






## Analysis of the effect of using divalent and trivalent iron compounds on sludge dewatering performance in thickening units

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### ABSTRACT

Sludge is one of the by-products of wastewater treatment plants (WWTPs). To assist biological processes, part of the produced sludge is returned to the treatment process and the excess is removed from the treatment plant whereby it undergoes thickening and dewatering. Numerous studies have been conducted to investigate the effect of different coagulants on dewatering from sludge, but the effect of using divalent iron for this purpose has not been investigated so far. In this research, the effect of divalent and trivalent iron compounds (ferrous sulfate and ferric chloride) on the dewatering of excess sludge of the first module of the Bojnourd WWTP (Iran) has been investigated. In this regard, first, the effect of different doses of each coagulant to optimize the dewatering characteristics of sludge was investigated and then the effect of pH change by adding lime was investigated. The results showed that the addition of optimal doses of  $\text{FeSO}_4$  (0.6 and 0.4 g/l) and lime (0.664 and 1.5866 g/l) reduced the capillary suction time of sludge by 30.6 and 32.7%, respectively, while reducing the moisture content of sludge cake by 26 and 30.6%.

**Key words:** capillary suction time, coagulant, iron compounds, sludge dewatering, sludge volume index

### HIGHLIGHTS

- A novel methodology of sludge technology.
- First implementation of the new materials for sludge dewatering.

### INTRODUCTION

In recent years, due to population growth, urban development and giving greater importance to environmental protection, more wastewater has been treated and the amount of excess sludge generated in municipal wastewater treatment plants (WWTPs) has increased (Garrido-Cardenas *et al.* 2019). Excess sludge often contains contaminants such as pathogens, heavy metals and pesticides, and can adversely affect the quality of the environment, human health and agriculture. For this reason, the treatment and disposal of excess sludge is a major issue in municipal WWTPs (Wu *et al.* 2017). Control of biological solids (sludge) has been considered complex and difficult due to the large volume of solids and the presence of organic matter (Metcalf *et al.* 2004). According to available data, each person produces 35–85 g of dry matter daily (Jamshidi *et al.* 2011). Sludge treatment is one of the most expensive processes available in WWTPs, which accounts for approximately 50% of the operating cost of the wastewater treatment system. The purpose of sludge treatment is to minimize its further decomposition and spread of unpleasant odors, and reduce the volume to economize the treatment, management and disposal (Groff & McLaughlin 1994). To reduce the exorbitant costs of investment, management of treatment facilities, sludge stabilization and prevention of environmental pollution, it is necessary to reduce the volume of sludge produced in WWTPs as much as possible (AbdulAzeez *et al.* 2016). Thickening and dewatering methods are used for this purpose. Because the sludge is modified, it is easily thickened (concentrated) and dewatered. Therefore, sludge preparation operations are of special importance in WWTPs. Sludge preparation is a two-step process involving coagulation and flocculation. The main purpose of sludge preparation is to increase the particle size, overcome the effects of hydration and repel electric charge between particles. In other words,

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the preparation of the sludge causes the accumulation of fine dispersed and colloidal particles in the sludge and the release of the bonded water between them. In most cases, chemicals are used to prepare the sludge, which increases the production of sludge (AmanaliKhani *et al.* 2016).

In addition to the basic processes of sludge treatment (thickening, stabilization and dewatering), it is possible to apply additional treatment procedures, such as thermal drying or even incineration, which further solve the problem of stabilizing sludge and reducing its volume. However, these processes are highly energy-intensive and therefore often represent more expensive solutions to sludge treatment and disposal problems. With regard to the reduction of sludge volume and the increase of dry matter concentration depending on the sludge treatment phase, the values shown in Table 1 can be given. Other authors cite different values of dry matter concentrations in sludge that has undergone different stages of treatment. Thus, for example, Donatello and Cheeseman state that primary and biological sludge usually contain 1–4% dry matter, while further processing (thickening) of sludge achieves concentrations of 3–8% dry matter and the last stage of removal of water from sludge (dewatering) produces a sludge cake with 18–35% dry matter (Donatello & Cheeseman 2013).

**Table 1** | Reducing the volume of sludge and increasing the concentration of dry matter through the stages of its treatment (Qasim 2017)

Parameter	Raw sludge	Thickened sludge	Dehydrated sludge	Dried sludge	Incinerated sludge
Dry matter concentration (%)	1	5	25	90	100
Volume reduction relative to the raw sludge	1	5	25	90	330
Residual volume (%) (relative to raw sludge volume)	100	20	4	1.11	0.30

Numerous studies have been performed on the effect of chemical, organic and polymeric coagulants on sludge dewatering as well as the removal of environmental pollutants. In a study by Guo *et al.*, the effect of modified corn-core powder (MCCP) using sodium hydroxide (NaOH) and cetyltrimethylammonium bromide (CTMAB) surfactant was investigated. The results of this study showed that MCCP as an environmentally friendly, available and cost-effective sludge coagulant can effectively increase the degradability and dewatering of sludge (Guo *et al.* 2019). Lin *et al.* (2019) the improvement of dewatering properties of activated sludge using green coagulants of chitosan hydrochloride (CTSCL) and lysozyme (LZM). They investigated the effects of CTSCl, LZM and cationic polyacrylamide (CPAM) as coagulants on sludge dewatering performance against capillary suction time (CST) parameters and specific resistance to filtration (SRF) and moisture examined dewatered sludge. The results of this study showed that LZM coagulant has the best improvement in sludge dewatering and can reduce sludge moisture content after dewatering by 19.84% (Lin *et al.* 2019). The innovative combination of potassium permanganate (KMnO<sub>4</sub>) and proximonosulfate (PMS) has been used for sludge dewatering by Luo *et al.* (2019). Wu *et al.* (2017) investigated the effectiveness of using coal/FeCl<sub>3</sub>/KMnO<sub>4</sub> to enhance sludge dewatering (changes in sludge properties in the sludge treatment mechanism). The results of his research showed that the optimal doses of KMnO<sub>4</sub> and sludge cake charcoal reduce the specific resistance of sludge to filtration (Wu *et al.* 2017). Improvement of sludge dewatering using the aluminum-iron-starch composite coagulant has been investigated by Lin *et al.* (2015). In a study by Wu *et al.*, FeCl<sub>3</sub>-modified rice husk charcoal (MRB-Fe) was used to increase sludge dewatering. They found that with an optimal concentration of FeCl<sub>3</sub> (3 mol/l), ultrasound time (1 h) and optimal dose of MBR-Fe (DS 60%), the sludge-specific filtration resistance (SRF) was reduced by 97.9% (Wu *et al.* 2016). In 2016, Abdulazeez *et al.* investigated the effect of using *Moringa oleifera* seed extract along with aluminum sulfate in dewatering sludge. They found that the optimum condition for the 50:50 mixture (w/w) for *M. oleifera* and alum was  $SRF = 0.833E + 11$ , and it was good enough to reduce the amount of alum by only 50.2% compared to using alum only decreases (Abdulazeez *et al.* 2016). Improved secondary sludge dewatering using a hybrid coagulant-silicon-aluminum-iron-starch was investigated by Lin *et al.* in 2015. They synthesized a new combination of silicon-aluminum-iron-starch with a bond of silicon, aluminum and iron on the structure of starch and studied its effect on improving the dewatering of secondary sludge. The results of his research showed that when copolymer was added, it had good dewatering efficiency in a wide range of pH (0.11–0.3) and has better dewatering performance compared to coagulants such as polyaluminum chloride

(PACl), polyacrylamide (PAM) and ferric chloride (Lin *et al.* 2015). In the study of Zemmouri *et al.* in 2015, the effect of chitosan, synthetic cationic polyelectrolyte CF802 (Sed CF802) and ferric chloride ( $\text{FeCl}_3$ ) on improving the dewatering of municipal sewage sludge was investigated. The results of their study showed that the use of the optimal dose of chitosan and Sed CF802 reduces the turbidity by 94.86 and 87.85%, respectively, and the optimal dose of  $\text{FeCl}_3$  reduces the turbidity of the drain by 54.18% (Zemmouri *et al.* 2015). In 2014, Zhou *et al.* used a combination of zero-valent iron (ZVI) and hydrogen peroxide (HP) at  $\text{pH} = 2.0$  to improve the dewatering capability of excess waste-activated sludge (WAS). They found that using a combination of ZVI (0–750 mg/l) and HP (0–750 mg/l) at  $\text{pH} = 2.0$  significantly improved the dewatering capability of excess activated sludge (Zhou *et al.* 2014). Fitria *et al.* in 2013 evaluated different shapes of mixers (radial, axial, wheel, three-blade and magnetic), different fast mixing speeds, fast mixing times and coagulation upon sludge dewatering. These experiments showed that using different forms of mixers, different fast mixing speeds and different fast mixing times did not lead to a significant change in CST (Fitria *et al.* 2013). In a study in 2010, by Chen *et al.*, the improvement of sludge dewatering capacity using coal fly ash modified by sulfuric acid (MCFA) was investigated. The results of this research showed that the SRF of sludge is significantly reduced by adding coal ash and the purification effect of MCFA is much stronger than raw coal fly ash (RCFA) (Chen *et al.* 2010). AmanaliKhani *et al.* in 2016 investigated the effectiveness of PAM coagulant modified with aluminum oxide nanoparticles in dewatering the sludge produced by the Yazd WWTP. The results of this study showed that the coagulant aid modified with aluminum oxide nanoparticles at the optimum pH and concentration (4 and 5 mg/l, respectively) has the best performance so that the filtration time and moisture of sludge cake, turbidity of all filtered water solids ratio in the control sample (CPAM) is reduced by about 24.4, 11.2, 57.2 and 58%, respectively (AmanaliKhani *et al.* 2016).

Improvement of wastewater sludge dewatering using ferric chloride, aluminum sulfate and calcium oxide was investigated by Ranjbar *et al.* (2021) and the optimal concentration, pH and coagulation/flocculation time were determined. Yang *et al.* investigated the synergistic effects between CPAM and synthetic fibers, which could improve wastewater sludge dewaterability and resource utilization. They found the sludge water content decreased 30.0% after conditioning with the combination of CPAM (Yang *et al.* 2023). Electro-coagulation combined with added free nitrous acid were used for dewatering municipal wastewater sludge by Wang *et al.* and the optimal applied voltage for EC, the optimal dosage of free nitrous acid and the optimal pH value were determined according to the dewaterability of the sludge (Wang *et al.* 2022).

It is observed that a lot of research has been done to increase the dewatering efficiency of sludge but previous researchers have not investigated the effect of using divalent iron for dewatering sludge. In the present research, the use of divalent and trivalent iron compounds to increase the dewatering performance of sludge is investigated.

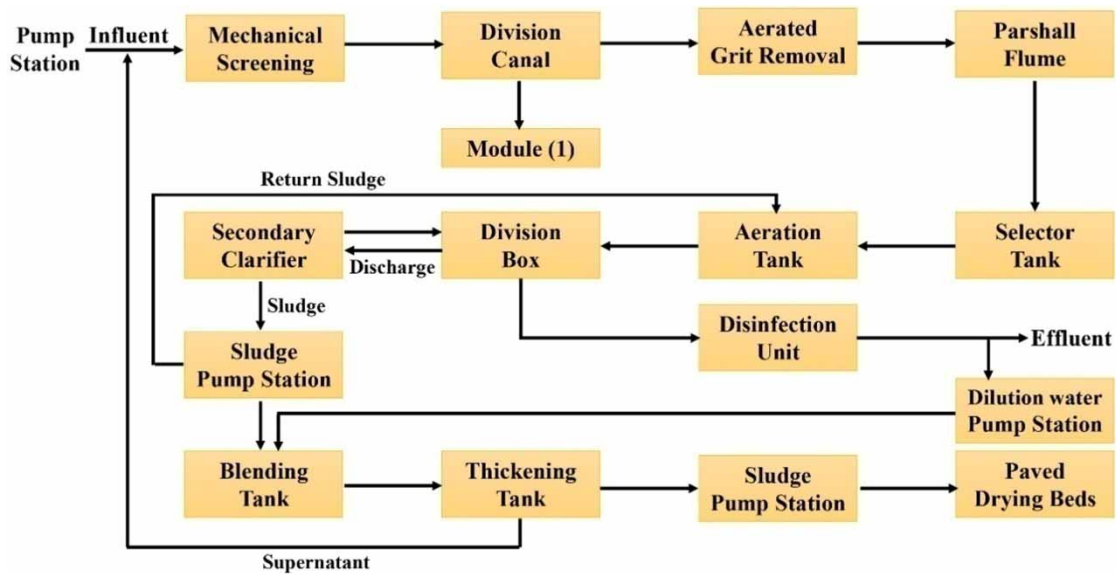
## MATERIALS AND METHODS

This research is an experimental study and has been done on a laboratory scale. The coagulants used are divalent iron compounds including divalent iron sulfate ( $\text{FeSO}_4$ ) and divalent iron chloride ( $\text{FeCl}_2$ ) and trivalent iron compounds including trivalent iron sulfate ( $\text{Fe}_2(\text{SO}_4)_3$ ) and trivalent iron chloride ( $\text{FeCl}_3$ ). Lime was also used as a coagulant.  $\text{FeSO}_4$ ,  $\text{Fe}_2(\text{SO}_4)_3$  and lime were purchased from DAYA EXIR Co. and  $\text{FeCl}_2$  and  $\text{FeCl}_3$  were purchased from XCHEM Co. The tested sludge was supplied from the excess sludge of the first module of the Bojnourd city WWTP (sludge entering the sludge gravitational thickener unit). The flow diagram of the Bojnourd city WWTP is presented in Figure 1.

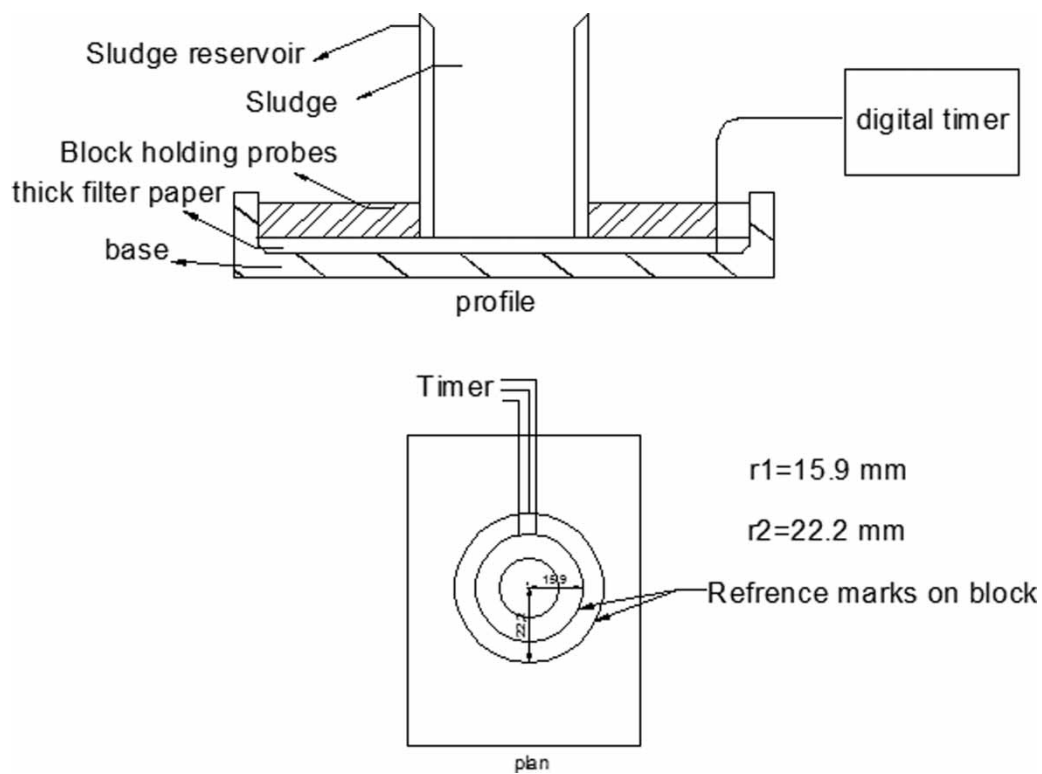
The parameters studied in this study including pH, coagulant concentration and coagulant adjuvant concentration were selected. For the laboratory testing in this study, a CST device was built according to the design described in Baird *et al.* (2017). A schematic representation of the device is shown in Figure 2. Also pH meter model 540GLP to measure temperature and pH parameters, sensitive analytical balance with an accuracy of 0.0001 g model GR-200 for weighing chemicals and Whatman grade 1 filter paper to measure TSS parameter of sludge, JLT6 model Jar test machine, vacuum pump model C55JXHRL-4205 and universal oven Model UNB 400 were used.

### Test method

In this study, to evaluate the improvement of sludge dewatering capability, sludge volume index (SVI) and CST were analyzed. Initially, the sludge samples were taken from the excess sludge inlet of the first module of the Bojnourd WWTP to the gravity concentration unit and the tests were carried out in the laboratory of the treatment plant. Then different doses of each coagulant (to determine the optimal dose range) were stirred on the sludge in



**Figure 1** | Flow diagram of the Bojnourd city WWTP (Iran).



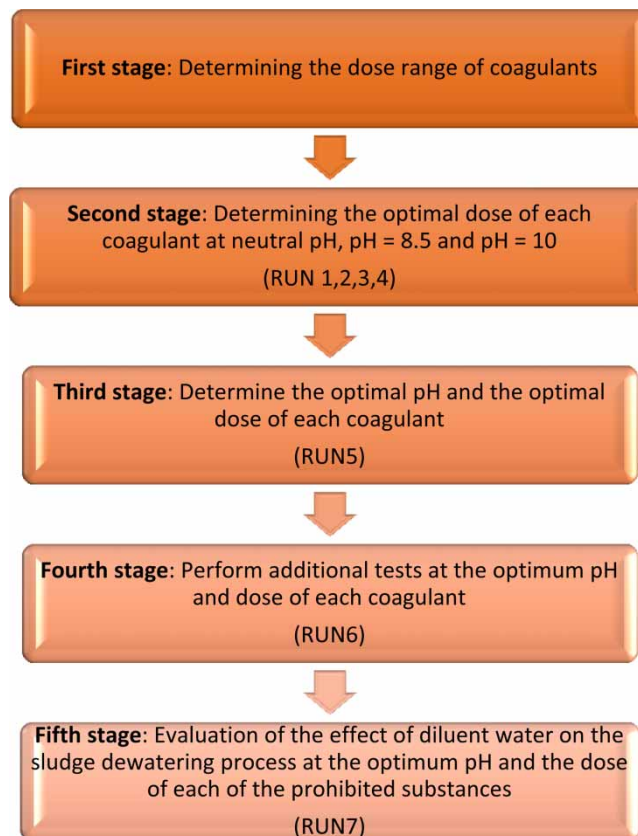
**Figure 2** | Scheme of the device used to perform the CST test (Baird *et al.* 2017).

the jar test machine. The sample size was 1,000 ml per human being and rapid mixing at 240 rpm for 1 min and slow mixing at 40 rpm for 5 min were considered. Control tests at this stage were the  $SSV_{30}$ , SVI, CST and TSS. To increase the accuracy of the CST test, this test was performed 5 times in each step and the average of the collected individual results was recorded as a final result (Baird *et al.* 2017). After determining the dose range of coagulants, the effect of selected doses of each material alone and using pH change with the help of lime coagulant was investigated. Control tests at this stage also included the  $SSV_{30}$ , SVI, CST and TSS. To determine the amount of lime required, the jar test was performed in several stages with different doses of lime injection on

the sludge and the pH parameter of the sludge was measured. Then, the relationship between the amount of lime used and the pH changes of the sludge was extracted as follows:

$$\text{pH}_{\text{Sludge}} = 1.6627\text{gr}_{\text{CaO}} + 7.342$$

The best results were obtained for three sludges used: with neutral pH, with pH about 8.5 and with pH of about 10, and in the next step, the experiments were repeated with the sludge of the same quality and optimal doses. The results of this experiment determined the optimal consumption of each coagulant as well as the amount of coagulation aid required for each coagulant. Control tests at this stage were the  $\text{SSV}_{30}$ , SVI, CST and TSS. In the next step, to ensure the results, the experiments with the same sludge and the optimal dose of coagulants were repeated and the moisture content of the sludge cake was also examined. Due to the injection of diluent water in the gravity thickener unit, in a supplementary experiment, the effect of dilution water injection on the performance of coagulants was also tested. The control test at this stage was the SVI parameter. It should be noted that in all stages of testing, the biological quality of the tested sludge was examined by a microscope, which always used the sludge clots in the position of the needle clot. More details of used standard test methods were presented by [Ranjbar \*et al.\* \(2021\)](#). The test steps are summarized in [Figure 3](#). As can be seen, all tests were performed in five stages and seven runs, and the results of each run determined the process of performing the tests in the next run.

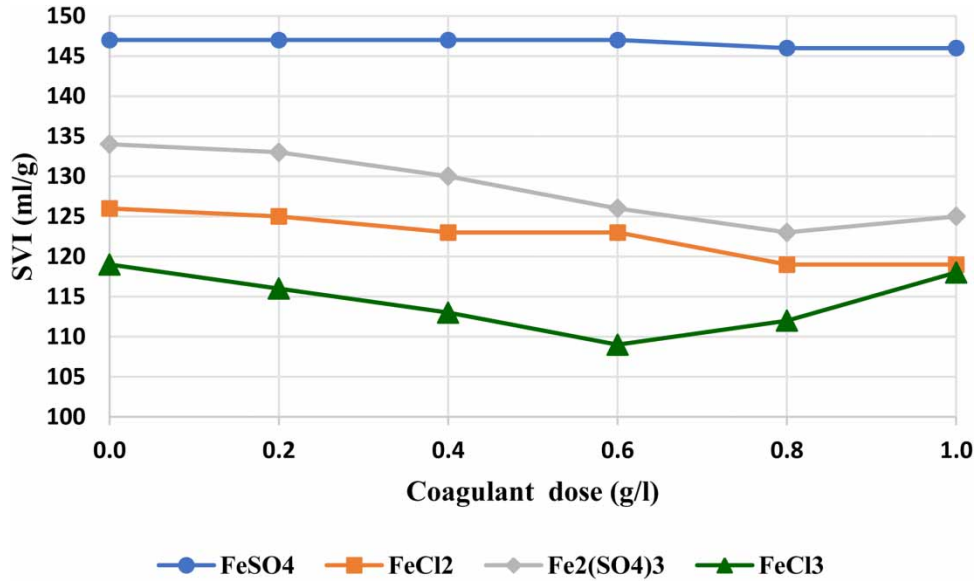


**Figure 3** | Flowchart of the performed test stages.

## RESULTS

As mentioned, in this study, the SVI, CST and TSS control tests of sludge surface were used to evaluate the effect of coagulants (iron compounds II and trivalent iron). According to the obtained results, the use of dilution water in the mixing tank of the sludge gravity concentration unit does not have a significant effect on the sludge dewatering process when adding coagulants. [Figure 4](#) shows the effect of each of these coagulants in improving sludge dewatering by examining the SVI parameter. As can be seen in [Figure 4](#), by increasing the coagulant dose to the

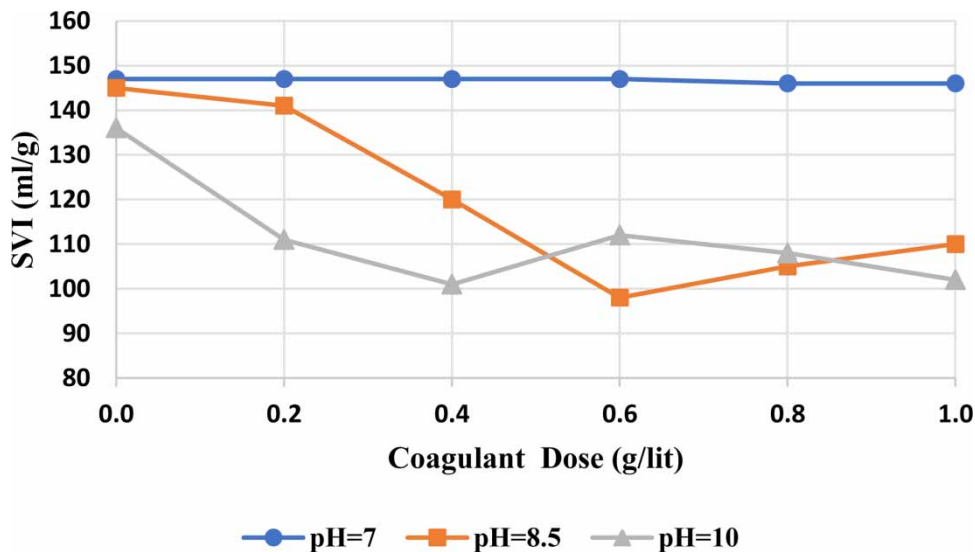




**Figure 4** | Effect of different doses of coagulants on the SVI of neutral pH sludge at an ambient temperature.

optimum level, the SVI parameter, which is directly related to the sludge dewatering process, is improved, while by increasing it further, the opposite result is obtained. This stage of the experiment was performed without injection of coagulant (pH change) and the results showed that iron sulfate (II), iron chloride (II), iron sulfate (III) and iron chloride (III) were in the optimal case, the volume index of the sludge improves by 0.7, 8, 8 and 6% and the dose range of the coagulant between zero and one gram is selected. This improves the volume of sludge by 0.7, 8, 8 and 6%, and the dose range of coagulant was selected between 0 and 1 g.

Then, the effect of using lime coagulant on improving the dewatering process of each coagulant to reduce the dose of coagulant and also increasing the pH of the final sludge to control the odors spread within the WWTP was investigated. Different doses of lime (pH = 8.5 and pH = 10) were used for this purpose. As shown in Figure 5, the use of a coagulant (lime) and increasing the resulting pH have a significant effect on improving the results and the obtained SVI: injecting 0.6 g/l of divalent ferrous sulfate into the sludge with 8.5 pH decreases the SVI by 32%. Also, the injection of 0.4 g/l of divalent ferrous sulfate into the sludge with pH = 10 reduces the SVI parameter by 26%.



**Figure 5** | Effect of different doses of divalent iron sulfate coagulant (FeSO<sub>4</sub>) on the SVI of the sludge with pH of 7, 8.5 and 10.

Figure 6 shows the effect of using different doses of ferrous chloride on reducing the SVI of sludge with pH of 7, 8.5 and 10. As shown in the figure, adding 0.8 g/l of divalent iron chloride to the sludge with pH = 7 and adding 1 g/l of divalent iron chloride to the sludge with pH = 8.5 and pH = 10 reduces the SVI by 26.6 and 44%, respectively.

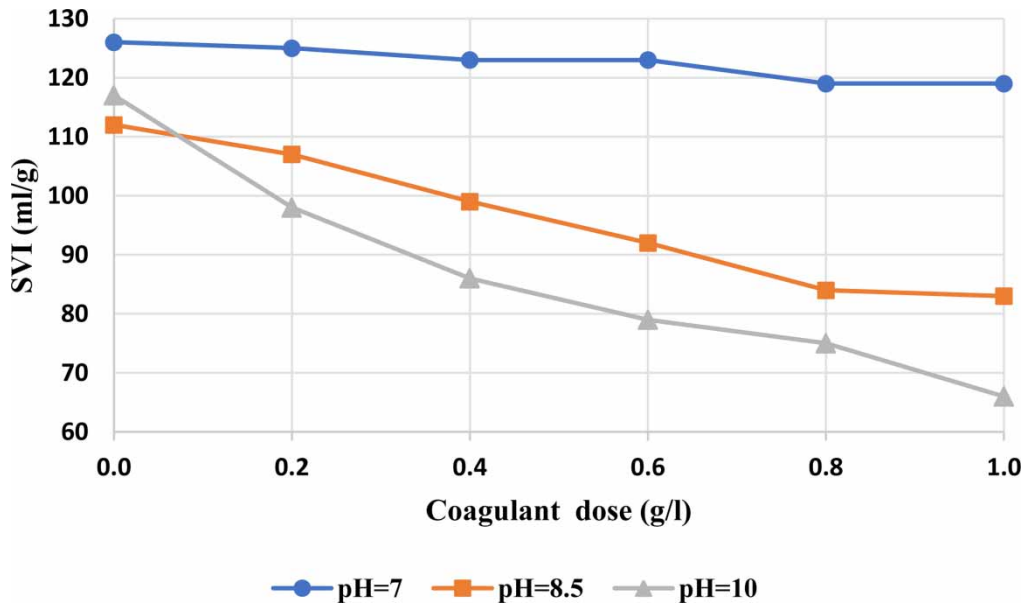


Figure 6 | Effect of different doses of divalent iron chloride (FeCl<sub>2</sub>) coagulant on the SVI of the sludge with pH of 7, 8.5 and 10.

Figure 7 shows the effect of injecting trivalent iron sulfate at doses of 0.8, 0.1 and 0.4 g/l into sludge at pHs of 7, 8.5 and 10, and as can be seen, the SVI of sludge is reduced by 8, 11 and 13%, respectively.

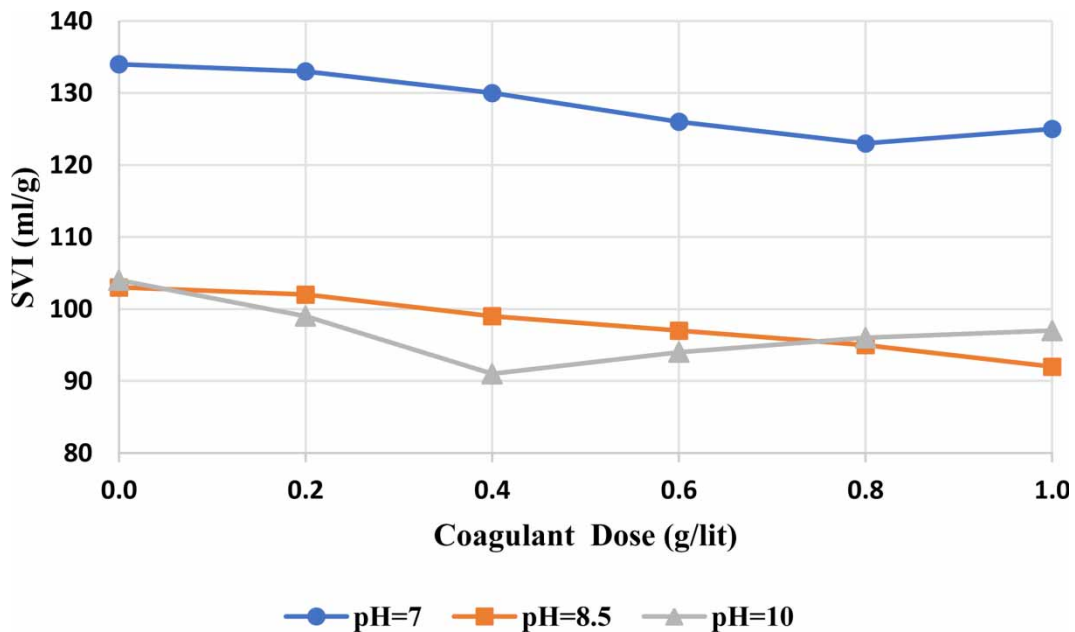
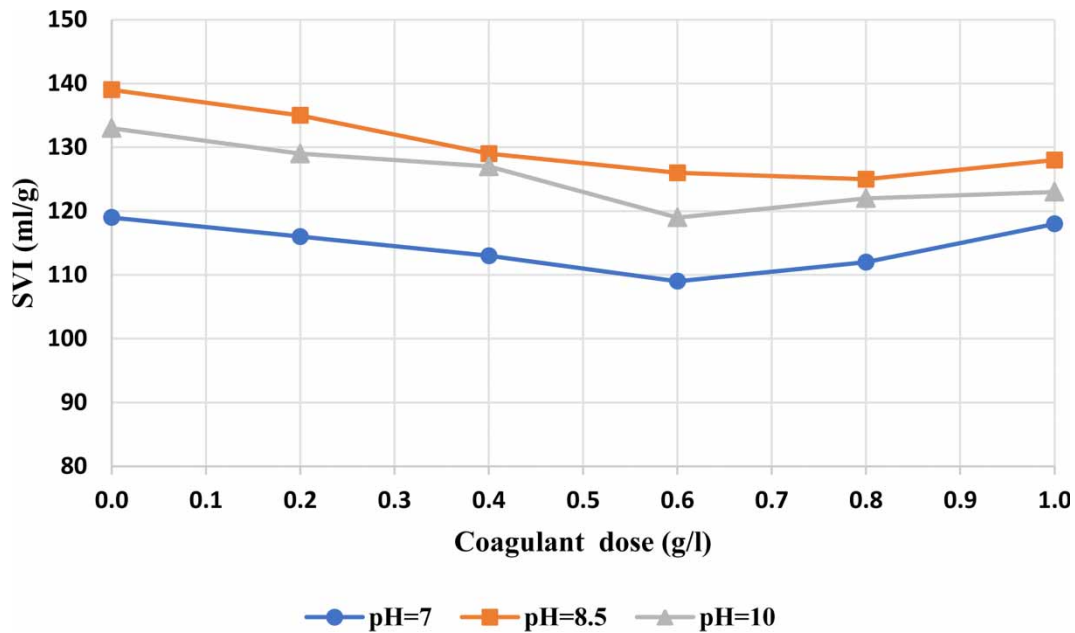


Figure 7 | Effect of different doses of trivalent iron sulfate coagulant (Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>) on the SVI of the sludge with pH of 7, 8.5 and 10.

Figure 8 shows the effect of injecting different doses of trivalent iron chloride coagulant on sludge at different pHs. Adding doses of 0.6, 0.8 and 1.0 of this coagulant to the sludge with pH of 7, 8.5 and 10 reduces the SVI by 8, 10 and 11%, respectively.



**Figure 8** | Effect of different doses of trivalent iron chloride ( $\text{FeCl}_3$ ) coagulant on the SVI of the sludge with pH of 7, 8.5 and 10.

The effect of optimal doses of coagulants on the dewatering of the inlet sludge of the gravity concentrating unit is summarized in Table 2. As can be seen, the addition of optimal doses of  $\text{FeSO}_4$  and lime reduced CST of sludge by 30.6 and 32.7% and also reduced the moisture content of sludge cake by 26 and 30.6%. Also, adding optimal doses of  $\text{FeCl}_2$ ,  $\text{Fe}_2(\text{SO}_4)_3$  and  $\text{FeCl}_3$  with the help of lime coagulant reduced capillary suction of sludge by 35.1, 27.3 and 15.3%, and sludge cake decreased by 36.6, 17.1 and 8.7%, respectively.

**Table 2** | The effect of optimal doses of coagulants on sludge dewatering

Parameter	Control sample	Sample (1)	Sample (2)	Sample (3)	Sample (4)	Sample (5)
Coagulant	–	$\text{FeSO}_4$	$\text{FeSO}_4$	$\text{FeCl}_2$	$\text{Fe}_2(\text{SO}_4)_3$	$\text{FeCl}_3$
Coagulant dose (g/l)	–	6.0	4.0	0.1	4.0	60.
Dosage of coagulant aid (CaO)(g/l)	–	6,964.0	5,986.1	5,986.1	5,986.1	5,986.1
SVI (ml/g)	133	82	77	76	95	108
CST (s)	19.4	91.2	82.2	72.2	0.3	55.3
Moisture content of sludge cake (%)	1.78	8.83	8.84	1.86	9.81	1.80
TSS (mg/l)	38	5	4	5	6	6

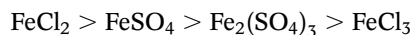
According to Table 2, it can be said that  $\text{FeCl}_2$ , compared to other coagulants used in this study, can achieve lower SVI and lower TSS. Accordingly, the use of  $\text{FeSO}_4$  and  $\text{Fe}_2(\text{SO}_4)_3$  coagulants is the next priority and  $\text{FeCl}_3$  is not a priority of use compared to other coagulants. Economic issues are also important in choosing a coagulant for WWTPs. Considering that the price of  $\text{FeCl}_2$  in Iran is lower compared to other coagulants, this coagulant can be suggested due to its better performance.

## CONCLUSION

In this study, the effect of divalent and trivalent compounds of iron (as coagulant) and also the change of pH by adding lime (as coagulant aid) on improving the dewatering of sludge entering the gravity thickener on the Bojnourd WWTP was investigated. The use of iron-based coagulants with increasing pH had a significant effect on improving the dewatering of incoming sludge to the gravity thickener. The optimal doses of  $\text{FeSO}_4$ ,  $\text{FeCl}_2$ ,  $\text{Fe}_2(\text{SO}_4)_3$  and  $\text{FeCl}_3$  with the help of a lime coagulant reduced the CST of sludge and decreased the amount



of sludge by 32.7, 35.1, 27.3 and 15.3%, respectively. Depending on the type of coagulant applied, the moisture content of the sludge cake also decreased by 30.6, 36.6, 17.1 and 8.7%, respectively. The addition of lime as an aid in the use of three divalent and trivalent iron sulfate coagulants and also trivalent iron chloride by reducing the dose of coagulant and in the use of divalent iron chloride coagulant leads to a significant increase in the efficiency of dewatering. Dilution water injection did not have a significant effect on the sludge dewatering process when adding coagulants. According to the results and based on the optimal doses of coagulants and pH of sludge, the ranking of the coagulants tested is



## DATA AVAILABILITY STATEMENT

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

## CONFLICT OF INTEREST

The authors declare there is no conflict.

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