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Coagulation/flocculation optimization and sludge production for pre-treatment of paint industry wastewater

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ABSTRACT

This study examined the optimization of paint industry wastewater pre-treatment by means of coagulation and the quantity of produced sludge generated. In preliminary tests, high concentrations of alum, PACI, and $FeCl_3$ were used, and removal efficiencies were determined to be 86–88%, 100%, and 46–72% for chemical oxygen demand (COD), SS, and color, respectively. Actual studies were conducted by adding lower concentrations of coagulants to reduce the produced sludge quantity due to the addition of coagulants at high concentrations. In those studies, COD removal efficiency decreased by 10%, while suspended solids and color removal increased. The optimum removal efficiency was obtained by adding 250 mg/L alum and $FeCl_3$, and the optimum anionic polyelectrolyte dose was found to be 4 mg/L. When equal amounts of coagulants were dosed, the quantities of sludge were found to be $FeCl_3 > PACl > FeSO_4 \geq$ alum. Considering the treatment efficiency and quantity of sludge together, the most economical solution with 0.077 \$/m^3 wastewater was obtained by $FeSO_4$. The cost of sludge disposal in the wastewater treatment plant is remarkable. The amounts of sludge should also be considered along with the removal efficiency during the detection of suitable treatment alternatives.

Keywords: Optimization; Coagulation–flocculation; Paint industry; Sludge volume

1. Introduction

The paint industry is one of the primary manufacturing processes in developing countries. By the year 2015, the global paint need is estimated to be 45.6 million tons. While paint consumption decreases around the world, the demand is expected to increase by 10.4% in the Asia/Pacific region [1]. A wide range of paint and by-products that can be used in different environments and materials are manufactured by the paint industry, using pigment, solvent, binder, and additives mixtures. Potential environmental risks can be reduced through determination and minimization of the wastes produced during paint production [2]. Highly polluted wastewaters are produced during the washing of intermittently operated mixer systems and other equipment in the paint industry [3]. Due to the varying degree of chemicals being used, the wastewater contains considerable amounts of concentrations of BOD, chemical oxygen demand (COD), suspended solids (SS), heavy metals, toxic compounds, and color.

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The chemicals used in paint and dye material manufacturing plants lead to pollution in the environment and aquatic environment [4].

Various methods are successfully used in treatment of paint industry wastewater. The most commonly used methods are coagulation/flocculation [5–7], electrochemical methods [8,9], adsorption [10,11], oxidation [12], filtration [13], membrane [14], and advanced oxidation processes [15,16]. However, the most suitable method should be defined, as the composition of paint industry wastewaters varies.

The coagulation/flocculation process is widely used in pre-treatment of industrial wastewaters [17]. To this end, alum, iron [18], lime [19], PACl [20,21] coagulants, and anionic, cationic, and nonionic polyelectrolytes are added to the wastewater. The products being formed by the hydrolysis of metal salts used in coagulation/flocculation process react to colloidal particles by three mechanisms. These mechanisms are load neutralization, adsorption, and sweep flocculation. At first, the negative loads of dissolved organics and colloids in the water are neutralized, and particles co-precipitate. Meanwhile, adsorption occurs due to the changing load after neutralization on colloidal surface. Thus, floccules co-precipitate and suspended forms in water environment are trapped (sweep flocculation). Therefore, the quantity of sludge produced in the coagulation/flocculation process directly depends on the chemicals used, the quantity of water in the sludge, and the settleable solids in untreated water. Particularly, the coagulants that are used for treatment significantly affect the amount of sludge produced. For an accurate treatment approach, apart from wastewater characterization and treatment efficiency, the control and minimization of sludge should also be taken into account [22]. In the coagulation/flocculation process, sludge in the form of metal hydroxides is generated firstly by the use of inorganic coagulants [23]. Due to different molecular weights of $Al(OH)_3$ and $Fe(OH)_3$, Al-based coagulants generate 20–50% less sludge than Fe-based coagulants do. In some particular applications, aluminum chlorohydrate and polyaluminum chlorides are 25–30% more effective than alum, and the amount of sludge produced decreases in uses of the same quantity of coagulants. However, in order to reduce the quantity of sludge, in situ dewatering can be applied before disposal. In such cases, selecting a suitable coagulant will enhance dewatering efficiency and will significantly reduce total sludge quantity and disposal costs. Although a high level of efficiency is obtained from removal of paint industry wastewaters, there is a limited body of research on the quantity of sludge produced during coagulation/flocculation. The produced sludge in treatment processes leads to secondary problems, and sludge disposal costs exceed treatment costs [24].

Optimum conditions of coagulation/flocculation process that is frequently used in treatment of paint industry wastewaters should be determined. On the other hand, it will not be sufficient to determine an appropriate treatment method only by identifying removal efficiency. This study will determine the most economic pre-treatment alternative through examination of the quantity of sludge that is generated during the coagulation/flocculation process.

To this end, pollution characteristics of paint industry wastewaters were identified; COD, SS, and color removal efficiencies were determined by jar testing which used alum, PACl, $FeCl_3$, and $FeSO_4$ for removal. The most economic method was determined by identifying the quantity of produced sludge in all tests.

2. Materials and methods

2.1. Wastewater

Wastewater samples used in coagulation/flocculation studies were supplied from a balance tank where water used for washing in a paint factory in Istanbul is collected. Color and pollution loads of the wastewater generated in intermittently operated reactors showed variations. Firstly, COD, color, SS, and turbidity analyses were conducted for the characterization of wastewater. Wastewater analysis results are presented in Table 1.

Furthermore, turbidity measurement was conducted for the sample, and the result was found to be greater than 5,000 NTU.

2.2. Jar test

The jar test was applied to determine the effectiveness of various coagulants applied. Coagulation/flocculation studies were performed on a four-pedal jar test setup (lovibond® floc-tester ET 740) that permitted a programmable mixture. Numerous studies were

Table 1
Characteristics of paint industry wastewater

Parameters	Value
COD, mg/L	664–2,955
TSS, mg/L	220–2,375
Color, pt-co	443–2,719
SS, mL/L	6–800
pH	7.05–7.15

performed in jar test studies to determine the optimum coagulant dose. The tests were conducted intermittently in 1-L glass jars. Rapid mixing at 100 rpm/min for 5 min, slow mixing at 20 rpm/min for 30 min, and 60 min precipitation time were preferred for jar test procedure before sampling [25]. In preliminary studies, alum, PACl, and iron chloride at the concentrations of 500, 1,000, 2,000, and 4,000 mg/L, were used as coagulant. In the next stage, the amount of coagulant was decreased, and 50, 100, 250, 500, 750, and 1,000 mg/L alum, PACl, iron chloride, and iron sulfate were used. To ensure floccules formation, anionic polyelectrolyte at 1, 2, 3, 4, and 5 mg/L different doses were added. 6 N sodium hydroxide (NaOH) and 2 M sulfuric acid (H_2SO_4) were used for pH adjustment of wastewater samples.

2.3. Analytical methods

Turbidity was measured through a portable Hach Turbidimeter Model 2100 N, manufactured by Hach company. A portable electrode pH meter Orion 4 Star manufactured by Thermo was used for pH measurements. COD, total solids, volatile solids, settleable solids, and SS were measured in accordance with the standard methods [26].

2.4. Chemicals

Alum ($AlSO_4 \cdot 18H_2O$), iron (III) chloride ($FeCl_3 \cdot 6H_2O$), iron sulfate ($FeSO_4 \cdot 7H_2O$), and polyaluminum chloride (PACl) used in experimental studies were obtained from Sigma-Aldrich at analytical purity. NaOH and H_2SO_4 (Merck) at analytical purity were used for the pH neutralization of raw wastewater. Polyelectrolytes of two different characteristics, including powder anionic polyelectrolyte and powder cationic polyelectrolyte, were used.

3. Results and discussion

Firstly, paint industry wastewater contains high amounts of SS, and these were removed from the wastewater by settling in the appropriate retention time. With the precipitation of wastewater samples without any application of coagulation/flocculation, a slight decrease was observed in SS (35%) and COD (20%) values; however, the color content remained unchanged. It was observed that the pollution load in wastewater resulted from dissolved organics in the liquid phase rather than the suspended inorganic content.

At the beginning of experimental studies, the pH value of the wastewater was adjusted to 4.5, 7, and 10 and the variation of COD, SS, and color parameters was determined (Table 2).

While 10–20% COD and 30–40% SS removal were obtained by the addition of acid–base used for adjusting the pH of the wastewater, color parameter remained unchanged. Especially in basic pH values, metal content is known to decrease with the increase of OH^- forms; however, organic content remained unchanged [27]. On the other hand, the quantity of sludge produced during the neutralization of wastewater was quite high.

In coagulation/flocculation studies, pH is quite important and directly affects removal efficiency [28]. Solubility of metal hydroxides varies with pH. The pH value of the medium affects the solubility of the chemicals used in coagulation in water environment and particularly the generation of metal hydroxides. When aluminum and iron salts dissolve in water, $Fe(OH)^{2+}$, $Fe(OH)_2^+$, $Fe(OH)_3$, $Fe(OH)_4^-$ and $Al(OH)^{2+}$, $Al(OH)_2^+$, $Al(OH)_3$, $Al(OH)_4^-$ etc. forms are formed depending on pH value. Thus, organic substances in water are eliminated by load neutralization and adsorption mechanism [23]. 6.0–8.0 and 7.5–8.5 ranges are given as the lowest solubility values for alum and PACl, respectively [29]. Alum is least soluble at 6.0. This means that at pH 6.0, the maximum amount of coagulant is converted to solid-phase floc particles. At pH values higher or lower than this pH of minimum solubility, dissolved Al levels in the treated water will increase. PACl is more soluble and has a higher pH of minimum solubility than alum. To determine the optimum pH value taking sludge generation into account, pH was determined as 8 for alum and $FeSO_4$ and as

Table 2
The changes of COD, SS, and color at different pH values

Parameters	Preliminary tests			Actual studies		
	pH = 4.5	pH = 7.0	pH = 10.0	pH = 4.5	pH = 7.0	pH = 10.0
COD, mg/L	628	664	648	2,845	2,955	2,830
SS, mg/L	202	220	360	1,325	2,375	2,290
Color, pt-co	707	443	537	2,077	2,719	2,706

8.5 for $FeCl_3$. In similar studies [30], pH value was preferred as 8.5 for alum and $FeSO_4$. On the other hand, higher pH values are known not to increase removal efficiency [31]. In the coagulation, SS are destabilized by changing the water pH and profiting that zeta potential tends toward zero when the isoelectric point is approached [32].

3.1. Preliminary tests

In preliminary tests, the jar test study was conducted by adding alum, PACl, and $FeCl_3$ at high concentrations into paint industry wastewater at optimum pH value. The variation in COD, SS, and color parameters with the increasing coagulant quantities is presented in Figs. 1–3. COD and color removal efficiency increased by the addition of coagulants at high concentrations.

86–88% COD, 100% SS, and 46–72 color removal efficiency levels were achieved in studies in which coagulant dose was increased to 4,000 mg/L. Similar

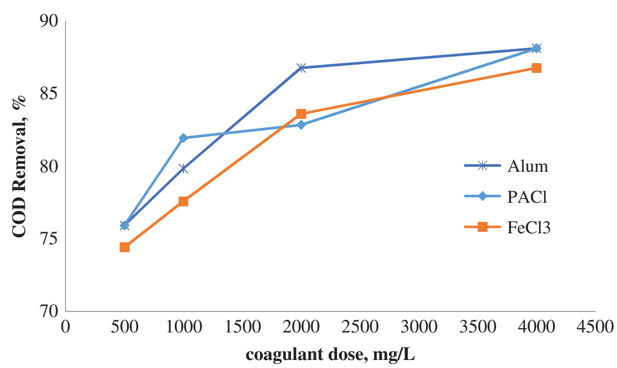

Fig. 1. The variation in COD removal with the addition of different quantities of coagulants.

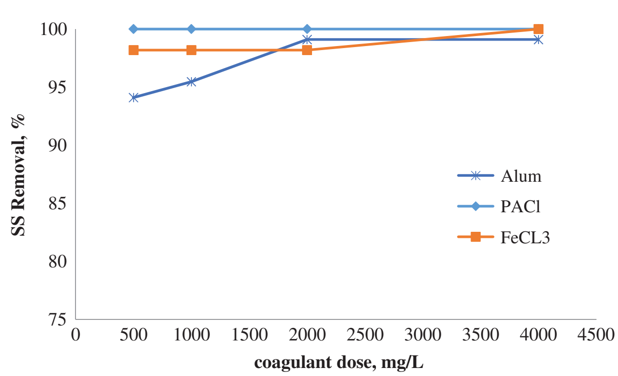

Fig. 2. The variation in SS removal by the addition of different quantities of coagulants.

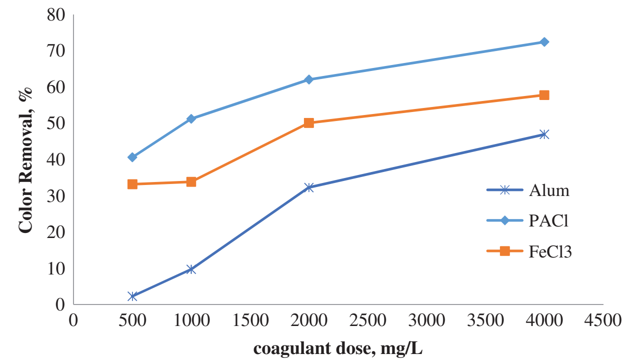

Fig. 3. The variation in color removal by the addition of different quantities of coagulants.

efficiency values were obtained for all coagulants in COD and SS removal efficiency. However, removal efficiency was found to be 72% with PACl in color removal. The quantity of sludge produced in preliminary studies was significant. On the other hand, increasing the coagulant dose from 500 mg/L to 4,000 mg/L did not significantly increase removal efficiency values. Therefore, treatability tests were conducted by means of coagulants at lower concentrations.

3.2. Actual studies

In follow-up studies, the coagulant dose that was previously applied to the wastewater at high concentrations was reduced. Alum, $FeSO_4$, PACl, and $FeCl_3$ at 50, 100, 250, 500, 750, and 1,000 mg/L concentrations were used. The amount of sludge produced would decrease by adding chemicals at lower concentrations. However, we aimed to achieve the necessary efficiency level. In these studies, turbidity was identified in addition to COD, SS, and color parameters. The variation of COD, SS, color, and turbidity removal efficiency with the coagulants added to the wastewater is presented in Figs. 4–7.

Addition of coagulant dose at higher concentrations than 250 mg/L did not significantly change the treatment efficiencies. Although coagulant dose used in previous studies was 10 times lower, COD removal efficiency decreased by 10%. Just as in preliminary studies, the SS efficiency rate (100%) was very high in these studies. However, COD removal efficiency decreased to 76%, while color removal efficiency increased to 99%. The fact that color parameter decreases with lower concentrations of coagulants can be explained by the color stemming from the coagulants. Similar to COD, SS, and color removal, turbidity removal efficiency was also examined. 700–750 mg/L

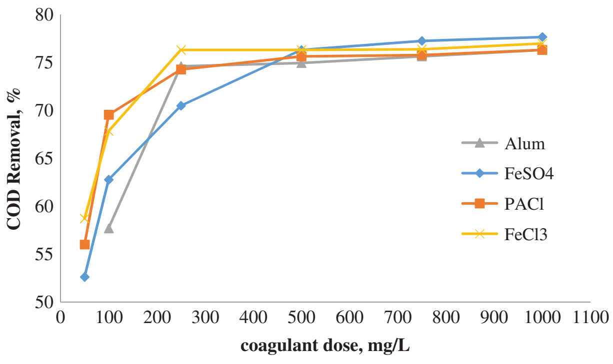

Fig. 4. The variation of COD removal efficiency in wastewater by the addition of lower doses of coagulants.

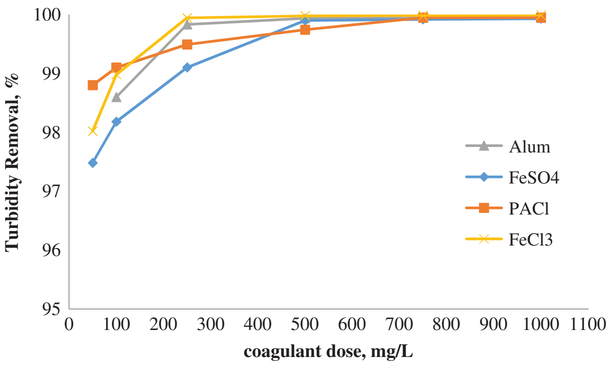

Fig. 7. The variation of turbidity removal efficiency in wastewater by the addition of lower doses of coagulants.

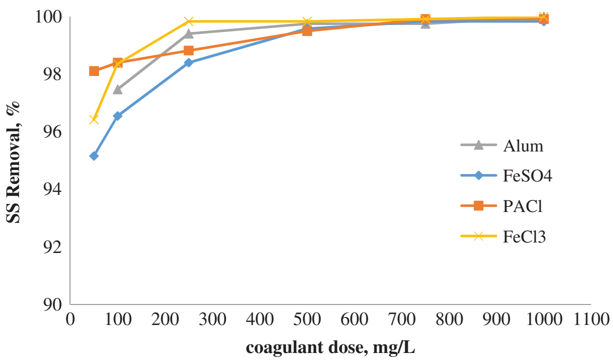

Fig. 5. The variation of SS removal efficiency in wastewater by the addition of lower doses of coagulants.

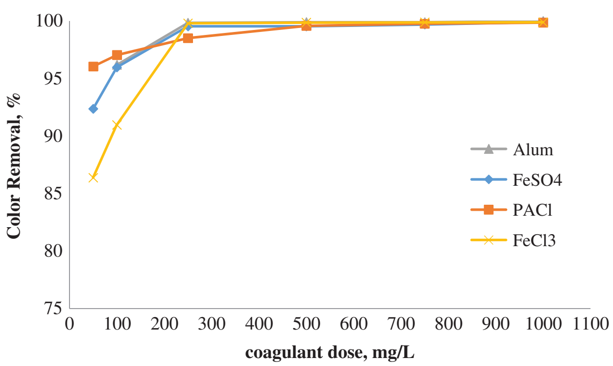

Fig. 6. The variation of color removal efficiency in wastewater by the addition of lower doses of coagulants.

COD, 14–28 mg/L SS, and 6–13 mg/L color contents were identified in treated water sample. However, significantly high removal efficiencies were observed in $FeSO_4$ and PACl at 500 mg/L coagulant dose. As for turbidity, despite the high treatment efficiency, a high turbidity value was observed in treated water for iron sulfate and PACI in comparison with other coagulants such as 45 and 25 NTU, respectively.

By reducing coagulant dosage, COD removal obtained in preliminary studies (90%) decreased in these studies (75%). Although color, SS, and turbidity decreased with low coagulant addition, dissolved contaminants remained. Removal efficiency obtained is important under the conditions in which pre-treatment alternatives are determined.

It was found that coagulant doses at concentrations higher than 250 mg/L did not significantly increase the removal efficiency values for all coagulant types. As for COD, the limit could not even be exceeded even at the levels with the highest dosing. Therefore, the treatment performed for this sample can be used as a pre-treatment. Then, a different treatment system is needed for COD removal.

An analysis of removal efficiency values obtained in experimental studies have revealed that 70–76% COD removal, 98–99% SS removal, and 98–99% color removal were determined by adding 250 mg/L coagulant. The highest treatment efficiency was achieved with iron chloride and alum among the coagulants at the same concentrations. When compared to previous studies, it was observed that high removal efficiencies were achieved despite the low amounts of chemicals used (Table 3).

The suitable polyelectrolyte amount was determined in coagulation tests which used alum. COD, SS, color, and turbidity removal efficiency with varying concentrations of anionic polyelectrolyte were determined at optimum coagulant dose (Fig. 8).

Optimum coagulant dose was reported as 4 mg/L in studies on polyelectrolyte dose. 1 and 2 mg/L doses, especially in COD removal values, gave repetitive results. COD removal efficiency increases by adding 3 and 4 mg/L polyelectrolytes. However, increasing polyelectrolyte doses also increased the

Table 3
Comparison of removal efficiency values in treatment of paint industry wastewater

Coagulant	Dose (mg L^{-1})	Optimum pH	Removal efficiency	References
Alum	250	8	75% COD	The present study
$FeSO_4$	500	8	76% COD	The present study
$FeSO_4$	2,000	9.7	65% COD, 93% Turbidity	[5]
$Al_2(SO_4)_3$	2,500		89% COD, 88% Turbidity	[5]
PACl	4,000	7	96% COD, 98% Turbidity	[5]
Alum	700	7.5	74% COD, 99.6% Turbidity	[13]
Sodium Bentonite	500		62% COD	[6]
$FeCl_3$	650	8–9	82% COD, 94% Color	[7]
Alum	300	6	55% COD	[24]
$FeCl_3$	500	5	59% COD	[24]
$FeSO_4$	500	9	54% COD	[24]

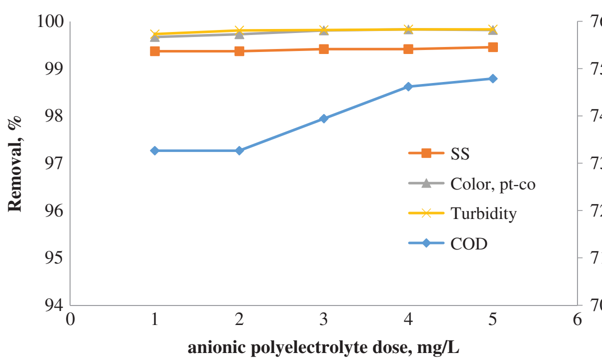

Fig. 8. The variation of COD, color, turbidity, and SS removal efficiency of wastewater by the addition of varying doses of anionic polyelectrolytes (coagulant; 250 mg/l Alum).

quantity of sludge and made selection of optimum dose difficult. As anionic polyelectrolyte dose increased, the settleable solid amount increased 18%, although removal efficiency showed no significant variation.

Combined use of alum and $FeCl_3$, which are the two coagulants giving the highest COD removal efficiency in wastewater, was analyzed. The COD removal ratio achieved, especially by the use of coagulants individually, increased from 76 to 86%. Only color value was slightly higher than those reported in studies which used coagulants individually. The quantity of sludge produced nearly equaled to the quantity of sludge produced by the use of iron chloride only.

3.3. Sludge production

We obtained significantly high removal efficiency values with the coagulants used in this study during the pre-treatment of paint industry wastewater through coagulation. However, as the coagulant dose increased, the settleable solid amount increased as well. To determine the suitable coagulant, the quantity of sludge produced should also be taken into account along with removal efficiency. The quantity of sludge produced in preliminary and actual studies was measured and is presented in Figs. 9 and 10.

The produced sludge amount was observed to be increased due to coagulant dose in preliminary studies. When compared to the sludge quantity produced in jar test, PACl > $FeCl_3$ > alum is observed. Characterization of the produced sludge was reddish color in powder form, gray color in flocculated form, and gray color in flocculated form, respectively, for alum, PACl, and iron chloride.

In actual studies, in parallel to the first study, increasing coagulant dose was observed to increase the quantity of sludge produced. While quantities of sludge generated at 250 mg/L coagulant dose were similar, quantity of sludge generated by PACl and $FeCl_3$ significantly increased as the coagulant dose increased. In the

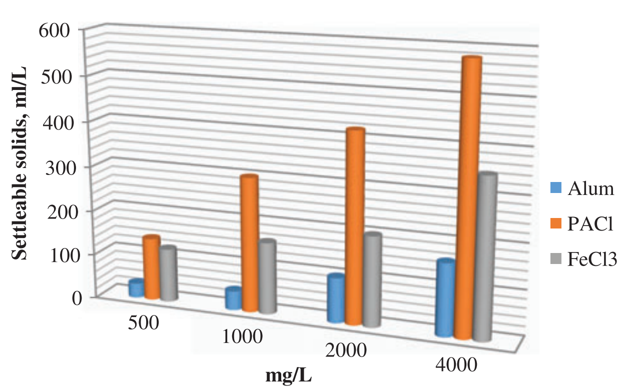

Fig. 9. The variation in sludge quantity with different coagulants in preliminary studies.

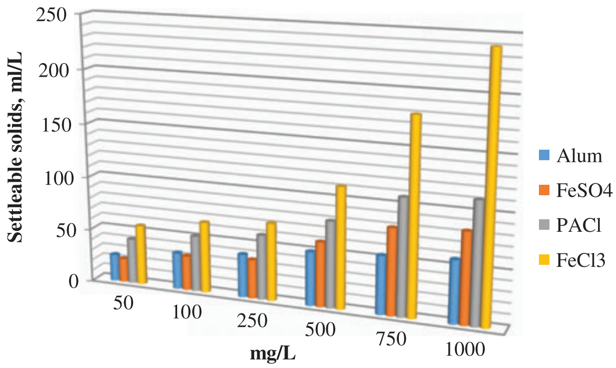

Fig. 10. The variation in sludge quantity with different coagulants in actual tests.

tests which used alum and $FeCl_3$ in combination, quantity of produced sludge remained unchanged. Comparison of quantities of sludge at equal coagulant doses showed $FeCl_3 > PACl > FeSO_4 \geq$ alum. Characterization of the sludge was gray, black, brown, and white colored for alum, iron sulfate, PAC, and iron chloride, respectively.

The settleable solids include 1 to 4% solid matter in coagulation process, and density of the produced sludge varies. Taking into account the density of the sludge is necessary in order to determine exactly the amount of sludge. In general, estimated sludge densities were higher at low coagulant dosages. The medium range belongs to ferric since sludge density is between 0.01 and 0.025 g cm^{-3}; finally, the lower density belongs to alum whose values are in the range of 0.002–0.007 g cm^{-3} [33].

3.4. Economic analysis

The cost of the coagulant doses which gave the optimum removal and the required cost for the removal of the produced generated was examined for economic analysis. The method preferred in economic analysis was based on the evaluation of the costs of the chemicals that are required to treat water at equivalent volume. Wastewater treatment costs in pre-treatment of paint industry wastewater were found to be 0.128 \$/m^3 wastewater for alum, 0.077 \$/m^3 wastewater for $FeCl_3$ and $FeSO_4$, and 0.157 \$/m^3 wastewater for PACl. Cost values include anionic polyelectrolyte cost 0.014 \$/m^3 wastewater used in coagulation tests.

Considering that the produced sludge has 100 \$/ton disposal cost, it should be evaluated with the costs of chemicals. Comparison of cost and efficiency values revealed that optimum treatment can be achieved by $FeSO_4$ with a suitable cost and low sludge quantity.

4. Conclusions

Pre-treatment of paint industry wastewater with the coagulation/flocculation method is effectively applied. It was observed that treatment efficiency did not increase by adding high amounts of (>2,000 mg/L) coagulants. Furthermore, a considerable quantity of sludge was produced. High removal efficiency values (>95%) were achieved by adding lower doses of chemicals (<500 mg/L) under appropriate working conditions. While alum and $FeCl_3$ gave the highest treatment efficiency, $FeSO_4$ gave the most economic treatment. Comparison of quantity of the produced sludge revealed that the most economical solution cannot be reached only by considering removal efficiency values.

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