Effects of Low Frequency Ultrasonic Irradition on the Anaerobic Sludge Treatment Process

Düşük Frekanslı Ultrasonik Radyasyonun Anaerobik Çamur Arıtım Sürecine Etkileri

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Öz

Atıksu arıtma tesislerinin işletilmesinde en önemli problemlerden biri aktif çamur sisteminin yönetimidir. Büyük miktardaki çamur sorununu çözmek için son yıllarda çamur minimizasyonu kavramı kapsamlı bir şekilde çalışılmıştır. Sorunun çözümü için kullanılan birkaç geleneksel yöntemlere ek olarak, bazı yeni teknikler de geliştirilme aşamasındadır. Bu tekniklerden biri, insan kulağının duyabileceği frekans aralığının üzerinde geniş bir frekans aralığında üretilen ses dalgaları olarak tanımlanan ultrason yöntemidir. Literatürde küçük kavitasyon baloncukları sayesinde atıksu arıtımında fiziksel, kimyasal ve biyolojik süreçlerin verimliliğinin arttırıldığı gösterilmiştir. Bu çalışmada ultrasonik radyasyonun etkinliğini belirlemek için, farklı ultrasonik yoğunluğa farklı sürelerde maruz bırakılan aktif çamur örnekleri üzerinde pH, toplam katı madde (TKM), toplam uçucu katı madde (TUKM), çözünmüş kimyasal oksijen ihtiyacı (ÇKOİ), diferansiyel çözünmüş kimyasal oksijen ihtiyacı (DÇKOİ) ve reolojik ölçümler yapılmıştır. Örnekler ultrasonik radyasyona maruz bırakıldıktan sonra her 24 saatte bir biyogaz ölçümleri gerçekleştirilerek 25 ve 35 °C'de 2 farklı anaerobik reaktör olarak 30 gün boyunca işletilmiştir. Sonuçlar ultrasonik güç ve sürenin artması ile birlikte ÇKOİ ve DÇKOİ'nin sırasıyla %27 ve 58 oranında arttığını çözünmüş katı madde (ÇK) ve TUK %21 ve 30 oranında azaldığını göstermiştir. Sonuç olarak; ultrasonik kavitasyon etkisiyle anaerobik reaktörlerde TKM, TUKM ve giderimi ile biyogaz oluşumu ham aktif çamurun karakteristiğine bağlı olarak artmıştır.

Anahtar Kelimeler: Anaerobik arıtma, kavitasyon, çamur, ultrases

Abstract

The management of waste activated sludge is one of the most challenges in the operation of wastewater treatment plants. Consequently, the concept of sludge minimization was studied extensively in recent years to solve the problem of the large amount of sludge. Additionally,several conventional methods that were used for the solution of the problem, some new techniques are also under development. One of these techniques is ultrasound, which is defined as sound waves produced in a wide frequency range over the range audible by humans. In the literature, it was demonstrated that the efficiency of physical, chemical, and biological processes of sewage treatment increases with the specific degree of the frequency and dosage of ultrasound by the formation of small cavitation bubbles. The study aimed to investigate changes in the physical properties of waste active sludge and determine the anaerobic decomposition dynamics, following the use of ultrasound irradiation treatment. pH, TS, TVS, DCOD, DDCOD, rheological measurements were conducted for waste activated sludge samples, which were exposed to ultrasonic intensity for different periods, to determine the effectiveness of ultrasonic radiation. After the samples were exposed to ultrasonic radiation, anaerobic batch reactors were operated at two different temperatures, at 25 and 35 °C, for a period of 30 days. Biogas production in reactors were measured every 24 hours on a regular basis. Finally, pH, TS, TVS, DCOD were analyzed to determine the dynamics of the anaerobic decomposition. The results demonstrated that DCOD and DDCOD increased by 58% and 27%, while TS and TVS were decreased by 21 and 30%, respectively as the ultrasonic power and time increased. As a result, removal of TS, TVS and DCOD and the formation of biogas in the anaerobic reactors increased with ultrasonic cavitation, depending on the reactor operating conditions and the character of raw waste activated sludge.

Keywords: Anaerobic treatment, cavitation, sludge, ultrasound

1. INTRODUCTION

Due to its low operating costs, the activated sludge treatment process is the most commonly used biological wastewater treatment method. The process involves various biochemical reactions and biomass conversion mechanisms. These reactions and mechanisms lead to the formation of a highly activated sludge (secondary sludge) (Sahinkaya, 2015). At the end of the process, most of the activated sludge is discharged back into the system (into aeration tanks), while the remainder is conveyed to other units for sludge treatment. The large volume sludge removed from the process, known as waste activated sludge (WAS), contains 99% water (Hall, 1995) therefore, the treatment and disposal of WAS constitutes almost 50% of the operational costs in a wastewater treatment facility (WTF) (Friedler and Pisanty, 2006; Nguyen, et al., 2017; Zhao, and Cheng, 2017). To reduce WAS volume, thickening, aerobic and anaerobic digestion, and composting and dewatering methods are utilized, respectively. Physical properties, such as flow rate, solid content, viscosity, the particle size of the sludge significantly affect the performance of these sludge treatment methods (Ferrasse, and Roche, 2003). One of the most promising methods of reducing sludge volume in WTF is the application of ultrasonic radiation and provides further advantages, such as ease of implementation and adaptability (Zhao, and Cheng, 2017; Civelekoglu et al., 2007; Wang et al., 2014). The ultrasound energy applied to the aqueous environment triggers physical and chemical reactions, which substantially modify the characteristics of dissolved particulate matter in the environment. These reactions occur during the inflation and deflation (contraction) process of cavitation bubbles that are formed due to sound waves (Neis, 2000). The rapid inflation and deflation of gas bubbles due to the ultrasound is called cavitation and, at this stage, very high gas phase temperatures (~5000 ºC) and pressures (~500 bar) are obtained. Cavitation mechanism induced strong cutting forces and jet streams affect the components in the aqueous environment physically, triggering chemical (sonochemical) reactions. These reactions are involved in the formation of two reactive radicals (•H, •OH) and the thermal degradation (pyrolysis) of components $H_2O \rightarrow \bullet OH + \bullet H$ (Nguyen, et al., 2017; Neis, et al., 2000; Moumeni et al., 2012).

Organic matter is dissolved in the activated sludge and became free substrates for microorganisms in the anaerobic process. At this stage, two phenomena should be addressed. First, the application of low-intensity ultrasound breaks up the sludge matrix, dissolving and releasing nucleic acids, fats, humic acids and proteins in the organic matter (Chu et al., 2012). Secondly, the application of high-intensity ultrasound destroys the existing microorganism cells, releasing the cytoplasm within the cell membrane. In turn, the cytoplasm can be used as organic substrate by microorganisms (Neis, et al., 2000). The management of waste activated sludge is one of the most problem in the operation of wastewater treatment plants. Consequently, the concept of sludge minimization was studied extensively in recent years to solve the problem of the large amount of sludge. Additionally,several conventional methods that were used for the solution of the problem, some new techniques are also under development (Edgar et al., 2006). One of these techniques is ultrasound, which is defined as sound waves produced in a wide frequency range over the range audible by humans. In the literature, it was demonstrated that the efficiency of physical, chemical, and biological processes of sewage treatment increases with the specific degree of the frequency and dosage of ultrasound by the formation of small cavitation bubbles. The study aimed to investigate changes in the physical properties of waste active sludge and determine the anaerobic decomposition dynamics, following the use of ultrasound irradiation. pH, TS, TVS, DCOD, DDCOD, rheological measurements were conducted for waste activated sludge samples, which were exposed to ultrasonic intensity for different periods, to determine the effectiveness of ultrasonic radiation. After the samples were exposed to ultrasonic radiation, anaerobic batch reactors were operated at two different temperatures, at 25 ºC and 35 °C, for a period of 30 days. Biogas production in reactors were measured every 24 hours on a regular basis. Finally, pH, TS, TVS, DCOD were analyzed to determine the dynamics of the anaerobic decomposition. The results demonstrated that DCOD and DDCOD increased by 58% and 27%, while TS and TVS were decreased by 21% and 30%, respectively as the ultrasonic power and time increased. As a result, removal of TS, TVS and DCOD and the formation of biogas in the anaerobic reactors increased with ultrasonic cavitation, depending on the reactor operating conditions and the character of raw waste activated sludge.

The anaerobic digestion process is widely employed in WAS stabilization. The process reduces WAS volume, while generating biogas through the removal of pathogenic microorganisms at high rates; however, since the process is slow, the sludge age is old, and the detention period is long, it requires large digestion tanks (Tiehm et al., 2001). To increase the efficiency of the process, pretreatment stages were developed for anaerobic digestion. These include mechanical, chemical and thermal decomposition methods (Salsabil, 2009). Nevertheless, these methods cause the efficiency of acid and methane generation phases after the hydrolysis

to drop, as they create additional sludge (Naddeo, 2009). In the ultrasonic pretreatment process, heat treatment or the addition of any other chemicals are not required. Due to the nature of ultrasonic treatment itself, the sonochemical reactions and the pyrolysis that occurs at the end of the gas phase, do not require any further processes. The ultrasound process and system optimization are requiried, to speed up the kinetics of, and to increase biogas generation, in the complex and slow anaerobic digestion process.
The objective of the present study is to investigate the optimal process design under different operating conditions by identifying the effects of ultrasonic radiation on WAS decomposition dynamics, biogas generation rates, and anaerobic process kinetics.

2. MATERIAL AND METHODS

2.1. Inoculum sludge and waste activated sludge

Waste activated sludge and inoculum sludge were collected from Isparta Urban Wastewater Treatment Plants transiently from aeration basin recycle line and anaerobic digester tanks, respectively and kept at 4ºC before use to preserve their sludge property and microbial activity.

2.2. Ultrasonic pretreatment

Sonics VC 750 was used as ultrasonic homogenizer for ultrasound application. It produces a maximum power of 750 W at a fixed frequency of 20 kHz. Ultrasonic irradiation time and power density applied to the raw waste activated sludge were determined based on preliminary tests conducted in the laboratory. Also, the volume of raw sludge samples to be subjected to ultrasound was set at 200 ml after preliminary tests. Probe-type ultrasonic homogenizer was applied by dipping 2 cm from the surface of the sample.

2.3. Setup and operation of the batch anaerobic reactors

Anaerobic batch reactors (control and operating) were operated at constant temperatures of 25°C (Test Set 1) and 35°C (Test Set 2). Reactors were stored in a temperature controlled orbital incubator with a horizontal mixing apparatus (Shellab/SI). Batch reactors in laboratory environment (operation and control reactors) were established to take samples from the gas phase only and they were operated at an endless sludge age. Thus, 500 ml volumetric socket glass flasks were used. Appropriately sized (N/S 29/32) silicone plugs were used to close the flask lids to ensure anaerobic environment thus to prevent air exposure. Leak proof chemical materials, such as silicon and glass adhesives, were used around the silicon plugs to prevent the biogas leakage during the operation of the reactor. Oxygen in anaerobic reactors was discharged by 99.95% pure nitrogen in the previously.

Twelve batch reactors were operated in an orbital incubator. Three of these reactors (9, 10 and 11th tests) were center point reactors. They included 20% sonicated sludge and 50 mL inoculum sludge. Control reactor contained a mixture of 200 mL raw WAS and 50 mL inoculum sludge. The other 4 operating reactors (1, 2, 3 and 4th tests) included a mixture of 10% sonicated sludge and 50 mL inoculum sludge. Rest (5, 6, 7 and 8th tests) included a mixture of 100% sonicated sludge and 50 mL inoculum sludge.

2.4. Measurement of the total gas volume

Biogas, which is released as a result of biological activity in reactors, was measured every 24 hours with a pH 2-3 HCI solution with the volume displacement method (APHA, 1995). The HCI solution was used to prevent CO_2 dissolution in the environment.

2.5. Analytical measurements

The analysis of raw WAS include the analysis of chemical oxygen demand (COD), dissolved chemical oxygen demand (DCOD), pH, total solids (TS), total volatile solids (TVS).

$$DDCOD = \frac{DCOD_{US} - DCOD_{Raw}}{DCOD_{NaOH} - DCOD_{Raw}} \quad (1)$$

DDCOD: Degree of COD disintegration
$DCOD_{us}$: The COD ultrasound is supernatant COD of the sonicated sample (mg/L)
$DCOD_{raw}$: COD_{raw} is supernatant COD of the original sample (mg/L), and
$DCOD_{NaOH}$: COD_{NaOH} is the maximum COD release in the supernatant after NaOH digestion (sludge and 0.5 M NaOH, ratio of 1:2 for 22 hour at 22 ^{0}C)

2.6. Physical measurements

Viscosity: Rheological measurements were carried out using a rotational viscometer (Brookfield DV-II+Pro). This device has an apparatus (UL adapter) capable of measuring very low viscosity. Sample temperatures were allowed to return to room temperature during measurement. The device measures apparent (dynamic) viscosity values. Accordingly, all mentioned viscosity values in the text of the study describe the apparent viscosity. This parameter is called "viscosity" in the manuscript, in order to avoid any duplication of the concept. The viscometer rotational axis is 9 long and 2.5 cm in diameter and was at a distance of 0.15 cm from the apparatus reservoir. The device was balanced with the available water balance. The viscometer operation was controlled and the data was collected using Rheocalc V3 1.1 software. Before the rheological experiments, the axis (spindle) was calibrated (auto zero) via the software. These software were run in speed ramp mode that depends on rotation per minute (RPM) and time to stop mode with a time adjusted constant rotation speed based analysis were made. The sample volume was kept constant at 16 mL. In the speed ramp mode, samples were measured once every one minute.

3. CONCLUSIONS AND DISCUSSIONS

3.1. Testing set 1 characterization tests of wastewater sewage sludge samples

The comparative characterization results for raw sludge and sonicated sludge are presented in Table 1. The data denote triad measurement means for each parameter.

Table 1. Waste activated sludge characterization before and after sonication

Parameters	**Raw Sludge**	**5min. 20% Amp.**	**30min. 20% Amp.**	**5min. 100% Amp**	**30 min. 100% Amp.**	**15min. %60 amp.**
Temperature (^{0}C)	17	19.4	19.5	19.5	19.4	19.5
pH	6.72	6.69	6.49	6.62	7.17	7.26
DCOD (mg/L)	3380	36600	36600	55900	62900	46200
TS (mg/L)	13763	12603	12603	12340	10027	9390
TVS (mg/L)	12710	9563	9563	10017	6720	6417
$DCOD_{NAOH}$ (mg/L)	83800	-	-	-	-	-

Raw waste activated sludge was sonicated using an ultrasonic reactor in different durations and frequencies (20% amplitude for 5 minutes, 20% amplitude for 30 minutes, 100% amplitude for 5 minutes, 100% amplitude for 30 minutes). As shown in Table 1, the amount of energy consumed also increased with the duration and power density of ultrasound radiation. Accordingly, DCOD values also increased, while TS and TVS values declined depending on the amount of energy consumed.

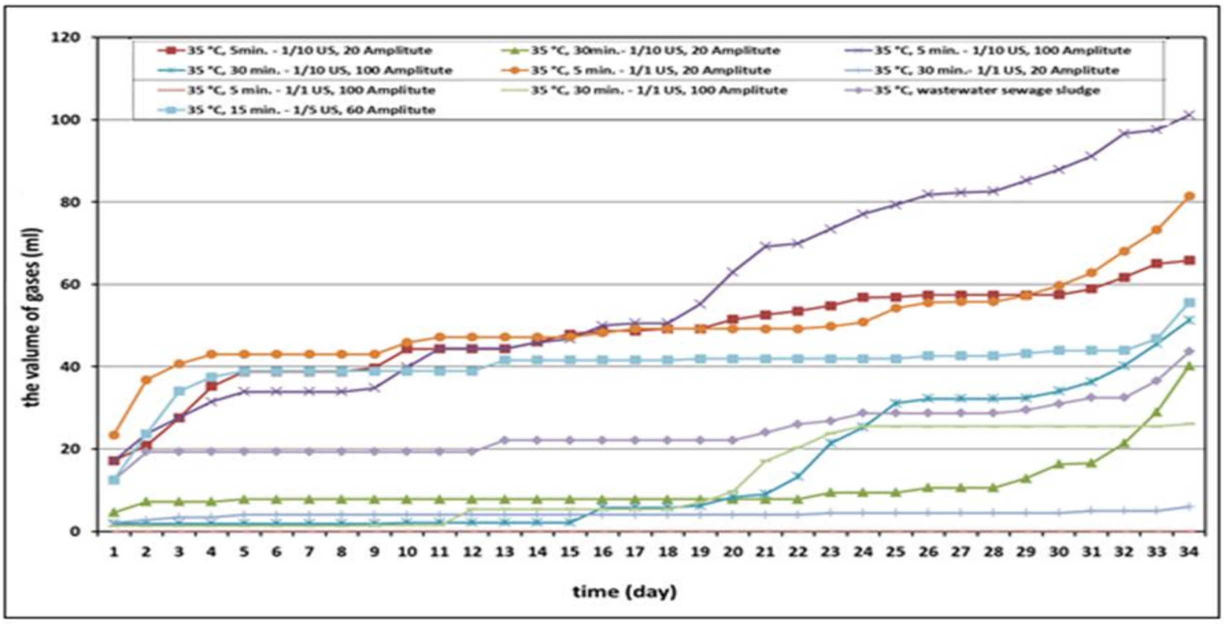

Figure 1. Total valome of gas occurring in the reactor for 34 days

Above figures demonstrated that the volume of biogas formed in the reactors increased over time. However, at the anaerobic reactor, which consisted sludge treated with ultrasound at 100% amplitude for five minutes and inoculation sludge at 1:1 ratio, biogas was not formed for 34 days. It was considered that the metabolic activity of microorganisms, inhibited due to high intensity of ultrasound treatment (100% amplitude), impaired their structure. Thus, the substances within the cells were dissolved in the liquid, which in turn created a ready nutrient media for other microorganisms. Yet, the ultrasonic intensity was maintained only for 5 minutes. It appeared that this duration was not enough for the disintegration (solution) treatment of sludge. The DDCOD (sludge disintegration) rate was very low, for example, 6% (Table 2). Meanwhile, only microorganism inhibition occurred, therefore, hydrolysis, i.e. the most important and the rate limiting step of anaerobic treatment, may not have been completed (Shimizu,1993; Naddeo, 2009).

Microorganisms in the inoculation sludge (not treated with ultrasound) might have consumed their energy for hydrolysis at this step to transform cellular substances into nutrient media. Since a batch reactor was used without any nutritional supplements, it was supposed that the catabolic activities of microorganisms stopped at the outset and consequently biogas was not formed, as the subsequent acidogenic and methanogenic phases did not take place. This was not the case with the anaerobic reactor, since the sludge that underwent disintegration treatment was 1/10 of the total reactor in volume, whereas the rest included inoculation sludge. Thus, an adequate amount of microorganisms in the reactor could sufficiently adapt during the reaction time (34 days) to complete their hydrolysis, acidogenic and methanogenic phases, and hence, generate biogas.

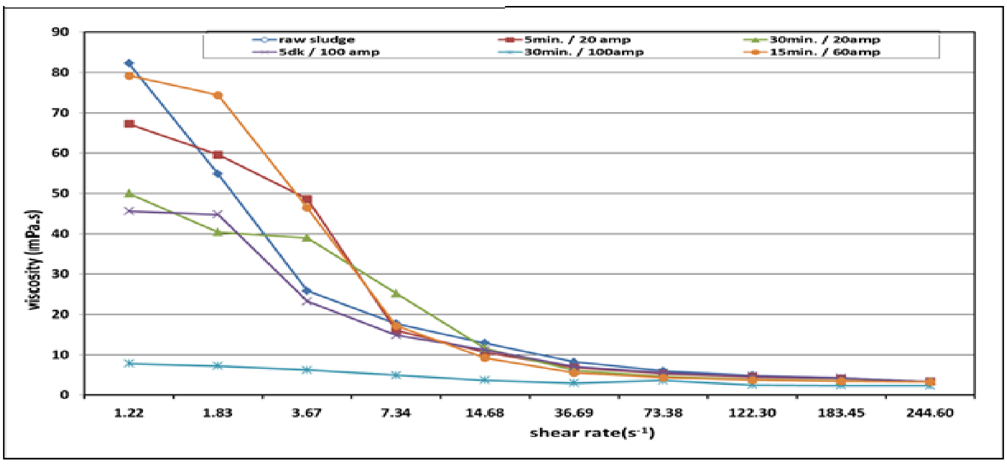

Figure 2. Effect of sonication time on viscosity

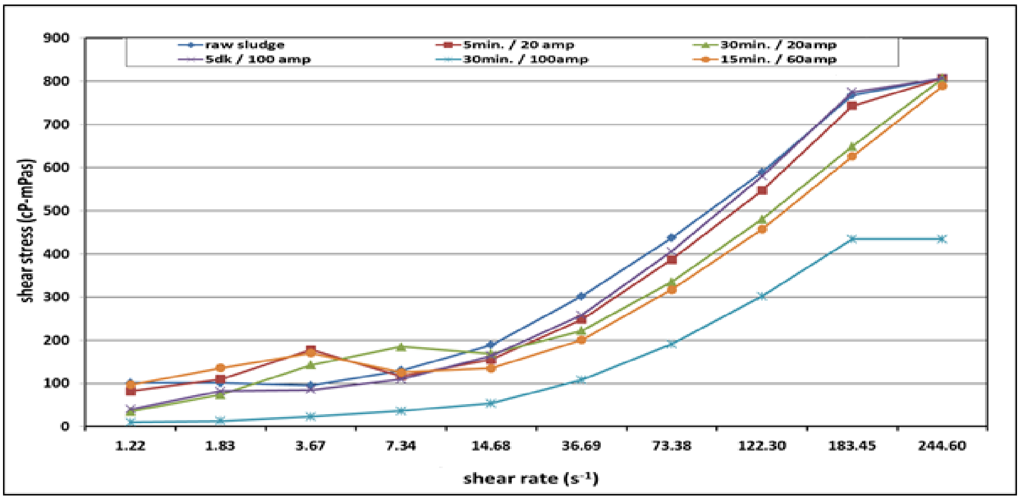

Figure 3. Effect of sonication time on shear stress

The results of physical and chemical analyses conducted on the raw sludge ultrasaound treated samples are given in Table 1. WAS DCOD values increased with the ultrasonic strength and intensity. It was established that ultrasound treatment solubilizes solid materials into particles and flocks. The flocky solid material in raw WAS declined as the strength and intensity increased, and viscosity rates showed a tendency to fall. Table 2 shows TS and TVS removal efficiencies, the increase in DCOD amounts and sludge disintegration efficiencies (DD_{COD}) after ultrasound pretreatment before anaerobic digestion. The findings were consistent with the literature.

Table 2. Disintegration and removal efficiencies after pretreatment with ultrasound before anaerobic digestion

Types of operation	**DCOD (mg/L)**	**TS (mg/L)**	**TVS (mg/L)**	**DDCOD (%)**	**DCOD increase (%)**	**Removal of TS (%)**	**Removal of TVS (%)**
5min. 20% amp. 1/10	36600	12603	9563	6	8	8	25
30min.20% amp. 1/10	59600	10664	7647	52	43	23	40
5min. 100% amp. 1/10	55900	12340	10017	44	40	10	21
30min.100% amp. 1/10	62900	10027	7120	58	46	27	44
5min. 20% amp. 1/1	36600	12603	9563	6	8	8	25
30min.20% amp. 1/1	59600	10664	7647	52	43	23	40
5min. 100% amp. 1/1	55900	12340	10017	44	40	10	21
30 min. 100% amp. 1/1	62900	10027	7120	58	46	27	44
15 min.60% amp. 1/5	46200	9390	6417	25	27	32	50
Raw sludge	33800	13763	12710	-	-	-	-
Inoculation sludge	-	21123	14810	-	-	-	-

It was suggested that cavitation initially reduces the particle size of flocks and, as the duration and intensity of the sonication applied increase, the amount of solid matters decrease since they dissolve. The decrease in total solid matter and total volatile solid matter, together with the increase in chemical oxygen demand, supported this vies.

Based on the literature, TS and TVS removal efficiencies vary. The organic and inorganic content of wastewater affect removal efficiencies. DCOD was increased with the duration and timing of the sonication applied. Literature indicated that the increase in DCOD was limited at low sonication duration and intensity, while it increases rapidly as both increased. Similar results were also observed in the present study. Even with the shortest sonication at the lowest intensity DCOD increased 8%, and as the duration and intensity increased, DCOD values increased as well. The maximum increase in DCOD was 46% at 100% amplitude for 30 min. DCOD increased as the specific energy amount applied on the samples treated with ultrasound increased.DCOD, TS and TVS removal efficiencies after digestion are presented in Table 3.

Table 3. Disintegration and removal efficiencies after pretreatment with ultrasound before anaerobic digestion.

Types of operation	TS (mg/L)	TVS (mg/L)	Removal of DCOD (%)	Removal of TS (%)	Removal of TVS(%)
5min. 20% amp. 1/10	8941	6092	7	29	36
30min.20% amp. 1/10	6749	3630	38	37	53
5min. 100% amp. 1/10	10761	6102	38	13	39
30min.100% amp. 1/10	9842	6683	40	2	6
5min. 20% amp. 1/1	8369	5506	2	34	42
30min.20% amp. 1/1	8386	6960	36	21	9
5min. 100% amp. 1/1	10766	7268	32	13	27
30 min. 100% amp. 1/1	8577	5851	41	14	18
15 min.60% amp. 1/5	8214	5726	23	13	11
Raw sludge	8902	6294	1	35	50

DCOD removal following anaerobic digestion reveals that the hydrolysis phase was exceeded and acidogenic and methanogenic phases were completed. Anaerobic treatment kinetics profiled after digestion are given in Table 4.

Table 4. Anaerobic treatment kinetics

Types of operation	COD Acquisition rate (mg O_2 /Lday)	Spesific COD Acquisition rate (mg COD /gTVS day)	Total Biogas Volume (ml)	Total biogas Acquisition rate (ml/day)	Spesific Total biogas Acquisition rate (ml/gTVS.day)	Total Biogas Acquisition rate (ml /gCOD.day)
5min. 20% amp. 1/10	87	25	66	1.94	0.56	6326.9
30min.20% amp. 1/10	750	187	40	1.18	0.29	446.7
5min. 100% amp. 1/10	707	181	101	2.97	0.76	1192.2
30min.100% amp. 1/10	830	1897	51	1.51	3.45	515.1
5min. 20% amp. 1/1	20	5	81	2.39	0.59	33916.7
30min.20% amp. 1/1	707	1029	6	0.18	0.26	70.8
5min. 100% amp. 1/1	590	215	ND	ND	ND	ND
30 min. 100% amp. 1/1	853	673	26	0.77	0.61	254.9
15 min.60% amp. 1/5	353	512	44	1.29	1.87	1030.7
Raw sludge	13	2	56	1.64	0.26	34750

As Table 4 illustrates, the rate (kinetics) of total gas compositions reached the highest levels (2.97 ml BG/day and 2.39 ml BG/day respectively) at the end of the 3 and 5th set of tests. Only the limitation of the ultrasound application period (5 min.) was common between these tests. Ultrasonic strength and intensity trigger an increase in TVS concentrations, and a decrease in DCOD. Yet, this trend was different in biogas generation. Sonication applied at low intensity, strength and duration accelerated low volume biogas generation. This was, under the specified conditions, due to the uninhibited cellular activities of microorganisms, and acceleration of their biogas generation as they utilize the ready made substrate formed as a result of sonication.

3. Testing Set 2 Characterization Tests of Wastewater Sewage Sludge Samples

The comparative characterization results of raw sludge and sonicated sludge are presented in Table 5. The obtained data denoteed triad measurement mean for each parameter.

Table 5. Waste activated sludge characterization before and after sonication

Parameters	raw sludge	5 min. 20% amp.	30 min. 20% amp.	5 min. 1000% amp.	30 min. 100% amp.
Temperature	17	19.4	19.5	19.5	19.5
pH	6.72	6.69	6.49	6.62	7.26
DCOD	33800	36600	59600	55900	46200
TS	13763	12603	10664	12340	9390
TVS	12710	9563	7647	10017	6417
$DCOD_{NAOH}$	83800	-	-	-	-

As shown in Table 5, the amount of energy consumed increased with the duration and power density of ultrasound radiation. Accordingly, DCOD values also increased, while TS and TVS declined based on the energy consumed.

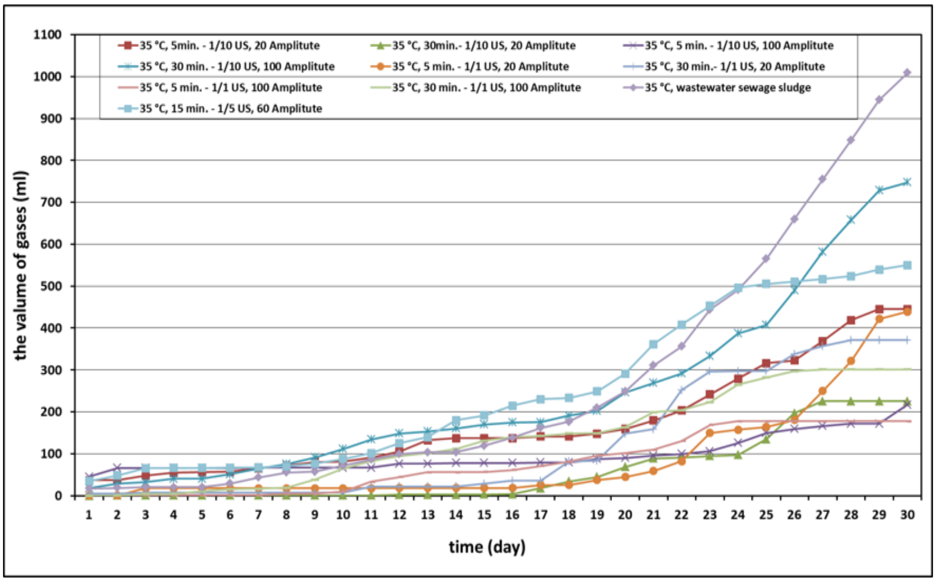

Figure 4. The total volume of gas occuring in the reactor for 30 days

As illustrated in the figures above, the biogas generated in the reactor eventually increased. The largest biogas volume in 30-day cumulative measurements was observed in the reactor with raw WAS and inoculation sludge, but the total gas generated in this reactor in the first 18 days was lower than others. It is likely that the biogas generation increased during the final 12 days in the raw WAS due to the relatively late completion of the hydrolysis phase. In general, the volume of total biogas formed in reactors with 1/10 ultrasound treated samples, raw WAS and inoculation sludge was more than that of the reactors with 1/1 ultrasound treated samples and inoculation sludge. It was suggested that this was due to the decline in the number of active microorganisms in the reactor, since ultrasound treatment destroyed microorganism cell membranes and cytoplasms, similar to the first test set. For instance, while the cumulative biogas volume measured for 30 days was 747.9 ml in the reactor which contained sludge treated with 1/10 ultrasound for 30 minutes at 100% amplitude, it was 301.3 in the reactor that contained sludge treated with 1/1 ultrasound for 30 minutes at 100% amplitude.

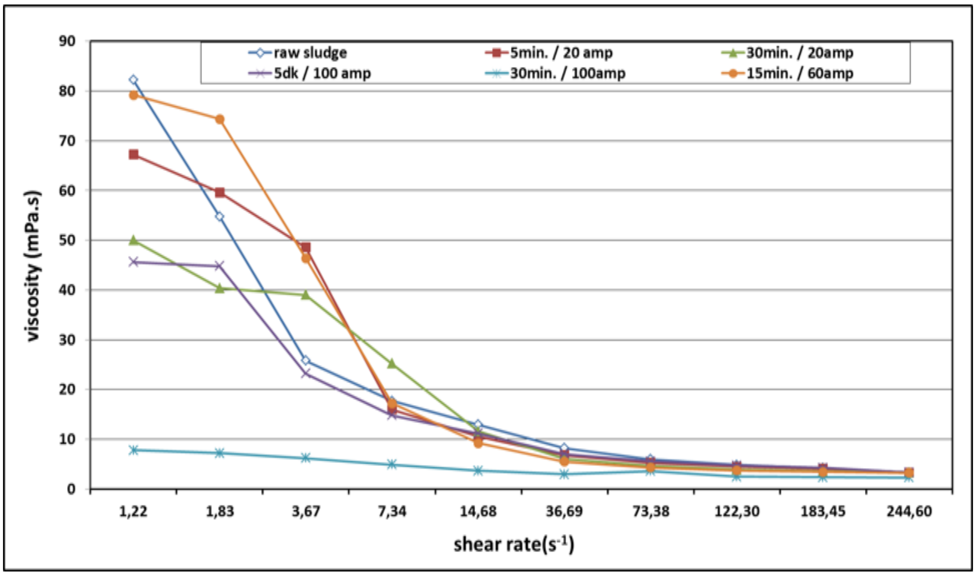

Figure 5. Effect of sonication time on shear stress

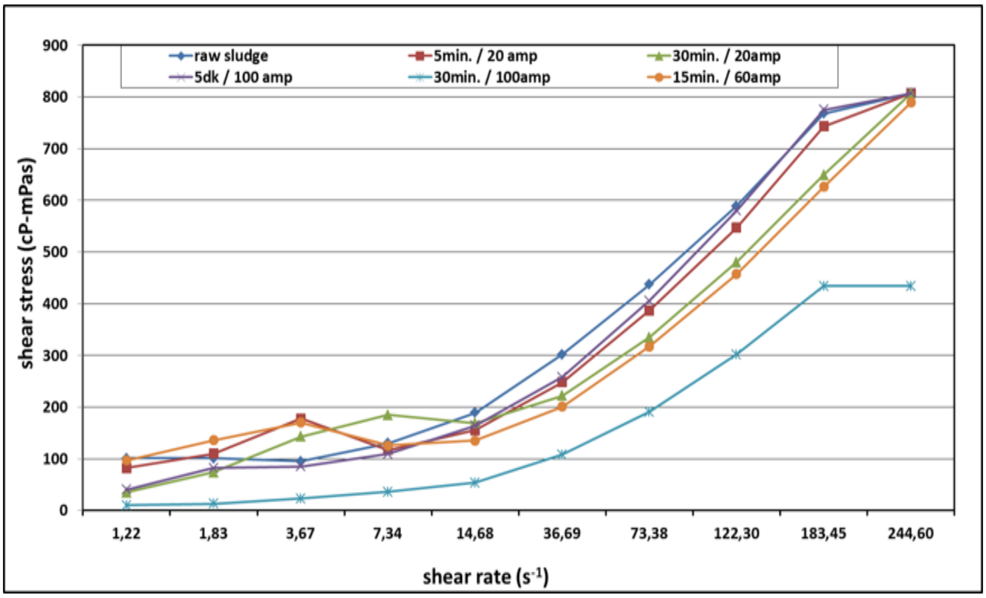

Figure 6 Effect of sonication time on viscosity

As mentioned above, the WAS DCOD increased with ultrasonic strength and intensity. It was discovered that ultrasound treatment solubilized solid material into particles and flocks. Flocky solid material in fresh WAS declined as the strength and intensity increased, and viscosity rates demonstrated a tendency to fall.

Table 6. Disintegration and removal efficiencies after pretreatment with ultrasound before anaerobic digestion.

Types of Reactor	DCOD (mg/L)	TS (mg/L)	TVS (mg/L)	DDCOD (%)	Rise of DCOD	Removal of TS	Removal of TVS
5min. 20% amp. 1/10	2715	11309	3163	2	8	4	5
30min.20% amp. 1/10	3015	10862	2847	4	17	8	14
5min. 100% amp. 1/10	3745	10224	2743.6	10	33	13	17
30min.100% amp. 1/10	5950	9345	2324	27	58	21	30
5min. 20% amp. 1/1	2715	11309	3163	2	8	4	5
30min.20% amp. 1/1	3015	10862	2847	4	17	8	14
5min. 100% amp. 1/1	3745	10224	2743.6	10	33	13	17
30 min. 100% amp. 1/1	5950	9345	2324	27	58	21	30
15 min.60% amp. 1/5	4350	10793	2790	15	42	9	16
Raw sludge	2503	11811	3319	-	-	-	-
Inoculation sludge	9250	9607	3974.7	-	-	-	-

Table 6. shows TS and TVS removal efficiencies, the increase in DCOD and sludge disintegration efficiencies (DDCOD) after pretreatment with ultrasound before anaerobic digestion. Similar to the literature and the first set of tests, ultrasonic radiation duration and ultrasound intensity increased with dissolved chemical oxygen demand, while the quantity of total solid matter decreased. The maximum TS removal efficiency in ultrasound treated sludge at 100% amplitude for 30 minutes in the first set of data was 27%, while TVSM removal efficiency was 44%. In the second set of data, TS removal efficiency was 21% and TVS removal efficiency was 30%. The decline in removal efficiencies were due to the change in sludge content. Similar to the literature and the first set of test, the increase in DCOD was limited at low sonication durations and intensity, but it increased rapidly as the duration and intensity increased. Even with the shortest sonication at the lowest intensity (20% amplitude for five minutes) DCOD increased 8%, and as the duration and intensity increased, DCOD values also increased. The maximum increase in DCOD was 58% at 100% amplitude for 30 minutes.

TS and TVS in fresh WAS decreased as specific energy increased. Maximum removal efficiency for TS was 21%. Similarly, TVS removal efficiency also increased with the specific energy, and maximum removal efficiency was achieved at 30%. In TS and TVS removal, increasing the duration of ultrasonic radiation application was more effective than increasing the ultrasonic intensity. DCOD, TS and TVS removal efficiency after digestion are given in Table 7.

Table 7. Disintegration and removal efficiencies after anaerobic digestion

Types of Reactor	TS (mg/L)	TVS (mg/L)	Removal of DCOD (%)	Removal of TS	Removal of TVS
5min. 20% amp. 1/10	10589	2705	7	6	14
30min.20% amp. 1/10	9910	2563	22	9	10
5min. 100% amp. 1/10	9196	2446	38	10	11
30min.100% amp. 1/10	7558	1440	51	19	38
5min. 20% amp. 1/1	10330	2516	5	9	20
30min.20% amp. 1/1	9622	2452	25	11	14
5min. 100% amp. 1/1	9483	2472	28	7	10

30 min. 100% amp. 1/1	6983	1692	46	25	27
15 min.60% amp. 1/5	10425	2341	29	3	16
Raw sludge	8804	2419	16	25	27

DCOD removal after anaerobic digestion revealed that the hydrolysis phase was exceeded and acidogenic and methanogenic phases were completed. Anaerobic treatment kinetics profiled after digestion are given in Table 8.

Table 8. Anaerobic treatment kinetics

Types of Reactor	**COD Acquisition rate (mg O_2 /Lday)**	**Spesific COD Acquisition rate (mg COD /gTVS day)**	**Total Biogas Volume (ml)**	**Total biogas Acquisition rate (ml/day)**	**Spesific Total biogas Acquisition rate (ml/gTVS.day)**	**Total Biogas Acquisition rate (ml /g.day)**
5min. 20% amp. 1/10	7	14	445	14.83	32.36	561868.7
30min.20% amp. 1/10	22	78	225	7.51	26.48	84736.8
5min. 100% amp. 1/10	48	160	217	7.23	24.27	37937.1
30min.100% amp. 1/10	102	115	748	24.92	28.18	61270.5
5min. 20% amp. 1/1	4	7	439	14.63	22.60	819029.9
30min.20% amp. 1/1	26	65	371	12.37	31.34	121307.2
5min. 100% amp. 1/1	35	127	ND	ND	ND	ND
30 min. 100% amp. 1/1	92	145	301	10.04	15.89	27420.8
15 min.60% amp. 1/5	42	92	550	18.33	40.78	110441.8
Raw sludge	13	15	18	33.63	37.36	11572

*BG: Biogas

ND = Not Detected

As indicated in Table 8, highest total gas generation rate (kinetics) was in the 4th reactor with 24.92 BG/day among ultrasound treated samples in reactor with sludge treated with 1/10 100 amplitude ultrasound for 30 min and inoculation sludge. In the 8th reactor with 1/1 mixture of ultrasound treated sludge for 30 min at 100amplitude and inoculation sludge, the kinetics of total gas generation was 15.89 ml BG/day. Biogas generation kinetics diminished in samples treated with ultrasound at 1/1 ratio as a result of the inhibition of certain microorganisms.

In conclusion, even though it depends on the characteristics of the raw WAS and the operating conditions of the reactor (mesophilic, thermophilic), ultrasonic cavitation triggered COD, TS and TVS removal and biogas generation in anaerobic reactors positively. Although the outcomes were in line with most studies in the literature, some of the test sets yielded deviations and results that were far from expectations (Erden and Filibeli 2010; Filibeli and Erden 2006; Demir, 2016; Gunes and Demir 2016).

4. CONCLUSION

Consistent with the literature, ultrasound treated raw WAS displayed a decline in total solid matter and volatile solid matter and an increase in dissolved chemical oxygen demand as the duration and intensity of ultrasound treatment increased. DCOD increased with the specific energy applied on the samples treated with ultrasound, the increase had a nonlinear trend at low specific energy, i.e. low ultrasonic intensity and sonication time. As the latter increased, the trend eventually linearized. Rheological measurements revealed that flocks were destroyed as a result of ultrasound pretreatment. Thus, ultrasound pretreatment caused macromolecule to micromolecule transformation, which in turn enabled the hydrolysis phase to be completed in a shorter time.

Two anaerobic reactors were set up at 25ºC and 35ºC, respectively. The literature suggests that biogas generation increases with higher ultrasonic power and intensity. Based on pretreatment data, it was assumed that the maximum biogas would be obtained with 30 minutes sonication at 100 amplitude, where maximum solid matter and volatile solid matter removal would be achieved and the increase in chemical oxygen demand would be the highest; however, the findings showed that while biogas formation was at the maximum in the reactor operated at 25ºC for a short time and at low intensity, biogas generation was at maximum in the raw WAS reactor operated at 35ºC. It was suggested that the deviation in biogas generation was due to the inhibition of various microorganisms in the WAS due to the increase in the duration and intensity of the ultrasound treatment. In general, in reactors that contained sludge treated with ultrasound and raw WAS at 1/10 ratio, biogas generation was larger than the reactors with sludge treated with ultrasound at 1/1 ratio. Thus, the above observation verified the assumption that the increase in ultrasonic power, intensity and duration destroyed and inhibited microorganisms. In this study, it was determined that ultrasonic radiation had a positive effect on the activated sludge treatment process. In the future, more comprehensive research could be conducted by applying ultrasonic radiation at different durations, power levels, and intensities and/or by applying different inoculum to sludge ratios to activated sludge, which may provide a solution for activated sludge management systems.

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