

## **Performance Evaluation of Tile Wastewater Treatment with Different Coagulants**

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### **ABSTRACT**

**Introduction:** The objective of this study was to investigate wastewater quality and the efficiency of removal of wastewater contaminants produced by a tile factory by using ferric chloride, ferrous sulfate, ferric sulfate, aluminum sulfate (alum) and poly-aluminum chloride (PAC) coagulants.

**Materials and Methods:** This is an applied study. A composite sample was taken of the wastewater production line of the factory. Wastewater characteristics including pH, EC, temperature, turbidity, TSS, TDS, TS and COD were measured in accordance with the standard methods. In the next step, the jar-test experiment was used to investigate the effect of changing doses of coagulants (0.15, 0.2, 0.25, 0.3, 0.35 g/L) and pH values (7, 9, 11) on the removal of contaminants. The effective dose and optimal pH were thus selected and the best coagulant was later determined.

**Results:** The optimum pH of 11 was obtained for removal of contaminants by ferric chloride, ferric, ferrous sulfate and 7 for alum and PAC. The optimum concentration of the five coagulants was obtained at 0.3, 0.3, 0.3, 0.25, 0.25 g/L respectively. PAC, with the turbidity removal EC, TSS, TS and COD removal of 99.92%, 17.74%, 99.93%, 89.8%, 75% respectively, had best performance at lower doses and alum, ferrous sulfate, chloride, ferric and ferric sulfate were placed in the next rankings.

**Conclusion:** Among the five studied coagulants, PAC is the most effective coagulant, and then alum, ferrous sulfate, ferric chloride and ferric sulfate are placed. Given that PAC is more expensive than other materials, care should be taken to select the most appropriate coagulant.

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### **Introduction**

In recent decades, the growths of consumption and increasing industrial production have led to a rapid decline in available natural resources (raw materials and energy resources). On the other hand, high quantities of waste are being generated, such that most of them are not directly recyclable<sup>1</sup>. Water is used as an initial material in many industries and the resultant wastewater is discharged into the environment<sup>2</sup>. In order to achieve conditions for sustainable development, the alignment of industrial development with

environmental development is inevitable. Important environmental cases in industrial parks include the provision of the water required by industries and the entry of industrial contaminants into groundwater sources. The construction of appropriate wastewater treatment systems prevents the contamination of water sources and the environment, and also provides a new source of water for reuse<sup>3</sup>. Today, shortage of water for both drinking and industrial consumption is a matter of global concern for communities. Therefore, protection of water resources is very

important. There are many studies on minimizing water consumption to solve this problem in industries, with different approaches <sup>4</sup>. Water management is a very important issue in most industrial sections, considering the large amount of wastewater produced by them <sup>5</sup>. Water is a very important raw material in the tile manufacturing industries, with its usage varying greatly between sectors and processes <sup>6</sup>. Water is consumed for the operations of various units, including preparation and cleaning of equipment, such as slurry and glaze preparation, glazing lines, washing gases from scrubbers, etc. The major wastewater produced in these parts is only caused by washing <sup>7</sup>. A significant amount of suspended solids and the turbidity of wastewater produced by these industries could be removed just by simple sedimentation. As a result, the resultant effluent can only be returned to the body preparation part. But the quality of the recycled water is not enough for use in other parts, especially glaze preparation. As a result, a significant amount of groundwater enters the production line as the fresh water source for production of glaze and the preparation of other coatings. However, groundwater resources are becoming limited day by day. Tile industries should find a solution to decrease their groundwater consumption rate <sup>5</sup>. Water recycling and reuse in the consumption cycle not only reduce consumption and increase economic efficiency, but also preclude the discharge of wastewater contaminants into the surrounding environment.

This should, as a principle, be a priority to prevent the contamination of the environment. The wastewater composition includes clays, frits and insoluble silicates, electrolytes, anions such as sulfate (100–500 mg/L), chloride (100–700 mg/L), heavy metals such as lead and zinc, COD (150–1000 mg/L) and BOD<sub>5</sub> (50–400 mg/L) <sup>6, 8</sup>. Organic materials mainly come from the additives used in decorating the tiles <sup>5</sup>. In contrast, municipal wastewaters basically work based on biological treatment methods. Experience has shown that industrial wastewaters cannot be treated easily with these mechanisms alone in many cases. Therefore, different methods are required, based on

physicochemical steps. Physical-chemical treatment methods that can be used for tile wastewater include homogenization, aeration, sedimentation, filtration, activated carbon absorption, coagulation and flocculation, ion exchange and reverse osmosis <sup>9</sup>. Nowadays, the use of iron coagulants, especially aluminum, is very common in water and wastewater treatment, and the use of these materials is increasing. In addition, these are materials that are very cheap and easily accessible. The selection of the coagulation type is one of the most important decisions for the wastewater treatment and would be based on the nature of wastewater. Poly-aluminum chloride (PAC) has been proved to be more efficient in low dosages and acts in wider pH ranges <sup>10</sup>. Nilsalab used aluminum sulfate for the treatment of wastewater in the ceramic industry and reported the highest removal efficiency of turbidity at pH 6–7, with an optimal dose of 200 mg/L <sup>11</sup>. In another study on the treatment of wastewater from stone-cutting industries, Fahiminia et al. investigated the effects of different doses of coagulants, including aluminum sulfate (alum), poly-aluminum chloride, polymer, ferric chloride (FeCl<sub>3</sub>) and lime in removing turbidity, total suspended solids (TSS), and total solids (TS). The results indicated that lime in dose 25 ppm is the best coagulant for turbidity removal (99.8%) and 100 ppm alum had the highest efficiency for TS removal (82.5%) <sup>12</sup>. Paula et al. (2014) used a combination of alum and *Moringa oleifera* for the treatment of the wastewater of the concrete industry and the turbidity removal efficiency of 90% was obtained <sup>13</sup>. The aim of this study was to investigate the quality of wastewater and use of a coagulation-flocculation process using coagulants of ferric chloride, ferric sulfate and ferrous sulfate, alum sulfate and PAC to reduce suspended solids and turbidity of wastewater in order to reuse it in the processing line.

### Materials and Methods

The composite sampling method was used in this study on processing of line wastewater in accordance with shift work and taking into accounts its changes. The following parameters pH, EC and temperature were measured at the

sampling site since their values change over time. Samples were collected in 20-liter containers and transferred to the laboratory. A multi-parameter model HQ40 company HACH was used to measure the pH and EC and turbidity was measured using the turbidity meter TB100 model,

manufactured by Eutech. TSS, TDS, TS, COD and BOD<sub>5</sub> parameters were calculated in accordance with the procedures set out in the standard method book 14. Physical-chemical characteristics of the raw wastewater are listed in Table 1.

**Table 1:** Physical-chemical characteristics of raw wastewater tile industry

Parameters	Unit	Minimum	Maximum	Average $\pm$ SD
pH	-	8.2	8.6	8.3 $\pm$ 0.6
Temperature	(°C)	30	32	31 $\pm$ 1
EC	( $\mu$ S/cm)	2142	2700	2484 $\pm$ 299.57
Turbidity	NTU	9500	13300	11100 $\pm$ 1969.77
TDS	(mg/L)	1096	1246	1185.33 $\pm$ 79
TSS	(mg/L)	13450	34414	21221.33 $\pm$ 11485.45
TS	(mg/L)	14546	35628	22390 $\pm$ 11529.71
COD	(mg/L)	151.2	490	361.33 $\pm$ 183.66
BOD <sub>5</sub>	(mg/L)	100.8	392.5	266.51 $\pm$ 149.58

The study was performed on a laboratory scale using the jar test equipment with five coagulants of ferric chloride, ferric sulfate and ferrous sulfate, aluminum sulfate as metal salts and PAC as hydrolyzed aluminum salt. These materials were

produced by Aquatech Company, Switzerland. Hydrochloric acid 1 normal and lime Ca (OH)<sub>2</sub> solution were used to adjust the pH value of wastewater during the treatment processes. Details of these coagulants are described in Table 2.

**Table 2:** Details of coagulants used in this study

Coagulants	Formula	Molecular weight (g/mole)	No. Artie	Concentration (%)
Ferric chloride	FeCl <sub>3</sub> .6H <sub>2</sub> O	270.30	3943	10
Ferric sulfate	Fe <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> .H <sub>2</sub> O	399.88	3926	10
Sulfate ferrous	FeSO <sub>4</sub> .7H <sub>2</sub> O	278.02	3965	10
Aluminum sulfate	Al <sub>2</sub> (SO <sub>4</sub> ) <sub>2</sub>	666.42	1102	10
Poly aluminum Chloride	Al <sub>2</sub> (OH) <sub>n</sub> Cl <sub>6-n</sub>	-	-	10

The coagulation-flocculation experiments were carried out using a jar test manufactured by HACH (model 7790-402). The samples were placed at room temperature after being taken out from the refrigerator for two hours, until temperature reached 22°C. In order to determine the best sedimentation time before coagulation and flocculation processes, different sedimentation times were tested on the sample. In order to determine the optimum pH of coagulant materials, different pH levels (7, 9, 11) were evaluated in the fixed amount of coagulants (iron-based compounds 0.25 g/L and aluminum-based compounds 0.2 g/L). By measuring turbidity, EC, TSS, TS and COD parameters for each pH, the pH of the sample with the highest removal efficiency for the parameters

desired was considered the optimum pH. Then the wastewater pH was set at the optimum value and various amounts of coagulants (0.15, 0.2, 0.25, 0.3, 0.35 g/L) were simultaneously added to the 1 liter wastewater, and its optimal amount was determined. Wastewater and coagulants were stirred at room temperature, first with rapid mixing for 1 min at 100 rpm, and slow mixing for 10 minutes at 20 rpm. In the end, 30 minutes' sedimentation was considered for the sample. After the sedimentation stage, the wastewater supernatant was extracted into the beakers using a plastic syringe for purposes of chemical analysis. Finally, the optimal dose of each coagulant was determined. To draw the relevant diagrams, Excel 2010 was used. In this research, in order to

increase the accuracy of experiments, all experiments were repeated twice. The mean values were reported as the final result.

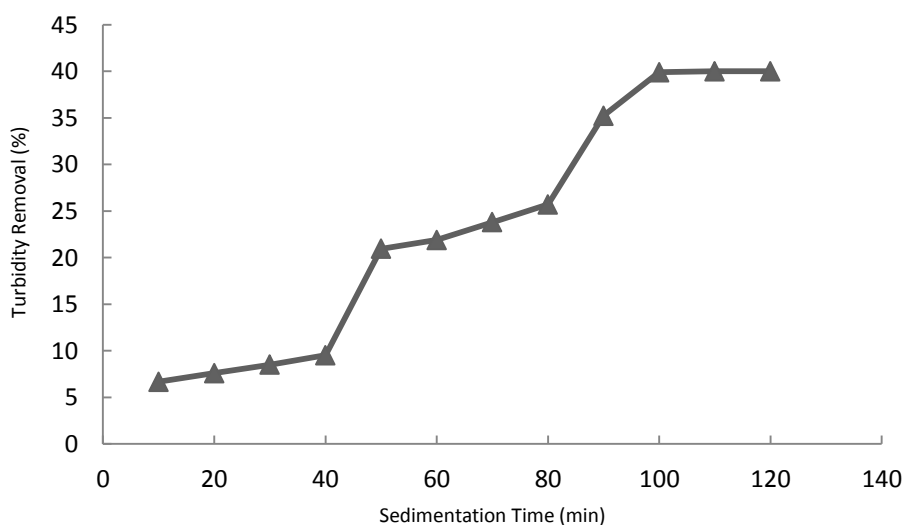
### Ethical issues

This study was conducted with the approval of Shahid Sadoughi University of Medical Sciences and Health Services, Medical Ethics Committee. Code: IR.SSU.SPH.REC.1394.15

### Results

Figure 1 shows the efficiency of turbidity removal during the sedimentation before the coagulation process. According to this diagram, different sedimentation times were tested on the tile raw wastewater before the addition of coagulants. The wastewater turbidity was decreased from 10500 to 6310 NTU at a sedimentation time of 100 minutes (turbidity removal efficiency of 39.9%). The efficiency of turbidity removal almost remained unchanged after 100 minutes. Figures 2 to 6 show the results of optimum pH about the coagulants used. Ferric chloride, ferric sulfate and ferrous sulfate were effective in alkaline pH and optimum pH was obtained at 11 by doing a jar test for each sample. The results of the experiments on alum and PAC coagulants showed that the optimum pH for these two coagulants was 7. Figures 7 to 10 show the effect of different doses of coagulants in removing contaminants. The results of the ferric chloride

coagulant experiment showed that it works well in the removal of the evaluated parameters in 0.3 g/L dosage and turbidity removal rate, EC, TSS and TS were respectively 99.84%, 20.46%, 99.83% and 90.09%, and COD removal rate was obtained at 50%. In the case of ferric sulfate, the results of different dosages indicate that the removal of the parameters studied at doses higher than 0.3 g/L had a constant process and the turbidity removal, EC, TSS and TS were respectively 99.69%, 22.45%, 99.71% and 90.23% and the COD removal rate of 72.5% was obtained. The results of the ferrous sulfate coagulant experiment showed that it performed well in 0.3 g/L in removing pollutants and the turbidity removal rate, EC, TSS and TS were respectively 99.9%, 26.47%, 99.9% and 90.51% and the COD removal rate of 61.2% was obtained. The result of effects of different doses of alum and PAC indicate that the removal of the parameters studied had a fixed trend from 0.25 g/L dose onward for both coagulants. As a result, 0.25 g/l dose was selected as the optimal dose for these two coagulants. The efficiency of turbidity removal, EC, TSS, TS and COD for aluminum sulfate was obtained at 99.88%, 24.95%, 99.86%, 90.44% and 60% respectively. The efficiency of turbidity removal, EC, TSS, TS and COD for aluminum sulfate for PAC was obtained 99.92, 99.93, 17.74, 89.8 and 75% respectively.



**Figure 1:** Effect of sedimentation before coagulation process for removal of turbidity

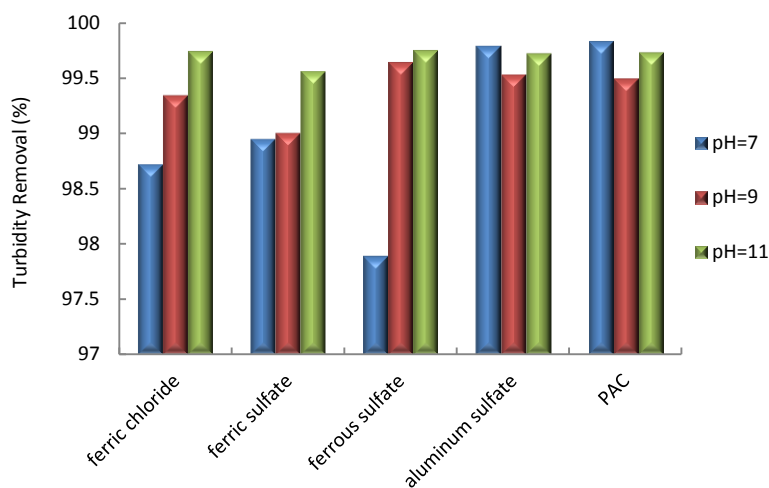


Figure 2: Effect of pH on removal efficiency of turbidity by various coagulants

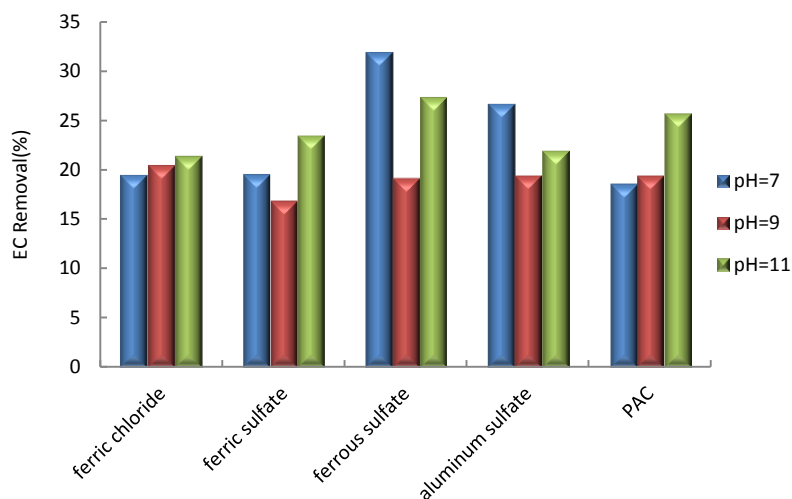


Figure 3: Effect of pH on removal efficiency of electrical conductivity by various coagulants

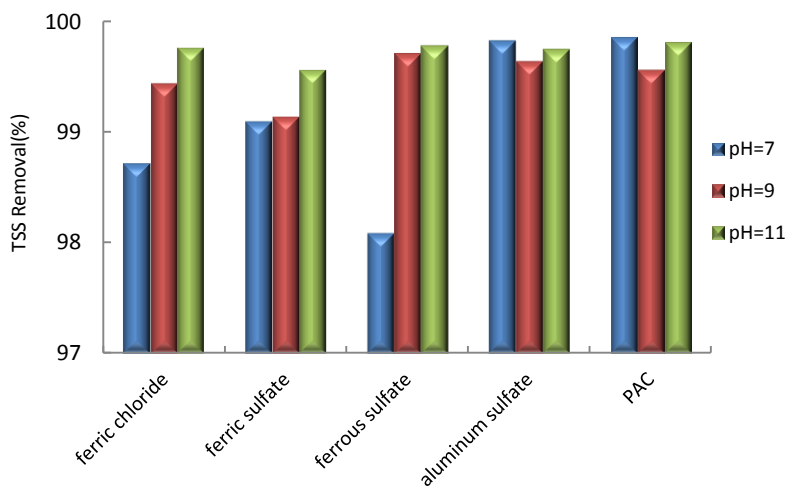


Figure 4: Effect of pH on removal efficiency of total suspended solids by various coagulants

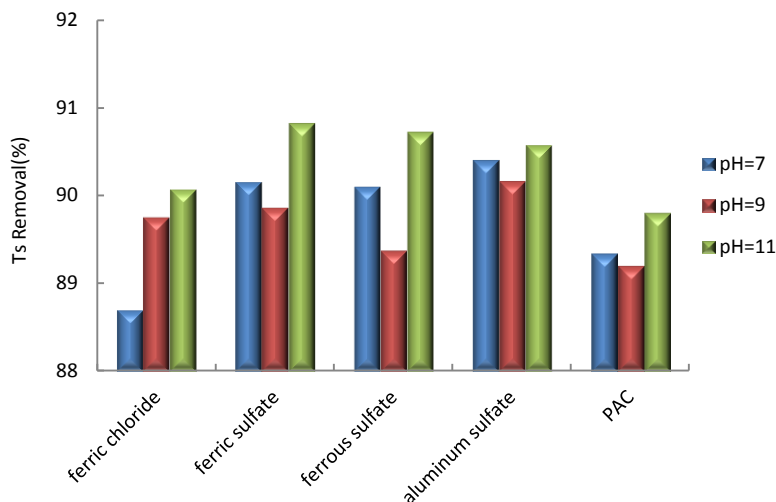


Figure 5: Effect of pH on removal efficiency of total solids by various coagulants

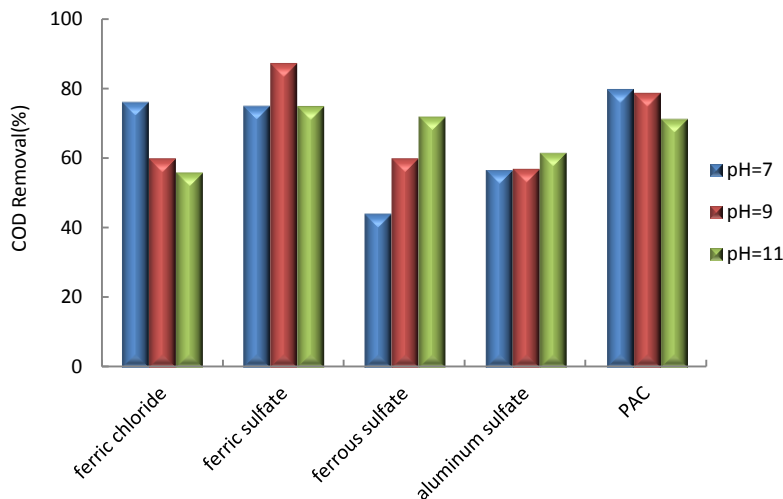


Figure 6: Effect of pH on removal efficiency on COD by various coagulants

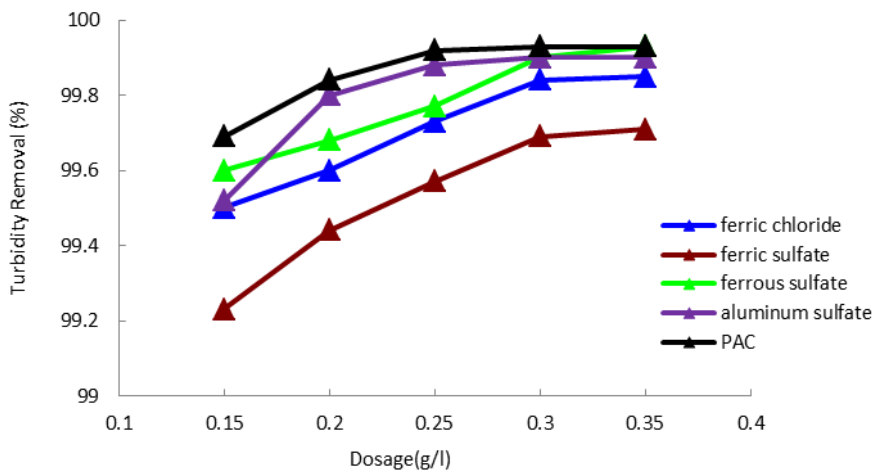
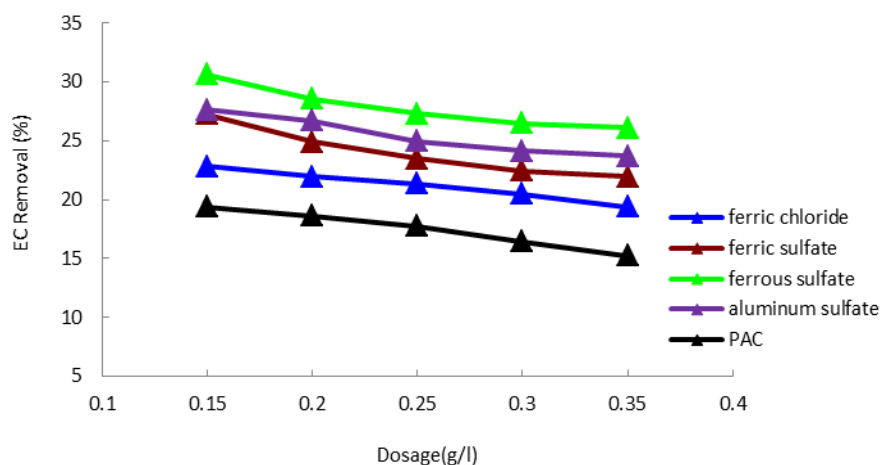
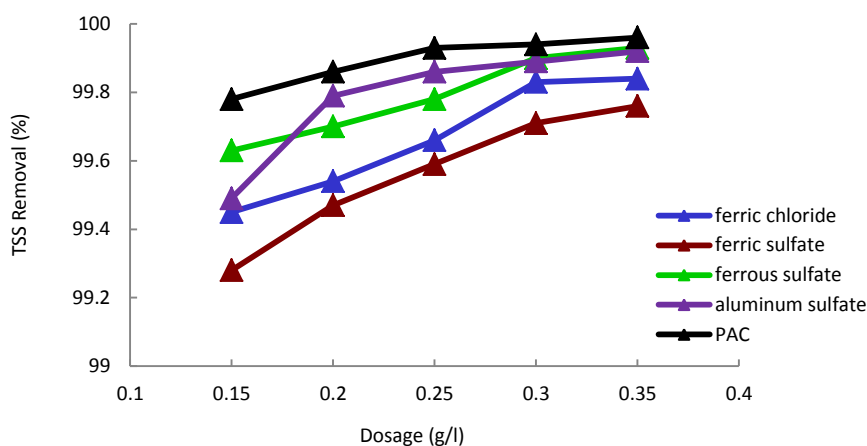


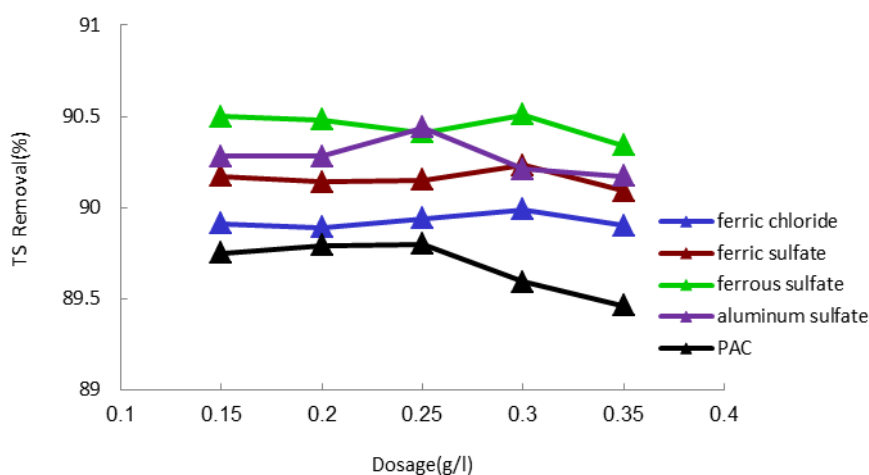
Figure 7: The effect of coagulant dosage on removal efficiency of turbidity



**Figure 8:** The effect of coagulant dosage on removal efficiency of electrical conductivity

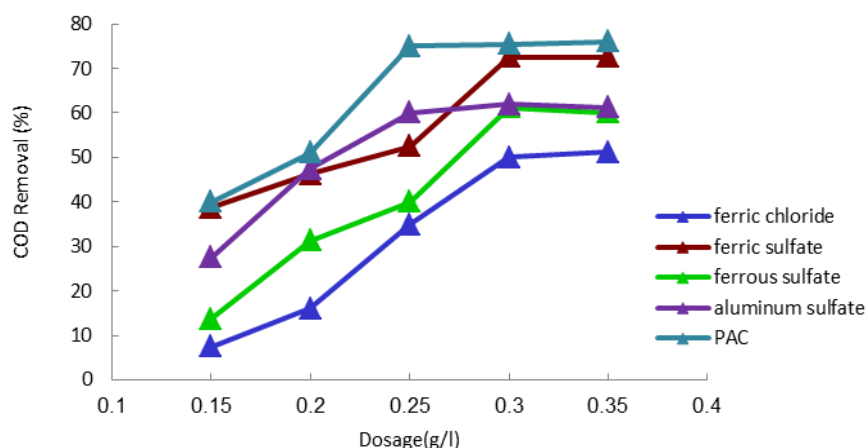


**Figure 9:** The effect of coagulant dosage on removal efficiency of total suspended solids



**Figure 10:** The effect of coagulant dosage on removal efficiency of total solids





**Figure 11:** The effect of coagulant dosage on removal efficiency of COD

## Discussion

According to Figure 1, the sedimentation time of 100 minutes was selected as the best time before the coagulation process. The results of the present research showed that aluminum sulfate and PAC had better performance at pH 7. The important reasons for such behavior include: 1- At low pH, presence of aluminum monomer particles causes neutralization of anionic particles of contaminants and better sedimentation is observed. 2- At low pH, concentration of dissolved aluminum decreased with decreasing  $\text{Al}(\text{OH})_4^-$ . Reduction in this ratio leads to an improvement of the sedimentation process and this anionic aluminum hydroxide reduces the effects of coagulation<sup>10</sup>. But less sedimentation and efficiency are seen at alkaline pH because of the formation of fine flocs. Aluminum sulfate and PAC coagulants in the lower dose showed higher turbidity removal efficiency, TSS, COD compared with other coagulants. The optimal pH and dose of aluminum sulfate were consistent with Nilsalab's studies<sup>11</sup>. However, in a study, Fahiminia<sup>12</sup> showed that ferric chloride had turbidity removal to the extent of 99.4% in 0.5 g/L dosage with much less turbidity, while in the present study, it showed higher removal efficiency of turbidity (99.84%) despite the turbidity being 16 times more than that of ferric chloride at dose 0.3 g/L. In a study, Paula<sup>13</sup> showed that aluminum sulfate had a removal

efficiency of 90% despite being combined with the other coagulant while in the present study, aluminum sulfate had a turbidity removal efficiency of only 99.88%. COD removal rate increased with increasing coagulants. The COD removal rate increased with increasing coagulants. These findings show that high doses of coagulants are required in order to achieve a significant COD removal rate. This could be because of the presence of large amounts of organic matter in effluents and their reaction with coagulants that causes the suspended matter in effluent to be oxidized, coagulated and eliminated. This process can reduce wastewater COD<sup>15</sup>. The results showed the electrical conductivity increases by increasing coagulants. But this increase is less when PAC is added to other coagulants. A comparison of the effects of these coagulants in removing contaminants showed PAC has a better performance compared to other coagulants. Being efficient at different pH levels, having better performance at lower temperatures, producing less sludge, and less need for pH adjustment are among the benefits of PAC that increased its consumption. In recent years, PAC has been used widely as an alternative to the aluminum sulfate and ferric chloride coagulants. It has been showed in practical applications that PAC has a coagulant effect two to three times better than conventional aluminum salts<sup>16</sup>. Given its lower consumption rate



under the same conditions, such as the initial turbidity and suspended solids, PAC use is ultimately economical.

### Conclusion

Coagulation and flocculation is a suitable method for removing wastewater turbidity and COD.

Owing to the high wastewater turbidity of tile industries, this method has a high potential for practical application in wastewaters with high COD and turbidity.

Considering its high efficiency, inexpensive costs and the lack of need for advanced technology, this method is recommended as a suitable solution for wastewater treatment in the production line of tile factories.

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### Conflict of interest

We have no competing interests.

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