge is the second stage of flock disintegration. Cell lysis leads to release of protein content into the liquid phase of sludge in the first stage of disintegration. In the second stage, disintegration enhances the degradation of extracellular polymeric substances (EPS) and the protein content of sludge decreases. Figure 6 shows the variation of protein concentration with SE. There was a slight decrease in protein concentration to 9.9% when specific energy was increased up to 5 MJ/kg TS. Further increase in SE application provided substantially better protein degradation. The highest decrease in protein concentration of 97.5% was obtained for the highest SE application of 100 MJ/kg TS. This value was recorded as 72.5% for optimum condition based on the DD parameter (30 MJ/kg TS of specific energy). The main purpose of disintegration is the elimination of the hydrolysis step to accelerate the anaerobic degradation. Results show that protein hydrolysis was performed successfully by ultrasonic pre-treatment.

Another indicator of the elimination of the hydrolysis step is volatile fatty acid formation in the sludge. Total volatile fatty acid (TVFA) analysis was carried out to assess the feasibility of using ultrasonic pre-treatment in order to improve the anaerobic biological degradation of meat processing sludge. Volatile fatty acid formation was not observed until 20 MJ/kg TS. Further increase in SE application substantially increased TVFA content in the sludge. For the highest SE application, 923 mg/L TVFA was obtained (Fig.7).

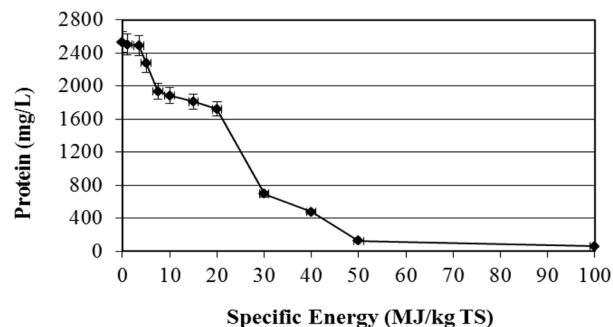


Figure 6. Variation of protein content of sludge with specific energy

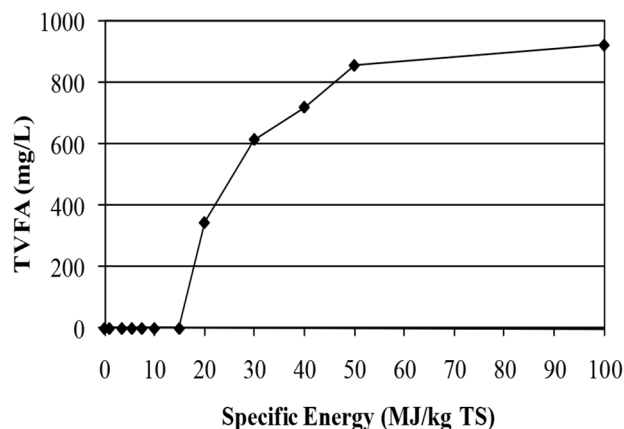


Figure 7. Variation of total volatile fatty acids content of sludge with specific energy

The effects of ultrasonic pre-treatment on sludge filterability

CST is a quick and simple method to evaluate the filterability of sludge. This method neglects the shear effect on sludge, and it cannot determine dewaterability differences between dewatering processes, but gives an approach to the dewatering capacity of sludge (Meeten et al., 1995). CST variations versus applied specific energy are given in Figure 8. CST gradually increased during the ultrasonic pre-treatment. CST results showed that ultrasonic pre-treatment deteriorates the filterability of biological sludge. Sludge filterability was also related to the sludge disintegration degree (Huan et al., 2009; Erden and Filibeli, 2010).

Correlation between CST and DD was examined using the Microsoft-Excel Statistical Correlation function and the correlation coefficient (R^2) was found to have a very high value of 97%. When the sludge disintegration degree was too low, sludge filterability was not significantly affected. When the sludge disintegration degree was high, many fine particles were produced, which led to increasing biosolid

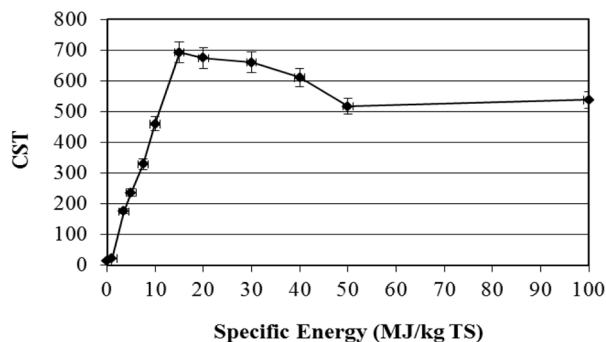


Figure 8. Variation of CST with specific energy

resistance to dewatering. The little decrease in CST above 20 MJ/kg TS may be related to the thermal effect of sonication. Increasing temperature with increasing SE may play a conditioner role for sludge and it led to a decrease in the biosolid resistance to dewatering.

CONCLUSIONS

Sludge taken from a meat processing wastewater treatment plant is either not biodegradable or very slow to degrade in biological processes. This investigation deals with disintegration of meat processing sludge with ultrasonic treatment. The disintegration degree of sludge increased with increasing specific energy. Besides, ultrasonic pre-treatment is an effective method for meat processing sludge disintegration. 30 MJ/kg TS of supplied energy is efficient for sludge solubilization. Ultrasonic pre-treatment led to a change of the physico-chemical characteristics of sludge. For instance, temperature increased and pH decreased gradually with increasing specific energy. Disintegration causes disruption of microbial cells in the sludge, thereby destroying the cell walls. The destruction of floc structure and disruption of cells results in the release of organic sludge components into the liquid phase. Thus, the sludge supernatant characteristics were also affected by the ultrasonic pre-treatment. For 30 MJ/kg TS, oil content, total nitrogen (TN) and total phosphorus (TP) in the sludge supernatant increased by 3.8 times, 58 times, and 12 times, respectively. Biochemical methane potential (BMP) results obtained in this study suggest that ultrasonic pre-treatment led to an increase in the anaerobic biodegradability of biological sludge. 2.4 times higher methane production in sonicated sludge (30 MJ/kg TS) was obtained compared to the raw sludge. Protein and oil hydrolysis was also performed successfully by ultrasonic pre-treatment. Furthermore, CST results showed that ultrasonic pre-treatment deteriorates the filterability of biological sludge. On

the other hand, DD results were in good agreement with CST results. Low disintegration degree in sludge did not significantly affect the filterability. In contrast, when high disintegration degree was achieved, many fine particles were produced and it caused the deterioration of sludge filterability.

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