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DISINTEGRATION OF BIOLOGICAL SLUDGE. A COMPARISON BETWEEN OZONE OXIDATION AND ULTRASONIC PRETREATMENT

The effects of ozone oxidation and ultrasonic pretreatment on anaerobic sludge bio-processing have been studied. Ultrasound (9690 kJ/kg total solids (TS) of specific energy) and ozone (0.1 g O₃/kg TS) were applied to sludge samples preceding anaerobic sludge digestion for disintegration purpose. Organic matter reduction and methane production were measured, as well as physicochemical characteristics (pH, alkalinity, and particles size) and dewatering characteristics of sludge during semi-batch anaerobic digestion. For each treatment, 5 days of sludge retention time were applied on mesophilic conditions for 30 days operation period. The highest volatile solids reduction was obtained with ozone oxidation. Moreover, disintegrated sludge with ozone oxidation and ultrasonic pretreatment provided 55% and 49% higher methane production comparing to the raw sludge, respectively. In terms of dewatering characteristics of digested sludge, ultrasonic treatment led to an increase in the sludge's resistance to dewatering. This negative effect was not observed in ozone oxidation.

1. INTRODUCTION

Disintegration was developed as the pretreatment process of sludge to accelerate the digestion processes. Advanced oxidation techniques such as ultrasonic treatment [1, 2]), ozone oxidation [3] and Fenton process [4, 5] were investigated for sludge disintegration purpose by several authors in half-scale, lab-scale plants and, full-scale facilities. Combination of ozone and ultrasonic pretreatment have also been studied [6, 7]. In the present work, two pretreatment processes have been compared: ozone oxidation and ultrasonic treatment in terms of the effect of anaerobic digestibility of biological sludge.

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Ozone oxidation, one of the commonly used oxidation techniques for wastewater treatment, is a new technique for sludge treatment. Through the implementation of sludge ozonation, refractory organic structures are oxidized and converted into biodegradable low-molecular compounds. Basically, the disintegration process is accomplished by the application of ozone to break down cell walls. Thus, cell walls are fragmented and intracellular compounds are released [8]. Consequently, ozone oxidation of sludge has been considered a pretreatment unit prior to a biodegradation process (aerobic/anaerobic digestion). The previous reports showed that the ozonated sludge can be utilized as a substrate in the anaerobic biological processes [3]. But there are a few studies about the effect of ozone oxidation on aerobic sludge bio-processing. Ozone dose is the key parameter for disintegration. Several authors reported that the optimal range of ozone dose is 0.05–0.1 g O₃/g of dissolved solids (DS) for an increase of sludge's solubilization and enhancement its anaerobic digestibility [3–9]. For ozone dose above 0.1 g O₃/g DS more soluble organics are hydrolyzed and disintegration degree of sludge is decreased [3].

Ultrasonic treatment is another alternative for sludge disintegration. 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 on liquid–gas interface, turbulence and high shearing phenomena in the liquid phase. Because of the extreme local conditions, OH[•], HO₂[•], H[•] radicals and hydrogen peroxide can be formed. Thus, three mechanisms (hydrochemical shear forces, thermal decomposition of volatile hydrophobic substances in the sludge, and oxidizing effect of free radicals produced under the ultrasonic radiation) are responsible for the ultrasonically activated sludge disintegration [10].

Although the increase in temperature with sonication helps to increase disintegration degree of sludge, the effects of temperature are limited about 4% [11]. Mechanisms of the ultrasonic process are influenced by supplied energy, ultrasonic frequency, and nature of the influent. Previous studies showed that low-frequency ultrasound like 20 kHz is very effective in the disintegration of activated sludge [12]. The effects of initial total solids content of sludge, power density, and sonication time on floc disintegration were investigated by several researchers [13, 14]. Previous studies showed that low density and long duration of sonication is more efficient than high density and short duration [11]. It is reported that 9000–10 000 kJ/kg TS specific energy level was efficient for sludge disintegration [15].

The objective of this study was to compare the effects of ozone oxidation and ultrasonic pretreatment of biological sludge on anaerobic digestion performance in the same digestion conditions (mesophilic temperature and 5 days of SRT) and on the same sludge sample. Organic matter reduction and methane production were measured, as well as physicochemical characteristics (pH, alkalinity, and particles size) and dewatering characteristics of sludge during semi-batch anaerobic digestion. For each treatment, 5 days of sludge retention time [16], specific energy of 9690 kJ/kg TS for sonication [15] and, an ozone dose of 0.1 g/kg TS for ozonation [3] were applied.

2. MATERIALS AND METHODS

Biological sludge was sampled from the municipal wastewater treatment plant (WWTP) in Izmir, which has extended aeration activated sludge plant with nutrient removal facilities. At the start-up of the digesters, anaerobic sludge taken from a full-scale up-flow anaerobic sludge blanket (UASB) reactor treating beer industry wastewater, Efes Pilsen Inc. in Turkey, was used as the inoculum for anaerobic digesters. Characteristics of sludges are presented in Table 1. All analyses were done according to procedures given in Standard Methods [17]. All values in Table 1 are the mean of three replicates.

Table 1

Properties of biological sludge and anaerobic inoculum sludge

Parameter	Biological sludge	Anaerobic inoculums sludge
pH	7.06	8.04
Electrical conductivity (EC), $\mu\text{S}/\text{cm}$	7.25	3.4
Total solids (TS), %	1.49	7.5
Volatile solids (VS), %	53.4	84.2
Soluble chemical oxygen demand (SCOD), mg/dm^3	420	1880
Capillary suction time (CST), s	113.5	248.5

In the experiments with ultrasonic treatment, specific energy (SE) of 9690 kJ/kg TS was applied to sludge samples. This value was an optimum value based on disintegration degree parameter. Ultrasonic treatment procedure has been described elsewhere [5]. For evaluation of disintegration performance, disintegration degree (DD) [3, 18] was considered the main parameter. Ultrasonic pretreatment led to a change of physicochemical characteristics of the sludge. The temperature increased from 22 °C in raw sludge to 65 °C for ultrasonic pretreated sludge with specific energy input of 9690 kJ kg/TS [15]. The samples were not cooled after sonication. For the ozone oxidation, ozone dose was 0.1 g O₃/kg TS. The same dose used in another study Erden and Filibeli [3].

For the anaerobic digestion studies, three 8.5 dm³ lab-scale anaerobic digesters were used. One of them was operated as control digester fed with raw sludge, while the others were fed with disintegrated sludge. The digesters were operated at 37±2 °C for 30 days of operation period. The digesters were heated and the temperature was kept constant by means of a heat transfer oil jacket made from stainless steel, operated with PLC. In the reactors, mechanical mixers were used. Based on our previous studies [16], digesters were operated as a semi-batch system with 5 day sludge retention time. The reference digester was coded as R, and the digesters fed with ozonated and sonicated sludge was coded as O and U, respectively. At the start-up phase, 8.5 dm³ of inoculum sludge was first fed to the digesters and the sludge was withdrawn each day till 1/2 volume of the

digester. The same amount of waste activated sludge was fed step by step to the digesters. After start-up of the digesters, 1.7 dm³ of digester content was withdrawn and the same volume of sludge was fed to the digesters every day during the operation period.

For system evaluations, pH and temperature were monitored daily while alkalinity and volatile fatty acids (VFA) were measured three times a week. For performance evaluations, TS, VS, VSS/SS (volatile suspended solids/suspended solids), methane productions and protein contents were measured during the operation period. TS, VS, SS and VSS were regularly determined according to the Standard Methods [17]. Methane production was determined by the liquid displacement method. By this method, gas passes through distilled water containing 3 vol. % of NaOH [19]. Due to the lack of a digital device to measure the amount of gas produced, gas valves of the reactors were first closed for about 1 h and then opened. The liquid displacements were converted to daily productions. Specific methane productions (SMP) were determined as cm³ CH₄/g VS based on volatile solids and daily methane productions. Gas components (CO, CO₂, and H₂S) were analyzed using a Dräger model X-am 7000 multi-gas analyzer. Extracellular polymeric substances (EPS) were extracted from the samples using the heat extraction technique [20]. Protein contents of samples were analyzed using protein assay kits (Procedure No. TP0300 Micro Lowry, Sigma). To evaluate filterability characteristics of digested sludge, CST was analyzed with a Triton A-304 M CST-meter. The belt-press simulator of crown press supplied from Phipps and Bird, Richmond, VA was also used for evaluation of dewatering properties of the sludge. Sludge slurry (200 cm³) was drained through a screen and the volume collected after 2 min was measured. The solids remaining on the screen were then pressed and the final cake solids determined.

3. RESULTS AND DISCUSSION

The disintegration degree (DD) enables evaluation of the highest level of sludge solubilization. Increased DD is determined as the substance can be readily used to produce methane during anaerobic digestion [10]. In our previous studies, the higher DD values were obtained with the ultrasound (9690 kJ/kg TS of specific energy) [15] and ozone (0.1 g O₃/kg TS) [3] applications. Sludge's DD values were determined as 51.1% and 57.9%, respectively. These values are indicators of effective disintegration of sludge and values are close to each other. Disintegration caused changes in some sludge characteristics. The disintegration of the sludge cells is reflected in decreasing SS contents. SS values given in Table 2 showed that ozone oxidation and ultrasonic pretreatment play an important role in destruction and solubilization of sludge particles. Reductions of SS with respect to the raw sludge were 34% and 24% for ozonated sludge and sonicated sludge, respectively. Break down of the floc structure and deterioration of cells results in the release of organic sludge substances into the liquid phase [8]. So,

effective disintegration causes nitrogen and phosphorus increase in sludge's supernatant. Ultrasonic pretreatment and ozonation used for the disintegration process were very effective, leading to an increase of nitrogen and phosphorus in sludge's supernatant.

Table 2

The effect of disintegration processes on sludge characteristics

Parameter	Raw sludge	Ozonated sludge	Sonicated sludge
Soluble chemical oxygen demand (SCOD), mg/dm ³	420	1680	1530
Suspended solids (SS), mg/dm ³	9600	6340	7296
Total nitrogen in sludge's supernatant (TN), mg/dm ³	8	26	65
Total phosphorus in sludge's supernatant (TP), mg/dm ³	40.2	120.6	123.7
DD, %	–	51.1	57.9

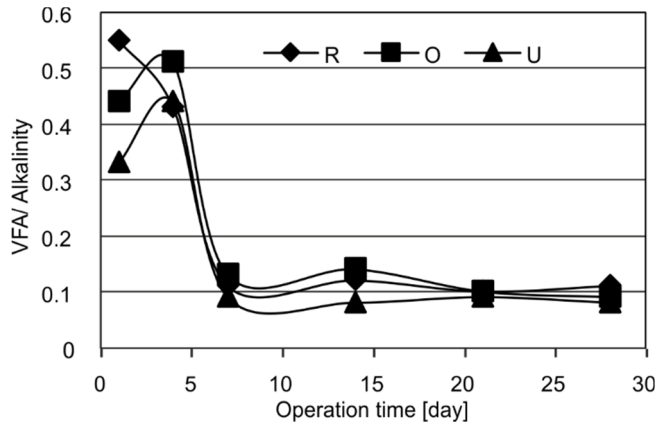


Fig. 1. VFA-to-alkalinity ratio in the digester contents of R, O, and U as a function of operation time

pH varied from 7.02 to 8.63 in digesters. The temperature was kept at 37 ± 2 °C. During the operation period, the alkalinity ranged from 1400 to 4760 mg CaCO₃/dm³. VFA content was also checked, as VFA values exceeding 1500 mg/dm³ are not recommended for anaerobic methanogens [21]. VFA exceeded 1500 mg/dm³ as HAc only in the first operation day, especially in reactors fed with disintegrated sludge. Then the VFA values decreased with increasing operation time. It has been reported that the buffering capacity was sufficient when the VFA-to-alkalinity ratio was maintained below 0.4 [22]. The VFA-to-alkalinity ratios for both digesters contents were monitored to compare the buffering capacities (Fig. 1). In this study, the VFA-to-alkalinity ratios were around 0.08–0.14, with the exception of the first operation days. These indicate that the buffering capacities in the digesters were sufficient for sludge digestion. The contents of gas

components (CO, CO₂, and H₂S) of the digesters were analyzed during the operation to control the digester stability. H₂S content is an important factor for digester stability. Its content in biogas did not exceed the toxic level of 6% for all digesters during the operation period. For the R reactor, the highest H₂S content was determined as 0.07%, these values were 0.11% and 0.13% for O and U reactors, respectively. Disintegration processes led to increase in H₂S levels in the reactors.

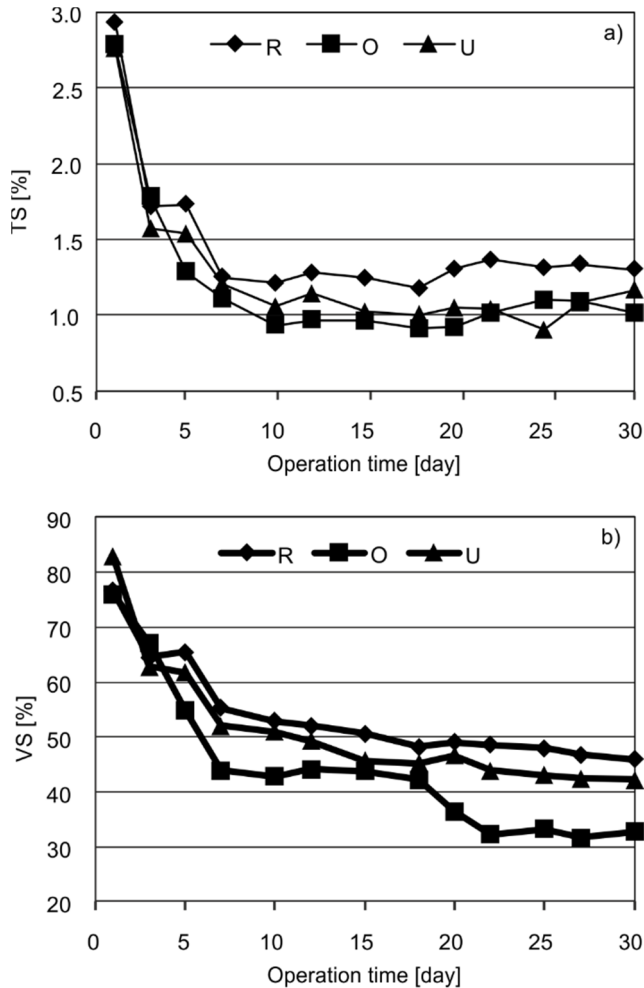


Fig. 2. Sludge' solids in the digester contents of R, O, and U as a function of operation time: a) total solids, b) volatile solids

Time dependences of total solids contents in digesters are shown in Fig. 2a. During the operation, TS values decreased. Disintegration played an important role in the decrease of TS-content and ozone oxidation was more efficient than ultrasonic treatment

in terms of solids reduction. At the end of the 30th operation day, TS values decreased to 12.8%, 32.2%, and 22.1% with reference to the raw sludge for R, O, and U, respectively.

VS values are shown in Fig. 2b. Better VS reductions were observed in disintegrated sludge compared to the reference one. The digester fed with ozonated sludge gave best results. At the end of the 30th operation day, VS values decreased to 14%, 38.2%, and 21.3% for R, O, and U, respectively.

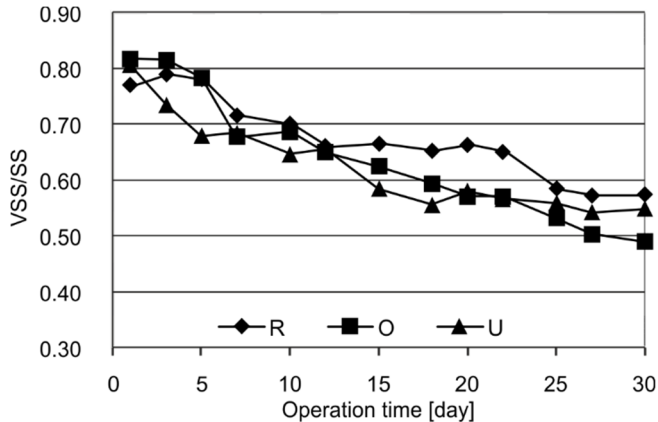


Fig. 3. VSS/SS in the digester contents of R, O, and U as a function of operation time

Microbial activity of bacteria in the sludge can be expressed by the VSS/SS ratio [23]. High VSS/SS ratios indicate that high bacterial activity can be accomplished in the sludge. VSS/SS ratio decreased with increasing operation time during the first fifteen days for all digesters due to decreasing organic matter contents of sludge then stayed relatively constant for each digester (Fig. 3). VSS/SS ratios decreased from 0.77, 0.82, and 0.81 at the first operation day to 0.57, 0.49, and 0.55 at the end of the operation for R, O, and U, respectively. The highest decrease in VSS/SS (40.2%) was observed in the O reactor, the lowest one (26%) in the R one. The other result was the higher VSS/SS ratio was observed in digesters fed with disintegrated sludge compared the R one. This result can be attributed to increasing solubilization degree of organic matter with disintegration processes [3]. Organic matter is oxidized and converted into biodegradable low-molecular compounds by the disintegration processes. The final step of disintegration is disruption of microbial cell and release of intracellular compounds. Hence, the biomass in the biological sludge is converted to substrate which can be easily uptaken by aerobic bacteria in aerobic digestion unit. In this study, the conversion of the biomass to the substrate was confirmed using ozone and ultrasonic disintegration process. According to the VSS/SS values obtained from this study, it can be concluded that level of biodegradability of sludge is increased with ozone oxidation and ultrasonic treatment.

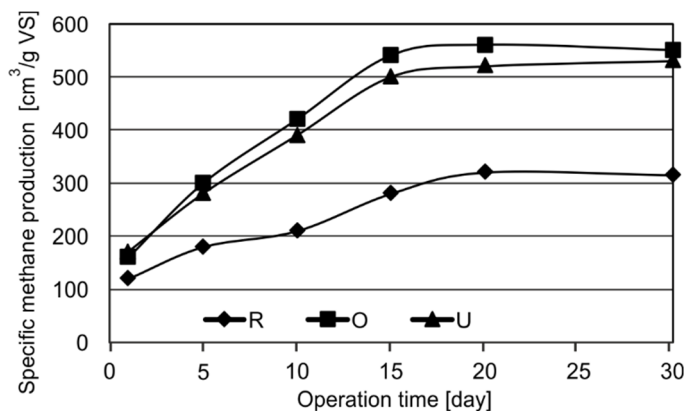


Fig. 4. Methane production in the digester contents of R, O, and U as a function of operation time

Figure 4 shows the specific methane productions during the operation period, which increased upon increasing operation time for the first 15 days, then remaining constant. Reactors fed with disintegrated sludge gave higher methane production rates compared to the control reactors. At the end of the 30 day period, the specific methane productions were 355 cm³, 550 cm³, and 530 cm³ methane/g VS for R, O, and U, respectively. Disintegrated sludge with ozone oxidation and ultrasonic pretreatment provided 55% and 49% higher methane comparing to the raw sludge, respectively, at the end of the 30 day operation period. Besides, disintegration processes did not lead to an increase in H₂S and CO levels in the reactors and H₂S component of biogas content did not exceed 1000 mg/dm³ which is not recommended for anaerobic methanogens reactions for both reactors [24].

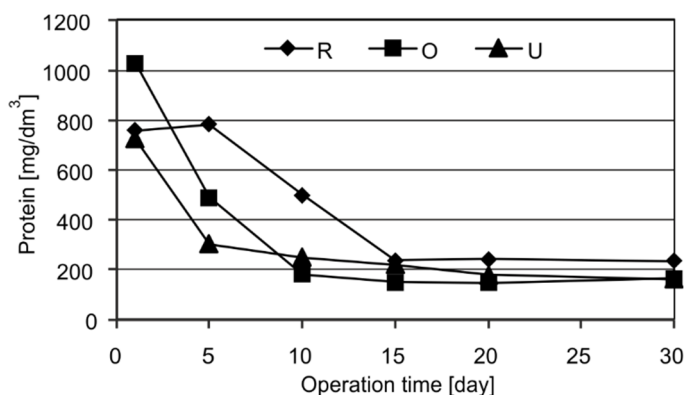


Fig. 5. Protein in the digester contents of R, O, and U as a function of operation time

The main purpose of disintegration is the elimination of hydrolysis to accelerate anaerobic degradation. In the hydrolysis step, protein degradation is achieved. The protein concentrations of digester contents decreased with operation time in all digesters as shown in Fig. 5. Protein was hydrolyzed in the reactors operated with disintegrated sludge faster than the control one. Protein contents decreased with increasing operation time for all digesters during the operation, especially for the first 15 operation days then remained constant. The initial values were 760, 1030, and 725 mg/dm³ and decreased to 236, 165, and 162 mg/dm³ at the end of the operation for R, O, and U, respectively. The highest decrease in protein concentration was achieved in the digester operated with ozonated sludge.

Table 3

Particle sizes for the 1st, 15th and 30th operation days in the digester contents [μm]

Sludge (ID)	Day	Surface weighted mean [3, 2]	Volume weighted mean [4, 3]	$d(0.1)$	$d(0.5)$	$d(0.9)$
R	1st	104.226	636.451	39.202	605.392	1384.721
O		98.801	524.602	39.406	427.091	1201.333
U		75.216	602.159	25.822	582.124	1333.156
R	15th	23.310	183.569	16.427	54.052	788.552
O		24.32	122.067	16.499	59.02	283.737
U		12.827	39.847	11.111	28.300	39.847
R	30th	21.520	141.016	14.318	48.794	181.983
O		22.542	96.052	15.777	50.061	161.351
U		11.080	31.180	9.112	24.138	59.284

The results of particle size analyses given in Table 3 indicate floc disintegration. The particle sizes decreased during the operation period. Particle size distribution shown as $d(0.1)$, $d(0.5)$, and $d(0.9)$ in Table 3 indicates that 10%, 50%, and 90% of particles (in volume) have diameters lower or equal than values given in particular columns. The higher reductions were obtained in digesters fed with disintegrated sludge than in the reference one and the highest reduction was observed in the digester fed with sonicated sludge. The effect of ultrasonic treatment on the particle size can be clearly seen in Table 3. At the end of the operation period, particle size reduced by 77.4%, 81.7%, and 94.9%, for R, O, and U, respectively comparing to first operation day [4, 3].

CST is a rapid and easy method to evaluate the filterability of sludge. This method neglects the shear effect on sludge, and the dewaterability differences among dewatering processes cannot be determined but it gives an idea on the dewatering capacity of sludge [25]. Depending on the CST data, we can say that using ozone oxidation preceding anaerobic sludge digestion has no significant effect on sludge filterability (Fig. 6). A little decrease in CST was observed in the digester fed with ozonated sludge comparing the reference one. On the other hand, ultrasonic treatment preceding anaerobic di-

gestion did not improve CST reduction in digested sludge. CST decreased with increasing operation time for all digester but CST in digester content of U was still higher than raw sludge at the end of the operation. CST of sludge decreased to 83% and 89% according to the raw sludge for R and O, respectively at the end of the 30 days of operation.

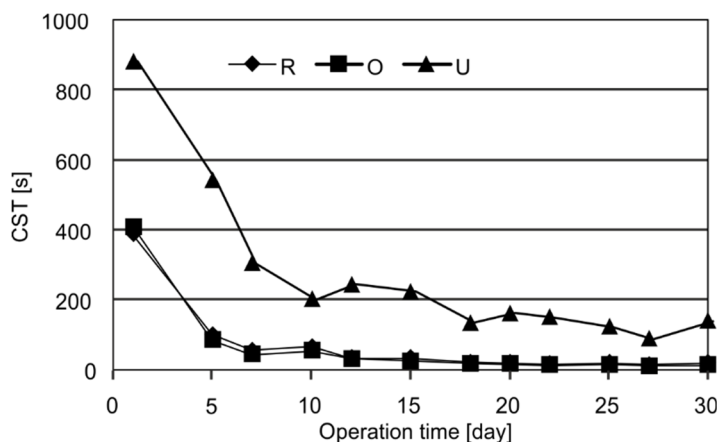


Fig. 6. CST in the digester contents of R, O, and U as a function of operation time

For evaluation dewatering characteristics of digested sludge, a crown press was used as a simulator for the belt press. The digester contents were regularly processed through a crown press during the 30 days of operation period. Final cake solids obtained from crown press application during operation period were summarized in Table 4.

Table 4

Final cake solids obtained from crown press application during the operation period

Reactor's code	Day					
	1st	5th	10th	15th	20th	25th
R	12.85	9.20	9.00	9.02	9.51	6.25
O	13.03	11.36	9.24	no cake formation		
U	9.29	8.95	5.27			

Anaerobic digestion negatively affected the dewatering characteristics of the sludge. In the control digester, while 12.85% cake solids were obtained in the first operation day, 6.25% cake solids were observed at the end of 25th operation day. On the other hand, disintegration processes preceding anaerobic digestion did not improve sludge's cake formation. Cake solids decreased during the operation and they were not obtained after ten days of the operation for O and U. In some studies such as Pilli et al. [4] and Kaynak

and Filibeli [16] it has been reported that the other disintegration method of Fenton pretreatment increases dewaterability characteristics of sludge. But, sonication and ozonation processes have not same effect on sludge dewatering characteristics. These processes are more effective and cause higher DD values for sludge disintegration. The mechanisms of dewatering may be explained by more detailed studies. For example, analysis of EPS content of sludge supernatant may give explanation to understand dewatering mechanism of sludge for these processes. Ozonation and sonication processes may enhance EPS degradation and lead to decrease in the biosolids' resistance to dewatering in reactors.

Both ozonation and ultrasonic pretreatment are the energy-requiring systems. Ozonation requires energy for ozone production (12.5 kWh/kg O₃), and transfer to the sludge (2.5 kWh/kg O₃) [26]. According to this assumption, total energy consumption for ozone oxidation is approximately 15 kWh/kg O₃ [27]. For 0.1 kg O₃/kg DS ozone dose, total energy consumption is approximately 1.5 kWh/kg DS. This value is equal to 5396 kJ/kg DS. So, energy requirement in ozone oxidation is less than the energy requirement in ultrasonic treatment (9690 kJ/kg DS). But for comparison of these two systems, more detailed study is required. In the cost evaluation, additional investment cost must be included as well as cost reduction due to biogas production and less sludge production.

4. CONCLUSIONS

Anaerobic degradability of sludge can be enhanced using ozone oxidation and ultrasonic treatment. With reference to the raw sludge, the highest reduction in volatile solids was 38.2% in the digester fed with ozonated sludge at the end of the operation. The ratios were 21.3% and 14% for the digester fed with sonicated sludge and control digester, respectively. On the other hand, disintegration processes preceding anaerobic digestion increased methane production, and ozone oxidation led to the highest methane production during anaerobic digestion. While ultrasonic treatment led to increasing the sludge's resistance to dewatering, this effect was not observed in ozone oxidation. In addition, disintegration processes reduced the dewatering performance of sludge in mechanical dewatering units.

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