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Effects of ultrasound coupled with potassium permanganate pre-treatment of sludge on aerobic digestion

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Abstract. The biodegradability and decomposition efficiencies increase with the pre-treatment of sludge in a digestion process. In this study, the feasibility of ultrasound coupled with potassium permanganate oxidation as a disintegration method and digestibility of aerobic reactor fed with disintegrated sludge with ultrasound coupled potassium permanganate were investigated. The first stage of the study focused on determining the optimum condition for ultrasonic pre-treatment for achieving better destruction efficiency of sludge. The second part of the study, the aerobic digestibility of sludge disintegrated with ultrasound and potassium permanganate oxidation alone and combined were examined comparatively. The results showed that when 20 min of ultrasonic pre-treatment applied, the specific energy output was 49384 kJ/kgTS with disintegration degree of 58.84%. During the operation of aerobic digester, VS/TS ratios of digesters fed with disintegrated sludge decreased indicating that disintegration methods could obviously enhance aerobic digestion performance. The highest reduction in volatile solids was 75% in the digester fed with ultrasound+potassium permanganate disintegrated sludge at the end of the operation compared to digester fed with raw sludge. Total Nitrogen (TN) and Total Phosphorus (TP) levels in sludge supernatant increased with this combined method significantly. Besides, it promoted the production of •OH, thus enhancing the release of Carbon (C), Nitrogen (N) and Phosphorus (P) from the sludge. Disintegration with all methods used in this study could not improve Capillary Suction Time (CST) reduction in disintegrated digesters during the operation. The results demonstrated that the combined ultrasound treatment and potassium permanganate oxidation method improves the biodegradability compared to control reactor or their single application.

Keywords: aerobic digestion; disintegration; ultrasonic treatment; potassium permanganate disintegration

1. Introduction

The activated sludge process generates large amounts of waste activated sludge (Tchobanoglous *et al.* 2003). Rapid industrial growth and unplanned extension of urban zones leads to sludge disposal problem in many developed and developing countries with the increase in the quantity of sewage sludge produced. The sludge must be stabilized because of the pathogenic bacteria content with proper treatment for the environmental protection (Chang *et al.* 2011).

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Anaerobic and aerobic digestions are widely employed in most wastewater treatment plants (WWTPs) to minimize and stabilize excessive sludge production. Anaerobic digestion is commonly used in large scale WWTPs. However long sludge retention time is required for methane production and high operation skill is needed compared to aerobic digestion. In contrast, aerobic digestion is more adequate for small scale WWTPs due to its short sludge retention time requirement. Beside this, the cost of large oxygen demand requirement and the loss of the energy value of the sludge by oxidation to carbon dioxide can be considered as disadvantages (Cho *et al*. 2013). The rate-limiting step of sludge digestion is the hydrolysis of large organic molecules associated with microbial cells. In order to speed up the low rate of biodegradation and enhance the digestibility sludge requires pre-treatment via disintegration method (Chang *et al*. 2011). The disruption of the cell wall and releasing of extracellular and intracellular matter are occurred in the sludge disintegration (Gong *et al*. 2015, Zhang *et al*. 2011). Various sludge disintegration methods have been investigated for pre-treatment including mechanical, thermal, chemical and biological methods such as alkaline treatment (Li *et al*. 2008), thermal treatment (Bougrier *et al*. 2006, Salsabil *et al*. 2010), alkaline combined with thermal hydrolysis (Neyens *et al*. 2003a), ultrasonic treatment (Jin *et al*. 2009), ozone oxidation (Dytczak and Oleszkiewicz 2008), hydrogen peroxide (Neyens *et al*. 2003b) and biological hydrolysis with enzymes (Ucisik and Henze 2008). Ultrasonic methods could overcome the rate-limiting step successfully and organic matter was released from sludge flocs to the aqueous medium after ultrasonic pre-treatment, thus the digestibility and dewaterability of sludge were improved (Gong *et al*. 2015). In ultrasonic treatment, hydro-chemical 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 sludge disintegration

The effects of the initial total solids content of sludge on disintegration must be determined for the optimization of the ultrasound sludge pre-treatment. The higher the sludge concentration leads to the higher efficiency (Gonze *et al*. 2003). The duration and power applied during the pre-treatment as specific energy are the key factors for the effectiveness of the ultrasonication (Gonze *et al*. 2003).

One of the inorganic chemical compound is Potassium permanganate with the chemical formula $KMnO_4$ (https://en.wikipedia.org/wiki/Potassium_permanganate). In so many applications, potassium permanganate is very useful because of the excellent oxidizing property. It is used as an oxidizing agent in many chemical reactions in laboratories and industry. It can be used for water disinfection, toxic matter oxidation, inhibiting the growth of algae as a strong oxidant (Salsabil *et al.* 2009). As an oxidant, potassium permanganate can act as an antiseptic. Potassium permanganate is used extensively in the water treatment industry. It is used as a regeneration chemical to remove iron and hydrogen sulfide. Besides, it is used in the synthesis process of many organic compounds. In wastewater treatment it is used to neutralize hydrogen sulfide (http://www. healthguidances.com/potassium-permanganate). Historically, it was used to disinfect drinking water. (https://en.wikipedia.org/wiki/Potassium_permanganate). In sludge pre-treatment as a disintegration method. $KMnO_4$ presents some advantages than ozonation and chlorination, such as safe, non-toxic and conveniently dosed. However, limited investigation is related to the disintegration of waste activated sludge with $KMnO_4$. But the literature on the effect of sonication on aerobic digestibility of activated sludge is scarcely available and the effectiveness of aerobic digestion after sonication remains unclear (Salsabil *et al*. 2009).

Therefore, in this paper, $KMnO_4$ was used to oxide the extracellular polymeric substances and the aerobic digestibility of sludge was investigated using $KMnO_4$ alone. Beside this, single ultrasound pre-treatment alone are most commonly used for disintegration of sludge. $KMnO_4$. Herein, we also report the aerobic digestibility of sludge pretreated with ultrasound coupled with $KMnO_4$ oxidation. The technique combines the advantages of the mass transfer and cavitation effect of ultrasound and the strong oxidation function of $KMnO_4$ reagent, resulting in improved disintegration of sludge and stabilization efficiency (Gong *et al*. 2015). This study has a novelty in that the aerobic digestibility assisted by combined ultrasonic and potassium permanganate pre-treatment to investigate the synergistic effect of ultrasound pre-treatment with potassium permanganate on aerobic sludge digestion. The results were evaluated with regards to the effect of the pre-treatment methods on solubilization of sludge in terms of volatile solid to total solid ratio (VS/TS), total nitrogen (TN) and total phosphorus (TP) concentration of supernatant were measured during the operation period on semi-batch aerobic digester fed with ultrasonically disintegrated sludge, potassium permanganate disintegrated sludge and combined both of them compared to control reactor fed with raw sludge. Moreover, the dewaterability of sludge was assessed considering capillary suction time (CST) during the operation period.

2. Material and methods

2.1 Sludge sample

Biological sludge samples were taken from the municipal wastewater treatment plant (WWTP) in Elazığ, Turkey. At the start-up of the digesters, aerobic sludge taken from a full-scale aerobic digester of municipal WWTP used as inoculum for aerobic digesters. The sludge sample taken from aeration tank was collected and analyzed immediately for pH, total solids (TS), volatile solids (VS), pH, total COD (CODt), soluble COD (CODs) and total nitrogen (TN) and total phosphorus (TP) in supernatant and presented in Table 1.

2.2 Ultrasound pre-treatment process

The effect of ultrasonic treatment on sludge solubilization was evaluated under different sonication energies. The ultrasonic apparatus was VC 750 Ultrasonic Processor with an operating frequency of 20 kHz. Experiments were carried out without temperature control in a 250 mL glass beaker using 100 mL sludge sample. In the experiments with ultrasonic treatment, specific energy (SE) values varied 12346-74076 kJ/kgTS and vibration times (5, 10 15, 20, 25, 30 min) were

Table 1 Sludge properties

Parameter	Biological sludge
Total Solid (TS) (mg/L)	2430
Volatile Solid (VS) (mg/L)	1764
Total COD (COD_t) (mg/L)	2572
Soluble COD (COD_s) (mg/L)	207
TN (mg/L) (in supernatant)	30
TP (mg/L) (in supernatant)	40
pH	6.8

Table 2 Experimental conditions

Duration (min)	Ultrasonic Power (Watt)	Specific Energy (kJ/kgTS)
5	20	12346
10	20	24692
15	20	37038
20	20	49384
25	20	61730
30	20	74076

applied to sludge samples in order to determine the optimum value. The experimental conditions were presented in Table 2.

The effect of these operating conditions on sludge was investigated by measuring the change in CODs dissolution rate after pre-treatment. The results from these experiments were used to determine the optimum operating conditions of ultrasonic pre-treatment for achieving better destruction efficiency of sludge. All experiments were carried out at ambient temperature and atmospheric pressure.

The sludge dissolution ratio indicates the efficiency of the sludge disintegration. According to the soluble COD results, the experiments were carried out and the disintegration degrees were determined using Eq. (1) in order to optimize the specific energy value

$$DD\ (\%) = (COD_1 - COD_o)/(COD_t - COD_o) \quad (1)$$

Where,

DD (%) is the disintegration degree, COD_1, COD_o and COD_t are the CODs (soluble) of the sludge after ultrasound treatment, raw sludge and total COD, respectively.

SE was determined by using ultrasonic power (*P*), ultrasonic time (*t*), sample volume (*V*) and initial total solid concentration (TS_o) according to the following equation.

$SE = P(W) \times t(s)/V(L) \times TS_o\ (g/L)$

2.3 Potassium permanganate pre-treatment process

A series of bench-scale experiments have been already conducted with a sample volume of approximately 500 mL as a previous study by the author (Demir 2016). In each test, the sludge sample had been preheated to the room temperature. Then the reaction had been initiated immediately by dosing different $KMnO_4$ dosages. The previous study by the author demonstrated that the optimum disintegration degree was achieved with 500 mg/L potassium permanganate for 20 minutes of application time (Demir 2016). Therefore, optimum dose of $KMnO_4$ was added to the sludge and stirred for 20 min. After that, the disintegrated sludge fed to the aerobic digesters.

2.4 Aerobic digestion study

The aerobic digestion studies was carried out using four reactors. One of them was dedicated to the aerobic digestion fed with raw sludge as a control reactor and the others served as disintegrated digesters fed with disintegrated sludge. Air was supplied through and an air compressor to ensure a uniform concentration of 2 mg O_2/L. Each reactor had a working volume of 1.5L. The digesters were operated at 20^{o}C for 30 days of operation period. Sludge retention time (SRT) was 15 days The oxidation reduction potential (ORP) levels were maintained at >100 mV. The digestion system was operated in semi-continuous mode. The fresh sludge sample was fed to the digester and at the same time the equal amount of sludge is withdrawn from digester. A fixed amount of sludge was wasted according to the SRT. The sludge digestion efficiency of each digestion process was monitored by observing the VS reduction at different SRTs.

Control digester was coded as C, and the digester fed with sonicated sludge, disintegrated sludge with $KMnO_4$ and disintegrated sludge with $KMnO_4$+ultrasound were coded as U, P, U+P, respectively.

2.5 Chemical analysis

All COD analyses were done according to procedures given in Standard Methods (APHA 2005). pH measurements were carried out with Hach pH meter. TP and TP in sludge supernatant were measured using spectroquant Merck kits in a NOVA 60 photometer. Sludge supernatant was obtained with centrifugation carried out at 15,000 rpm and 4^{o}C for 20 min.

TS and VS were measured on the total sludge of centrifugation for 15 min. TS and VS concentrations were also measured according to standard methods (APHA 2005). Samples were heated at 105^{o}C for 24 h (determination of the total dry matter concentration) and then heated at 550^{o}C for 1h (determination of mineral matter). Organic matter concentration was then deduced.

3. Results and discussion

3.1 pH variation

pH variations of the operation were presented in Table 3. The SS content of the sludge decreases after the ultrasonication and the EPS contents of the sludge flocs were released into the solution. Consequently, after the ultrasonication, a drop in pH of the solution is observed (Chang *et al*. 2011).

In this study, the pH values were not significantly changed for the reactor fed with disintegrated sludge compared to control reactor. pH values varied from 6,99 to 8.24 in digesters. It can be attributed to the disintegrated sludge volume. According to the sludge age, the waste sludge from the digester was disintegrated ultrasonically and then returned to the digester. The pH drop in the sludge after disintegration was become neutral level after the recirculation to the digester. In this study, the temperature was kept at 20±2^{o}C in all the digesters. The alkalinity and ORP levels were monitored but not presented here. The range of alkalinity 710-2280 mg $CaCO_3$/L were measured during the operation period. ORP values were higher than 150 mV in the first operation days for all digesters. DO was kept above 2 mg/L which is recommended for aerobic digester during the operation.

Şahinkaya (2015) demonstrated that the waste sludge was effectively disintegrated by ultrasonic and ultrasonic-acid pretreatment methods. The sludge disintegration performance of acid pretreatment was negligible at higher pH values than pH 2. However, ultrasonic-acid pretreatment provided the highest sludge disintegration efficiency.

Table 3 pH changes during the operation period

Operation Period, day	pH value	
	C	U
3th	6.99	7.49
6th	8.17	8.03
9th	8.24	7.95
12th	7.88	7.65
15th	7.78	7.56
20th	7.74	7.81
25th	7.75	7.82
30th	7.74	7.8

3.2 Effects of ultrasound pre-treatment on sludge solubilization

The breakage of sludge floc and cell lysis and releases the intracellular organic content to the solution were occurred as a result of the ultrasonic pre-treatment of sludge. Therefore, an increase in the CODs of the solution is obtained at the end of the ultrasonic pre-treatment. Thus, the hydrolysis efficiency of ultrasonication was evaluated considering the CODs concentration. The higher CODs concentration means a better efficiency of ultrasonic vibration and disintegration of the sludge floc and cell wall structure. Therefore, the changes in CODs values are monitored as a function of vibration time.

Fig. 1 presents the changes in the CODs (soluble) concentration and disintegration degree at different sonication times for 750 W energy output. The initial soluble and total COD of sludge was CODs=206 and CODt=2150 mg/L respectively. An increase in the sonication time increases the CODs concentration and the disintegration degree also increases. It was observed that, when the sonication time below 10 minutes, the CODs did not increase significantly. On the other hand, the CODs increased linearly and rapidly from 242 mg/L to 1350 mg/L (for sonication time between 5 and 20 min). Beyond 20 min of vibration time, this ratio did not increase linearly thereafter the CODs increment rate slowed down drastically. It was attributed to that the particulates in sludge were liquidized to soluble fractions or converted into lower molecular weight compounds by ultrasonication treatment. The maximum disintegration degree was 58.84 % for 20 minutes of vibration time.

Gonze *et al*. (2003) found significant increase in the quantity of solubilized COD with the dissipated volumic energy. At 160 kJ/L and in the presence of low load sludge (1.2-3.2 g DS/L), the DD_{COD} is approximately 9-20 %. According to the results of the study by (Salsabil *et al.*, 2009, ultrasonic treatment of sludge at different specific energies: 3600, 31,500, 108,000kJ/kgTS led to solubilization of matter. TS, VS, Total Nitrogen, and COD solubilization increased with increasing specific energy supplied. The results showed that the improvement of aerobic and anaerobic performances in term of total COD removal was noticeable only for high specific energy (108,000kJ/kgTS). Şahinkaya (2015) investigated the individual and simultaneous effects of acid and ultrasonic pretreatment on the disintegration of municipal waste activated sludge. While the optimum pretreatment conditions were the ultrasonic power density of 1 W/mL and pretreatment time of 10 min in ultrasonic pretreatment; the optimum conditions of ultrasonic-acid pretreatment

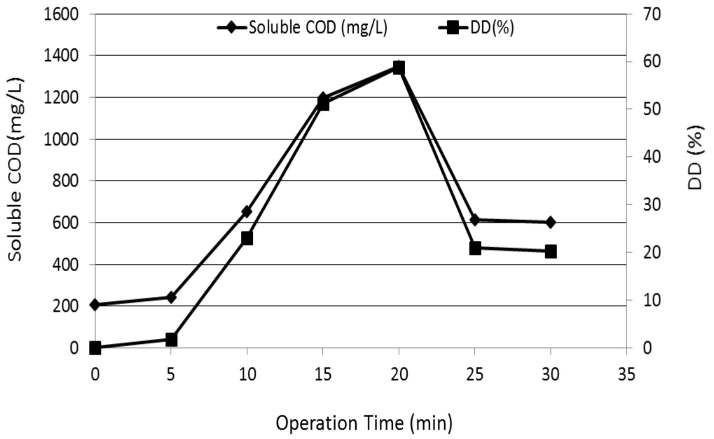

Fig. 1 Optimum specific energy value

were the ultrasonic power density of 1 W/mL and pretreatment time of 10 min and initial sludge pH of 2.0. Under these conditions, about 30% and 40% sludge disintegration degrees were obtained by ultrasonic and combined pretreatment. Zhang *et al.* (2007) studied the effect of ultrasound treatment on the solid content of sludge and biological activity, and the increase in the soluble chemical oxygen demand (SCOD), proteins and nucleic acids concentrations during sonication. The results showed that sonication effectively degraded and inactivated the sludge. The SCOD, supernatant proteins and nucleic acids concentrations, and sludge mass reduction and inactivation degrees increased with the sonication time and power density increases. Sonication at 0.5 W/mL for 30 min disintegrated the sludge flocs by 30.1%, reduced the solid mass by 23.9%, and decreased the sludge viability by 95.5%.

Liang *et al.* (2016) reported a combined technique of ultrasound (US) with $KMnO_4$ degradation of aromatic amines in a textile-dyeing sludge. Under the optimal operating conditions of a $KMnO_4$ dosage of 12 mM, an US power density of 1.80 W/cm^3 and pH, the removals of 58.7%, 88.3% and 24.0% were obtained for monocyclic anilines, other forms of aromatic amines, and TOC, respectively. US-$KMnO_4$treatment was proposed as a practical method for the disposal of aromatic amines in textile-dyeing sludge.

3.3 Aerobic digestibility study

3.3.1 Changes of VS/TS

TSS and VSS were usually applied as sludge digestion indicators (Xu *et al.* 2011). Using disintegration processes, organic contents are oxidized and converted into biodegradable compounds. In disintegration proces, the disruption of microbial cell occurs and intracellular compounds release. The biomass is converted to easily uptaken substrate by aerobic bacteria in aerobic digester. In this study, the biomass converted to the substrate using ultrasonic, potassium permanganate and combined (Ultrasonic coupled with potassium permanganate) disintegration process. According to the VS/SS values obtained from this study, it can be concluded that level of biodegradability of sludge is increased with disintegration processes. The SS content of the sludge decrease after the ultrasonication process and the EPS content present within the sludge flocs release into the solution (Chang *et al*. 2011). Fig. 2. shows the effect of with and without

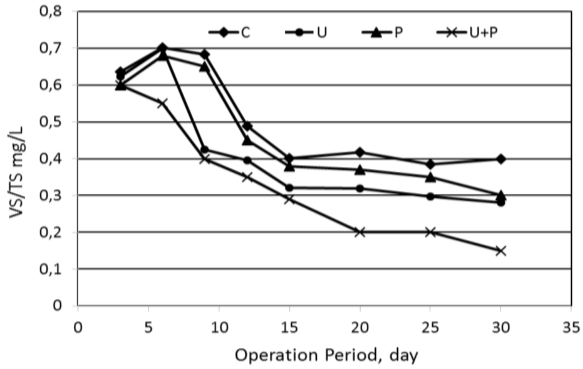

Fig. 2 VS/TS ratio variation during the operation

ultrasonic pretreatment sludge on VS/TS ratio during the operating period. During the operation, VS/TS values decreased for all digesters. Disintegration played an important role in the decrease of VS content and all disintegration methods were efficient in terms of solids reduction. Similar to the study by Chang *et al.* (2011) the results clearly indicate that ultrasonic pre-treatment could fairly reduce the organic matters in sludge. Besides, potassium permanganate alone and combined process (ultrasound+potassium permanganate) could achieve the better sludge reduction and the stabilization. At the end of the 30th operation day, VS/TS ratios of C, U, P, U+P digesters decreased from 0.63, 0.62, 0.6, 0.6 to 0.4, 0.28, 0.3 and 0.15 respectively, indicating that disintegration methods could obviously enhance aerobic digestion performance. In constrast to this study, Salsabil *et al.* (2009) presents that the ultrasonic pre-treatment of activated sludge did not improve the total sludge reduction under aerobic conditions. The improvement of global sludge reduction production of sonicated sludge under aerobic conditions is only due to the ultrasonic treatment itself. According to the study by Şahinkaya (2015), It can be said that the sludge concentration has a critical effect on the sludge disintegration. Under the optimum conditions, thickening of sludge from 0.5% to 1.0% TS content caused an enhancement in the sludge disintegration. However, the sludge disintegration efficiency was reduced at 2.0% TS content of sludge.

3.3.2 Changes of TN and TP during the operation

Microbial cells in the sludge were disrupted by disintegration process. As a results of the destruction of floc structure, organic sludge components were released into the liquid phase (Müller *et al*. 2004). The changes of total nitrogen (TN) in sludge's supernatant are depicted in Fig. 3. The disintegration processes were very effective to release of nitrogen to the liquid phase of sludge. The volume of disintegrated sludge increased in the reactors during the operation so, TN increased gradually with increase in the volume of disintegrated sludge in the reactors. After 20th days of operation, disintegration resulted in the increase of the total nitrogen concentration in the sludge's supernatant for all disintegrated reactors. It is expected that the release of total nitrogen reach to the steady state conditions like organic matter further days of the operation, but we ceased the operation because of the achieving of the organic matter stabilization.

TN concentrations of the digesters were increased from 30 mg/L to 65 mg/L for control digester, from 35 mg/L to 90 mg/L for ultrasonically disintegrated digester (U), from 32 mg/L to 100 mg/L for disintegrated digester with $KMnO_4$ (P) and from 40 mg/L to 125 mg/L for combined

$KMnO_4$ and ultrasonicated digester (U+P) during the operation as shown in Fig. 3.

TP concentrations of the digesters were also increased during the operation from 40 mg/L to 45 mg/L for control digester, from 50 mg/L to 85 mg/L for ultrasonically disintegrated digester (U), from 45 mg/L to 87 mg/L for disintegrated digester with $KMnO_4$ (P) and from 50 mg/L to 102 mg/L for combined $KMnO_4$ and ultrasonicated digester (U+P) as shown in Fig. 4.

Sludge disintegration could be achieved by ultrasonication (U) or $KMnO_4$ (P) oxidation alone. When the combination of U+P was applied, ultrasound enhances the diffusion of $KMnO_4$ providing thorough contact between OH• and organic matter. Thus, the efficiency of sludge disintegration was improved. U+P treatment significantly increases the amount of OH• and enhances the degradation of organic compounds. OH• enhances sludge cell disruption and release of N and P. Results indicate that the disintegration method with higher level of OH• generated also releases higher amount of C, N and P from sludge.

Gong *et al.* (2015) investigated the effects of ultrasound and Fenton reagent in ultrasonic coupling Fenton oxidation (U+F) pre-treatment processes on the disintegration of wastewater treatment plant sludge. Soluble COD, TOC, total N, proteins, total P and PO_4^{3-} concentrations in sludge supernatant increase with U+F treatment significantly. This combined method was more effective than ultrasonic (U) or Fenton oxidation (F) treatment alone. U+F treatment increased the soluble COD by 2.1- and 1.4-fold compared with U and F alone, respectively. U+F treatment increased the total N and P by 1.7- and 2.2-fold, respectively, compared with F alone.

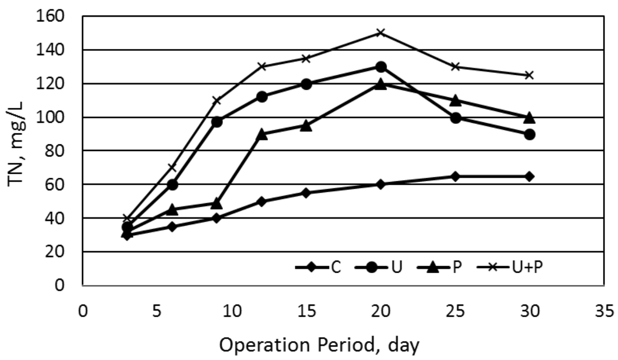

Fig. 3 TN variation during the operation

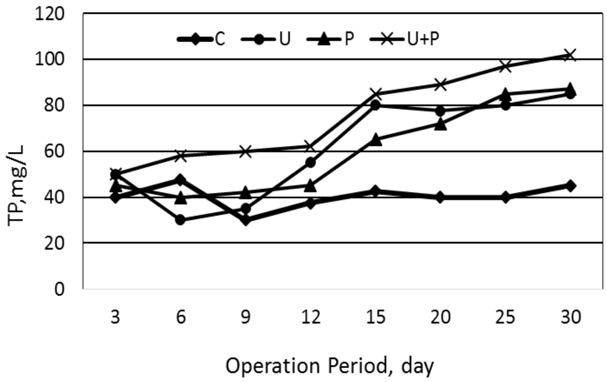

Fig. 4 TP variation during the operation

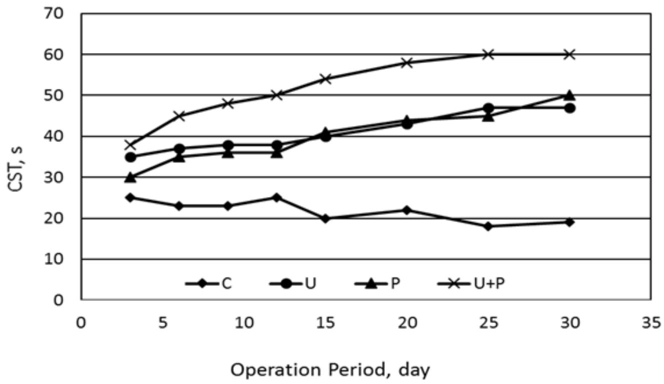

Fig. 5 CST variation during the operation

3.4 Evaluation of dewatering characteristics of digested sludge

Sludge dewatering is very important to reduce sludge bulk and improve its handling properties. Therefore, dewatering followed by digestion is essential in most sludge disposal plants (Appels *et al*. 2008). However, few studies have been conducted to investigate the dewaterability of ultrasonically pretreated sludge after aerobic digestion or the mechanism of the effects of ultrasound pre-treatment on sludge dewaterability after aerobic digestion. The dewaterability effects of disintegrated sludge with $KMnO_4$ for aerobic digestion considering CST has not investigated yet. In order to evaluate the filterability of sludge CST can be used as a rapid and easy method neglecting the shear effect on sludge, and it cannot determine the dewaterability differences among dewatering processes but gives an idea on the dewatering capacity of sludge. For aerobic digestion, disintegration with all methods used in this study could not improve CST reduction in disintegrated digesters during the operation as shown in Fig. 5. CST increased during the operation for all disintegrated digesters. Results of the study by Shao *et al*. (2012) indicated that the dewaterability can be improved by pH 8 and pH 9, but that it deteriorates when pH pretreatment is conducted above 9. In this study, pH was below 8 in a batch experiments for disintegration, thus, CST increased. Moreover, CST increases when the concentrations of protein and carbohydrate fractions increased (Shao *et al*. 2012).

4. Conclusions

• The effects of ultrasonic pre-treatment on the sludge digestion efficiency process were studied, and the optimum condition of ultrasonic pre-treatment was determined.

• The ultrasounds vibration time played an important role in the sludge disintegration process. Nearly 58,84 % DD rate was obtained at 20 min of vibration time and the CODs concentration increased up to 4500 mg/L at 750 W energy output (Es=49384 kJ/kgTS). This condition was selected as the optimum for the digestion process.

• Aerobic degradability of sludge can be enhanced using pre-treatment. In this report, the effects of U, P and U+P treatments on sludge disintegration were examined. According to the raw sludge, the highest reduction in volatile solids was 75% in the digester fed with U+P disintegrated sludge at the end of the operation.

• The U+P treatment significantly increased the soluble TN and TP levels in sludge supernatant. This approach was more effective than U or P treatment alone. Sludge subjected to the U+P treatment showed a considerably thinner and looser microstructure compared to the U and F treatments. Therefore, U+P treatment promoted the production of •OH, thus enhancing the release of C, N and P from the sludge.

• For aerobic digestion, disintegration with all methods used in this study could not improve CST reduction in disintegrated digesters during the operation.

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