Determination on inhibition effects of coagulants used in wastewater treatment plants on anaerobic digester

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ABSTRACT

Domestic wastewaters causing pollution contain inorganic and/or organic materials. When the domestic wastewater outflows to the receiving waters, it causes physical, chemical, and biological pollution in them, and deteriorates the ecological balance of those waters. In the treatment of wastewater, various treatment methods are available depending on the pollution strength of the wastewater. Besides mechanical and biological methods, wastewater treatment with physicochemical methods is still one of the most effective and economical options. Particularly in wastewater with a high concentration of suspended solids, this method is very successful, and obtaining high suspended solids removal efficiencies is very possible. In this study, the effects of the use of coagulant and coagulant aid to be used in a treatment plant where domestic wastewater treatment is carried out are determined to increase the treatment efficiency of a biological treatment that comes later in the stages of the treatment. The effluent of the pre-settling tank may contain a lot of suspended solids. This presence of excess suspended solids decreases the efficiency at other levels of treatment and causes energy loss. In the experiments, the standard jar and inhibition tests are done as a method. As a result of the conducted studies, it is determined that the $FeCl_3$, Synthetic coagulant LP 526, $FeClSO_4$, and the combination of anionic polyelectrolyte yield the best results in the removal of the parameters of chemical oxygen demand (COD), total suspended solids (TSS), and volatile suspended solids (VSS). While $FeCl_3$, APE 65, APE 85, Synthetic coagulant LP 526, and $FeClSO_4$ did not show any inhibition effect in the sludge, APE 67, CPE 84, and $(Al_2(SO_4)_3$ are found to cause inhibition in the sludge.

Key words | anaerobic treatment plant, coagulant, coagulation-flocculation, inhibition

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HIGHLIGHTS

- $FeCl_3$, APE 65, APE 85, Synthetic coagulant LP 526, and $FeClSO_4$ have no inhibition effect.
- $FeCl_3$, Synthetic coagulant LP 526, $FeClSO_4$ are best coagulants for COD, TSS, and VSS removal.
- APE 67, CPE 84, and $(Al_2(SO_4)_3$ have inhibition effect for microorganisms.

INTRODUCTION

In wastewater treatment processes, the coagulation-flocculation is a very important step and often uses synthetic or natural chemicals such as aluminum sulfate, calcium carbonate, synthetic organic polymers and ferric salts (Arslan-Alaton *et al.* 2008; Dotto *et al.* 2019; Hameed *et al.* 2020; Triques *et al.* 2020). The coagulation-flocculation process has been widely used in the past decade for pretreatment in other integrated treatment processes such as membrane filtration and biological treatment (Zahrim *et al.* 2017; Sengul *et. al.* 2018). In the well-designed and

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operated primary sedimentation tanks of the traditional wastewater treatment plants, chemical oxygen demand (COD), turbidity, color, and heavy metals removal efficiencies are high depending on the type of coagulant-flocculant (Tociu *et al.* 2017; Chakraborty *et al.* 2020). However, many of the chemicals used in the coagulation-flocculation process combine with the existing chemicals in wastewater, leading to more total dissolved solids (TDS) in the wastewater (Al-Mutairi 2006; Al-Mutairi *et al.* 2004).

Despite the effectiveness of these chemicals as coagulants, the sludge obtained from processes using synthetic chemicals has negative effects on the environment and human health. It is reported that anionic and nonionic polymers generally have low inhibition, but cationic polyelectrolytes have much higher inhibition, especially for aquatic organisms (Al-Mutairi 2006). Aluminum (Al) is the most abundant metal in the world and dissolves until toxic levels as free acid (3^+) under acidic conditions. It is an environmentally toxic substance and harmful to many organisms (Pan *et al.* 2004). Overdosing of alum in water or wastewater treatment can lead to a high aluminum concentration. Prolonged exposure to water with high aluminum content is associated with serious health problems such as Alzheimer's disease and senile dementia (Yin 2010; Freitas *et al.* 2015; Vunain *et al.* 2019).

The coagulant and flocculant selections are made by aiming at a minimum concentration of chemical use and getting maximum removal efficiency at optimum pH. However, the type and/or amount of the chemical may cause adverse effects on microbial activity in biological treatment processes. Toxic and inhibitory substances negatively affect the activity and treatment efficiency of microorganisms and also cause changes in the microbial community structure. The effect of coagulant takes place either in the form of inhibition of microorganisms directly in the biological treatment process or indirectly in the advanced treatment process of the chemical sludge. The degree of effectiveness is related to the treatment process applied to the dosing method and the coagulant type (Arslan-Alaton & Turkoglu 2008; Chen 2013).

The effect of coagulants on microorganism activity is listed as PAC (poly aluminum chloride) > $AlCl_3$ > $Fe_2(SO_4)_3$. Inhibition rates at 10 and 20 ppm are 11.9 and 33% for PAC; 15.8% and 9.6% for $AlCl_3$; and 8.5% and 5.4% for $Fe_2(SO_4)_3$, respectively. There is no direct inhibition effect of the backwash wastewater from the sand filter on the microorganism after the coagulants are added. The main reason for this is that stable precipitate is formed by the coagulant and phosphate and the free aluminum ion has a low concentration in water. However, backwash water causes an increase in inorganic components in the activated sludge. In the study, it is found that the volatile suspended solids/suspended solids (VSS/SS) ratio decreased from 0.65 to 0.54 after coagulants are applied (Chen 2013). Some coagulants, such as polyacrylamide (PAM), increase the precipitation, flocculation, and microbial activity of the activated sludge. However, the use of high concentrations (1 mg/L) of PAM results in the elimination of dissolved oxygen transfer, sludge disintegration, poor precipitation and low microbial activity (Luo *et al.* 2011). The primary sedimentation sludge, which used Fe or Al coagulants, is applied in anaerobic treatment for biogas production, and it is reported that the methane yield decreased by 20% to 50% compared to the raw primary sludge (Lin *et al.* 2017).

In this study, the effects of the use of coagulants and flocculants are investigated to increase the effectiveness of the treatment of domestic wastewater in a municipal wastewater treatment plant. It has been determined that the removal efficiency of the pre-settling tank in the treatment plant is not at the desired level, that the outlet water of the pre-settling tank contains quite a lot of solid particles. Since most of these solid particles are colloidal, they are suspended in the leaving water (effluent/outlet). In this suspension, the solid particles cannot be settled to desired levels, and they play an effective role in increasing the energy consumption of the facility, by increasing the hydraulic and organic loads of the next aerobic activated sludge unit. Therefore, to eliminate the negativities mentioned in this study, it is thought to reduce the pollution load sent to both the pre-settling tank and biological treatment by making coagulation-flocculation units before the pre-settling tank. In this way, it is aimed to increase the efficiency of the activated sludge unit and to create a more stable sludge for anaerobic digesters. Besides, it is aimed to investigate the toxic and inhibition effects of the chemicals used in coagulation-flocculation processes to the anaerobic digester located in the later stages of the treatment plant.

For this purpose, firstly, in this facility, standard jar test experiments are carried out with the wastewater, which consists of high suspended solids, taken from the effluent of the grit chamber, and the removal efficiency is determined. The aim of this is to reduce the pollution load of biological treatment. Then, the probable toxic and inhibition effects of the chemical substances used during the coagulation-flocculation process on the anaerobic digester are examined experimentally and the most appropriate coagulant is selected, and the results of the experiment are evaluated. The effect of these coagulant and coagulant aid substances on the gas outlet in the anaerobic digester is investigated by performing inhibition tests. Some coagulants are shown to prevent gas formation in experiments and have an inhibition effect on microorganisms. This study is a suitable treatment method for domestic wastewater with high suspended solids.

MATERIAL AND METHODS

Wastewater characterization

Municipal wastewater characterization used in the study is given in Table 1.

Wastewater treatment plant

The domestic wastewater treatment plant where the study is carried out is a municipal conventional domestic wastewater treatment plant. The plant has one coarse-fine grill, one grit chamber, three settling tanks, one trickling filter, one aerobic activated sludge unit, three anaerobic sludge stabilization reactors, and two chlorination units. The wastewater sample used in jar tests is taken from the effluent of the grit chamber. The wastewater is taken from this stage because the coagulation-flocculation unit can be placed there in a real treatment plant. The flow chart and sampling point of the plant are given in Figure 1. While most of the treated wastewater from the treatment plant is used for irrigation purposes in agricultural areas, a small amount of treated wastewater is discharged into the sea.

Table 1 | Characterization of municipal wastewater

Parameters	Raw wastewater	The effluent of grit chamber
COD_T (mg/L)	2,700	1,000
BOD (mg/L)	800	300
TSS (mg/L)	1,500	400
VSS (mg/L)	1,200	300
Conductivity (mmho)	2.2	2
pH	7.5	7.7
NH_4^+ (mg/L)	125	50
PO_4^{3-} (mg/L)	30	55
Cl^- (mg/L)	350	400

"BOD: biological oxygen demand; COD_T: total chemical oxygen demand".

Coagulation-flocculation experiments

Coagulation-flocculation experiments are carried at room temperature (20 ± 2) using a standard jar test apparatus (Velp Scientifica FC6S) equipped with six beakers of 500 mL volume. $FeCl_3$, $FeClSO_4$, Alum ($Al_2(SO_4)_3.18\ H_2O$) are used as chemical reagents, polymeric coagulant LP 526 as a coagulant, and Anionic Polyelectrolyte (APE), Cationic Polyelectrolyte (CPE) as coagulant aid and their combinations. 0.1 N HCl and 0.1N NaOH are used to adjust the pH if found necessary. Coagulation experiments are conducted in the following manner: coagulant addition, pH adjustment, flash-mixing (stirring for 1 min at 100 rpm), flocculation (stirring for 15 min at 50 rpm), settling for 10 min.

Inhibition experiments

The experimental setup is illustrated in Figure 2. Experiments are done by adding the optimum concentration of

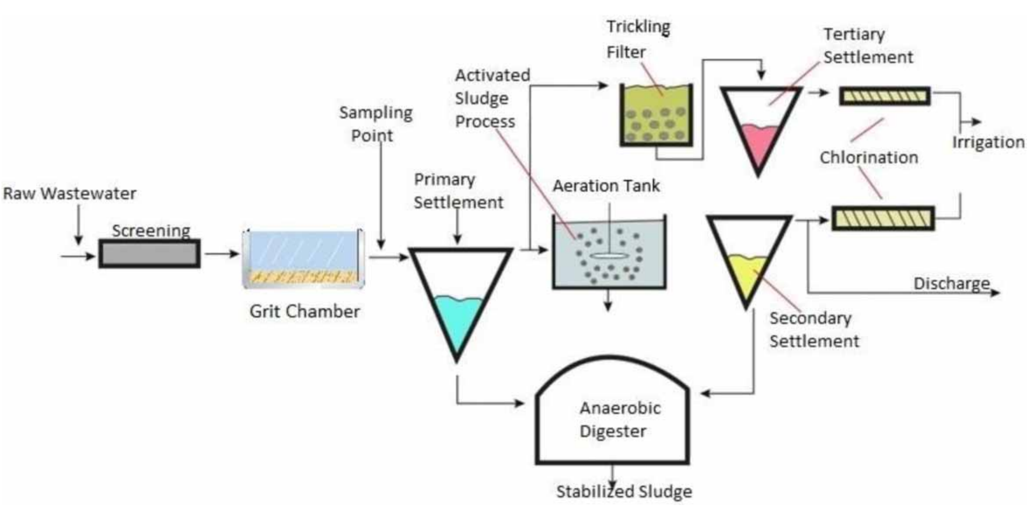

Figure 1 | Wastewater treatment plant and sampling point.

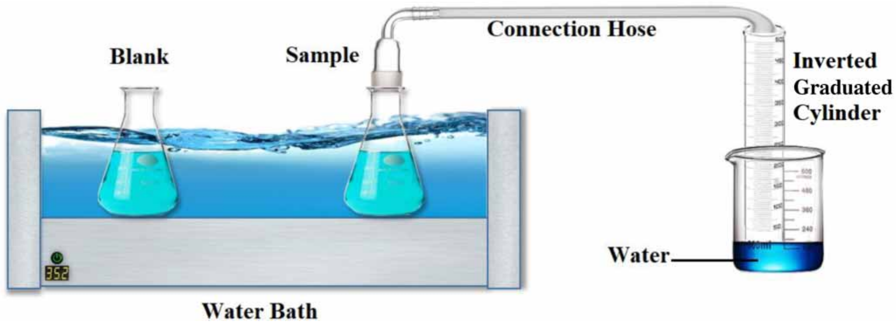

Figure 2 | Inhibition experimental setup.

the chemicals as $FeCl_3$, $FeClSO_4$, Alum, APE, CPE, LP 526, and their combinations. The sludge for the analysis is taken from the primary anaerobic digester of wastewater treatment plant and 250 mL of sludge is placed in the flasks. Constant temperature (35 °C) is maintained by immersing the reaction flasks into a water bath. The reaction flasks are well-mixed and their temperatures are kept constant by using a shaking water bath at 35 °C, 120 rpm, for 945 h. Inhibition experiments are carried out for eight different chemicals and their combinations as summarized in Table 2. In the experiments, the effect of the chemicals added to the sludge on the gas output efficiency is followed. Each inhibition test is performed in approximately 945 h.

RESULTS AND DISCUSSION

In this study, the coagulation and flocculation process on municipal wastewater treatment efficiency, the toxic and inhibition effects of the coagulant, and coagulant aid used on the anaerobic digester are investigated to enhance wastewater treatment efficiency of the wastewater treatment plant.

Table 2 | Coagulants, coagulant aids and their doses which are used in the experiment

	Chemicals	Dosage used, mg/L
Coagulant	$FeCl_3$	20
	$FeClSO_4$	20
	$Al_2(SO_4)_3.18H_2O$	20, 50, 100
	Polymeric coagulant LP 526	10
Coagulant aids	APE 65	1–2.0
	APE 67	1.0–1.5
	APE 85	1.0–1.5
	CPE 84	2.0
Combination	$FeCl_3$/APE 65	20/1.0
	$FeCl_3$/APE 67	20/1.5

Evaluation of results of jar tests

To determine the effects of coagulants and their aid used in this study on removal efficiencies, the jar test experiments are carried out in the wastewater taken from the grit chamber outlet. With the use of coagulants and aid in various concentrations, the COD and total suspended solids (TSS) removal efficiencies obtained at the end of the jar tests are given in Table 3.

Different combinations are formed with Polymeric coagulant LP 526 and coagulant aids APE 65, APE 85, and CPE. When the APE 65 dosage increased from 0.5 mg/L to 1.5 mg/L, the COD and TSS removal efficiencies decreased from 54% to 51% and 90% to 85%, respectively. The optimum dosage of LP 526/APE 85 is determined as 1.5 mg/L, and the COD and TSS removal efficiencies are 53% and 88%, respectively. The optimum dosage is determined as 2.0 mg/L with LP 526/CPE 84, and the COD and TSS removal efficiencies are found as 55% and 94%, respectively.

After the jar tests with coagulant aids (APE 65, APE 67, APE 85) used with $FeClSO_4$ with a fixed concentration of 20 mg/L, the optimum combination is obtained with APE 65 at 1.0 mg/L concentration by considering the COD and TSS removal efficiencies. At the end of this test, COD and TSS removal efficiencies are determined as 53% and 91%, respectively. Similarly, the optimum combination is determined as 50 mg/L $Al_2(SO_4)_3$ and 1.0 mg/L APE 67 after the tests with APE 65, APE 67, APE 85 coagulant aids at 1.0 mg/L dose. With this combination, the COD and TSS removal efficiencies are determined as 59% and 85%, respectively.

At the end of jar test experiments performed with $FeCl_3$, Synthetic coagulant LP 526, $FeClSO_4$, $Al_2(SO_4)_3$ coagulants and combinations of APE 65, APE 67, APE 85, CPE 84 coagulant aids in various concentrations, the results of COD and TSS removal efficiencies are ranged

Table 3 | Summary of the results of the jar test study

Exp. number	Coagulant	Dose (mg/L)	COD_T (mg/L)	COD_T removal efficiency (%)	TSS (mg/L)	TSS removal efficiency (%)
Influent	–	–	1,000	–	400	–
1	$FeCl_3$/APE 65	20/1.0	450	55	45	89
2	$FeCl_3$/APE 65	20/1.5	490	51	45	89
3	$FeCl_3$/APE 67	20/1.0	500	50	50	88
4	$FeCl_3$/APE 67	20/1.5	480	52	45	89
5	$FeCl_3$/APE 85	20/1.0	480	52	70	83
6	$FeCl_3$/APE 85	20/1.5	470	53	40	90
7	LP 526/APE 85	10/1.5	475	53	50	88
8	LP 526/APE 65	10/0.5	455	54	60	90
9	LP 526/APE 65	10/1.5	490	51	60	85
10	LP 526/CPE 84	10/2.0	450	55	25	94
11	$FeClSO_4$/APE 65	20/1.0	470	53	35	91
12	$FeClSO_4$/APE 67	20/1.0	480	52	40	90
13	$FeClSO_4$/APE 85	20/1.0	485	51	50	88
14	$Al_2(SO_4)_3$/APE 67	50/1.0	410	59	60	85

from 50% to 59 and 83% to 94%, respectively. The optimum COD and TSS removal efficiencies in these jar test experiments with wastewater taken from the grit chamber effluent are the combinations of $FeCl_3$/APE 65 (20/1.0 mg/L), $FeCl_3$/APE 67 (20/1.5 mg/L), $FeCl_3$/APE 85 (20/1.0 mg/L), Synthetic coagulant LP 526/CPE 84 (10/2.0 mg/L), $FeClSO_4$/APE 65 (20/1.0 mg/L) and $Al_2(SO_4)_3$/APE 67 (50/1.0 mg/L).

Evaluation of inhibition test results

After performing all the jar tests, reasonable results are acquired on the treatability of the wastewater sample taken from grit chamber effluent. The results of the inhibition tests are given in Figure 3.

Figure 3 shows the results of 945 h of inhibition analysis performed with the optimum doses of coagulant and

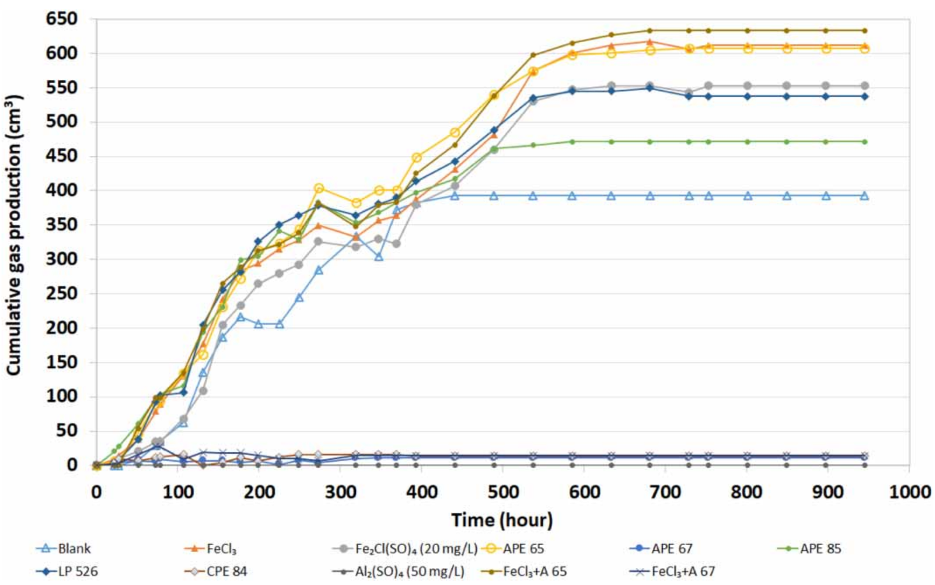

Figure 3 | Results of inhibition tests.

coagulant aids obtained as a result of jar tests. It is determined that the highest gas production is performed with 701 cm^3 in 700 h in the experiment using 1 mg/L APE 65 coagulant assistant. In inhibition experiments using only $FeCl_3$ with a concentration of 20 mg/L, approximately 620 cm^3 of gas production in 700 h, in another experiment, in which 20 mg/L $FeCl_3$ and 1 mg/L APE 65 coagulant assistant are used, approximately 620–650 cm^3 of gas production in 600 h is determined. In the experiments with synthetic coagulant LP 526, APE 85, and $FeClSO_4$, it is observed that approximately 545 cm^3, 470 cm^3, and 615 cm^3 of gases are produced after 600 h, respectively. According to the results in Figure 3, it is determined that $FeCl_3$, APE 65, APE 85, Synthetic coagulant LP 526, and $FeClSO_4$ did not cause inhibition effects in the sludge.

When the results of inhibition tests conducted with APE 67 1.5 mg/L, 20 mg/L $FeCl_3$/1.5 mg/L APE 67, and 50 mg/L $Al_2(SO_4)_3$ are evaluated, experiments performed with APE 67, CPE 84, and $Al_2(SO_4)_3$ showed no gas formation, and it has been determined that these coagulant and coagulant aids have an inhibition effect on microorganisms. In the experiments carried out without adding any coagulant and coagulant aids, at the end of the 945-h experiment, approximately 400 cm^3 of gas is obtained in only 400 h. It is also concluded from this that gas production increases while non-toxic coagulant and coagulant aids are used. CPE 84 cationic and APE 67 anionic polyelectrolytes are commercial organic coagulants, and they are acrylamide and acrylic acid derivatives. Such polyelectrolytes are petroleum products and not environmentally friendly (Bolto & Gregory 2007). Synthetic organic materials are highly resistant to biodegradation (Bolto & Gregory 2007; Brostow *et al.* 2009). Cationic polyelectrolytes have been reported to be highly toxic to aquatic organisms. These polyelectrolytes are thought to cause inhibition by causing toxic effects to microorganisms in the sludge (Singh *et al.* 2000).

Coagulation experiments are carried out in wastewater with combinations of $FeCl_3$/APE 65 (20/1 mg/L), synthetic coagulant LP 526/APE 65 (10/0.5 mg/L), and $FeClSO_4$/APE 65 (20/1 mg/L) which have high COD and TSS removal efficiencies and have no inhibition effects on sludge. The removal efficiencies obtained in various parameters at the end of these experiments are given in Table 4.

In experiments with wastewater where $FeCl_3$, LP 526 and $FeClSO_4$ coagulants are used alone: $FeCl_3$ 20 mg/L dose, 77% COD and 90% TSS removal; $FeClSO_4$ 20 mg/L dose, 78% COD and 85% TSS removal; LP 526 10 mg/L dose, 70% COD and 83% TSS removal are obtained. It is seen in Table 4 that the removal efficiency increases even more when coagulant aids are added.

As can be seen in Table 4, similar removal efficiencies are obtained in COD, TSS, and VSS parameters. However, the highest removal efficiencies in COD, biological oxygen demand (BOD), PO_4, TSS, and VSS parameters are provided with $FeCl_3$/APE 65 combinations. Along with these results, 82%, 75%, 33%, 96%, and 95% removal efficiencies are obtained in COD, BOD, PO_4, TSS, and VSS parameters, respectively. As BOD removal efficiencies are evaluated, it is seen that the best result is obtained with $FeCl_3$/APE 65 combination (75%). With the combination of synthetic coagulant LP 526/APE 65 and $FeClSO_4$/APE 65%, 56% and 50% removal efficiencies are obtained in this parameter, respectively. Only in the NH_4 parameter, the highest removal efficiency is obtained with Synthetic coagulant LP 526/APE 65 combination (44%).

When the results obtained are evaluated together, it is seen that the chemical treatment (coagulation-flocculation process) is very effective in the case of a high amount of

Table 4 | Results of the coagulation of wastewater with optimum coagulants according to inhibition and jar test experiments

Coagulant and dose (mg/L)	pH	COD (mg/L)	COD removal efficiency (%)	BOD (mg/L)	BOD removal efficiency (%)	NH_4 (mg/L)	NH_4 removal efficiency (%)	PO_4 (mg/L)	PO_4 removal efficiency (%)	TSS (mg/L)	TSS removal efficiency (%)	VSS (mg/L)	VSS removal efficiency (%)
Influent	7.5	2,700	–	800	–	125	–	30	–	1,500	–	1,200	–
$FeCl_3$ (20 mg/L)	7.5	621	77	232	71	85	32	20	33	150	90	120	90
$FeClSO_4$ (20 mg/L)	7.8	594	78	430	46	80	36	27	10	225	85	180	85
LP 526 (10 mg/L)	7.8	810	70	400	50	75	40	28	6	255	83	204	83
$FeCl_3$/APE65 (20/1)	7.7	485	82	200	75	80	36	20	33	60	96	55	95
LP 526/APE65 (10/0.5)	7.8	530	80	350	56	70	44	29	3	80	94	70	94
$FeClSO_4$/APE65 (20/1)	7.8	540	80	400	50	90	28	25	17	75	95	60	95

TSS in domestic/municipal wastewater. The produced and precipitated sludge which is formed before anaerobic digestion is always very important for the fate of the anaerobic degradation. The sludge must not have any inhibitory and toxic substances in its body, and the activity of the microorganisms should be as high as possible, and equal to their maximum activity. In the operation of coagulation-flocculation, due to the nature of the operation, a large amount of precipitated sludge is received, and this sludge mostly contains five or six times more chemicals than its usual value due to its compaction. This can sometimes be very dangerous for the microorganisms which are responsible for the degradation of the organic matter. With this consideration, in the last step of the experiment, the toxic and inhibitory effect of each coagulant and coagulant aid on the anaerobic digestion of mixed suspended solids is determined.

Considering the data obtained in the study, when one of these chemicals is used in the process, energy is saved in units such as activated sludge and anaerobic reactor due to reduced load. In other words, if the concentration of COD and BOD is reduced at least by 100 mg/L, it is thought that the energy is saved will be higher than the chemical cost (Soares *et. al.* 2020).

CONCLUSIONS

With $FeCl_3$, $FeClSO_4$, LP 526, and alum coagulants with the APE and CPE aid used in the experiments, removal efficiencies of 51–59% in COD and 83–90% in TSS are obtained. As a result of the 945-h inhibition analyses performed using optimum doses of coagulant and coagulant aids at the end of the jar test, the highest gas production is obtained in the experiment with the APE 65 coagulant aid. It can be seen that $FeCl_3$, APE 65, APE 85, Synthetic coagulant LP 526, and $FeClSO_4$ do not cause inhibition effects on sludge. In experiments with APE 67, CPE 84, and $Al_2(SO_4)_3$, no gas formation is observed, and these coagulant and coagulant aids used had an inhibition effect on microorganisms. It is also seen in the literature that aluminum and auxiliary chemicals containing acrylamide have toxic effects (Bolto & Gregory 2007). According to the results of the experiments, low cost and high energy saving operations can be realized with single and low concentrations of $FeCl_3$ and $FeClSO_4$ use. When the study is evaluated in general, it is concluded that the use of the chemicals used is very advantageous in terms of both removal efficiency and economics and that they can be used effectively in the treatment of domestic wastewater. Depending on the costs and removal efficiencies of these chemicals, the facility's contribution to energy saving is thought to be between 1.5 and 3 times.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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