Upgrading secondary wastewater plant effluent by modified coagulation and flocculation, for water reuse in irrigation
Mohammad Taghi Ghaneian, Mohammad Hassan Ehrampoush, Eghbal Ehsanzadeh, Ali Nikoonahad and Mohammad Miri

ABSTRACT
In this study, the feasibility of using coagulation, flocculation, and sedimentation (CF-S) for advanced treatment of secondary effluent released from the Yazd Intermittent Cycle Extended Aeration System was investigated. Four coagulants including ferric chloride (FeCl₃), polyaluminum chloride (PAC), ferrous sulfate (FeSO₄), and potassium ferrate (K₂FeO₄) along with Głog C-150 as flocculant polymer were used. In this study, returned chemical sludge was considered as a modification. Preliminary CF-S processes showed that FeSO₄ and K₂FeO₄ had low removal efficiencies. Thus, these two coagulants were abandoned and CF-S processes were continued only with PAC and FeCl₃ coagulants which had higher efficiencies in the removal of biological oxygen demand (BOD₅), chemical oxygen demand (COD), total suspended solids (TSS), and turbidity. Removal efficiency was higher when half of the chemical producing sludge was returned as compared with using both coagulants simultaneously along with 2 mg L⁻¹ of C-150 as flocculant. In the optimum dosage, when half of PAC and FeCl₃ sludge were returned, the volume of produced sludge was reduced by 40% and 28%, respectively, as compared without returned sludge. For the PAC coagulant in the optimum dosage with half of the sludge returned, all 2012 EPA standards of irrigation were met for both ‘processed and non-processed type’ agricultural crops.

Key words | advanced treatment, upgrading secondary effluent, water reuse, Yazd-ICEAS

INTRODUCTION
The city of Yazd, with a mean annual precipitation of 60 mm, is known as one of the most arid desert regions in Iran, where water shortage is considered to be a critical challenge. Climate change, water resources shortage, and increasing rate of conversion of water into wastewater due to population growth have led to serious consideration of water reuse. Reclaimed water can be used in different sectors, such as industry, recreation, and irrigation. The reuse of water has created great motivation to use innovation technology for upgrading wastewater effluent regarding agricultural irrigation (Löwenberg et al. 2014). In some parts of the world, especially in developing countries including Iran, where advanced technologies of wastewater treatment are not available, simple available technologies should be applied for advanced treatment of wastewater to be used in irrigation in agricultural sectors (Üstün et al. 2011; Väänänen 2014). Sustainable water resource development is an emerging issue in water and/or wastewater treatment (López-Maldonado et al. 2014). Optimized coagulation as well as new process alternatives are required for better removal of pollutants from wastewater (Matilainen et al. 2010). The size of a particle could affect how fast it sinks or floats, how it interacts with other particles, as well as many other properties of interest such as its hydrodynamics, transport, and aggregation in separation processes (Aguilar et al. 2003).
This study was conducted to determine the feasibility of coagulation, flocculation, and sedimentation (CF-S) processes for upgrading secondary effluent discharged from the Yazd Intermittent Cycle Extended Aeration System (Yazd-ICEAS). The purpose of this study was to achieve the best coagulants, most effective technique, and optimum concentrations of coagulants used to reach EPA standards and the conditions specified by the regional employer (Yazd Water and Wastewater Company).

**MATERIALS AND METHODS**

**Analytical procedure and materials used**

Accessibility and ease of use were the two main factors considered in selection of the chemical materials. The four coagulants used in this study were ferric chloride (FeCl₃, purity: 98%, Merck), polyaluminum chloride (PAC, 99% ±2, Merck), ferrous sulfate (FeSO₄, 99% ±1, Merck), and potassium ferrate (K₂FeSO₄, 97%, Sigma-Aldrich). C-150 was the polymer flocculant used in this study, with a purity of 97% (Aqua Tech, Portugal), which is referred to as C-150 hereafter. A comparative study by Yazd Water and Wastewater Company had previously confirmed the high efficiency of C-150 polymer flocculant in the Yazd-ICEAS treatment plant. Accordingly, we did not conduct any further investigation on other flocculants and C-150 was used as the sole flocculant in the present study.

In this study, both before and after each stage of CF-S process, conductivity and pH were measured by a conductivity/TDS meter and a pH-meter using a Hach HQ40d multi meter (Hach Co., Loveland, CO, USA). Turbidity was measured using a Hach 2100P turbidity meter. Hach DR5000 UV-Vis spectrophotometer was used to determine color, chemical oxygen demand (COD), and suspended solids (SS). Instruction standard method of 5210-B was used to determine biological oxygen demand (BOD₅). Fecal coliform (FC) before and after standard method of 5210-B was used to determine biological oxygen demand (COD), and suspended solids (SS). Instruction spectrophotometer was used to determine color, chemical using a Hach 2100P turbidity meter. Hach DR5000 UV-Vis (Hach Co., Loveland, CO, USA). Turbidity was measured TDS meter and a pH-meter using a Hach HQ40d multi meter.

**Sampling and CF-S process**

Sampling, testing, and CF-S process were performed in winter (the coldest season in Yazd), representing the worst possible efficiency situation for Yazd-ICEAS treatment plant effluent to ensure generalization of the results to any other conditions of effluent discharged from Yazd-ICEAS. Jar test experiments and sedimentation were used as a batch reactor in order to perform CF-S processes. The samples were transferred to the laboratory and different CF-S processes using varying coagulants and C-150 flocculant were performed. A total of 80 samples from Yazd-ICEAS effluent treatment plant were taken for laboratory analysis. About half of the samples were used in the process of a pry test to achieve optimum conditions for determining the duration and velocity of slow and fast mixing, as well as optimum conditions for the sedimentation term.

The survey was conducted on the actual wastewater discharged from Yazd-ICEAS treatment plant. In other words, pH, concentration, and dilution were not artificially defined. According to previous studies, changing wastewater pH requires a lot of chemicals due to abundant organic matter and thus it is not economical (Ismail et al. 2012; Chon et al. 2012). Polymer flocculant is not much affected by pH changes of water and wastewater (Ozkan 2003; Ozkan & Yekeler 2004).

The CF-S process was conducted in several stages for each of the coagulants to achieve optimum condition. Eventually, after several pre-test operations, coagulant concentrations between 5 and 55 mg L⁻¹, rapidly mixed (150 rpm) for 1 min, with a paddle velocity of 30 rpm for 10 min and settling for 15 min were selected as the optimum settings. These settings were selected as the base for all CF-S processes. In a study on the treatment of raw wastewater, PAC and ferrous sulfate were used with flash mixing started at 350 rpm and continued for 1 min along with addition of coagulants with dosages of 50, 60, 80, 100, and 120 mg L⁻¹ (Ismail et al. 2012). In another study on the treatment of raw wastewater, a series of jar test experiments were performed at 100 rpm for 1 min and 50 rpm for 20 min followed by 30 min for settling. In the mentioned study, concentrations between 150 and 450 mg L⁻¹ of alum were applied at pH ranging from 4 to 10 and room temperature (Guida et al. 2007). Given the difference between used wastewaters (secondary treated versus raw), the coagulant concentrations used in the mentioned studies were different (clearly higher concentrations compared to those used in the current study). In a study on tertiary wastewater
treatment, FeCl₃ and PAC were used at concentrations between 25 and 150 mg L⁻¹ (Üstün et al. 2011). The results of a study performed on secondary wastewater effluent showed that the most optimum conditions of PAC for removal of turbidity and microbial parameters were obtained at the pH of 8, flash mixing of 120 rpm, and optimal concentration of 30 ppm (Mirzaei et al. 2011).

**RESULTS AND DISCUSSION**

**Selection of the most effective coagulants**

In the present study, four coagulants of FeCl₃, FeSO₄, K₂FeO₄, and PAC were used to perform CF-S processes. The BOD₅, COD, turbidity, and total suspended solids (TSS) removal efficiencies were used as performance criteria to choose the best coagulants. Figure 1 illustrates TSS removal efficiency by each of the four coagulants. The percentage of TSS removal by K₂FeO₄, especially in lower dosages, was much lower than the other three coagulants. However, in general, TSS removal efficiencies by FeCl₃ and PAC (especially in high concentrations) were higher compared to those obtained using K₂FeO₄ and FeSO₄. Turbidity removal efficiencies in different dosages of the four coagulants are presented in Figure 2. The differences in turbidity removal efficiencies by various coagulants were slightly higher compared to TSS. The ordered turbidity removal efficiencies, respectively, belonged to PAC (60–90%), FeCl₃ (30–70%), K₂FeO₄ (0–45%), and FeSO₄ (5–40%). Figure 3 shows BOD₅ removal efficiencies by four different coagulants. As indicated in this figure, both PAC and FeCl₃ coagulants had better efficiencies at all concentrations compared with FeSO₄ and K₂FeO₄. The results of COD removal efficiencies by different coagulants are shown in Figure 4. As can be seen, at all coagulant concentrations, COD removal efficiencies by PAC and FeCl₃ are higher compared to FeSO₄ and K₂FeO₄. While the results of the current study presented in Figures 1–4 show K₂FeO₄ removal efficiency to be lower than the other three coagulants, a previous study reported removal efficiencies of 95%, 90%, 37%, and 15%, respectively, for turbidity, color, COD, and soluble COD (SCOD) by K₂FeO₄, but in raw wastewater (Jiang et al. 2006). Since FeSO₄ and K₂FeO₄ had low removal efficiencies...
efficiencies in this study, these coagulants were both abandoned and, therefore, the main CF-S processes were performed using PAC and FeCl₃ that showed higher efficiencies in preliminary tests for removing BOD₅, COD, TSS, and turbidity while using less coagulants. Applying appropriate technology for coagulation and flocculation in wastewater treatment will result in lower concentration of chemicals in the water resources as well as reducing treatment costs (Ramphal & Sibiya 2014). Research has shown that using PAC as coagulant has a number of advantages in clarification of water and wastewater, including rapid aggregation velocity, larger and heavier flocs, and lower required dosage (Gao et al. 2003).

Evolution of returned sludge as a modification, and flocculant used

In this part of the study, at the first stage, the effects of simultaneous application of C-150 flocculant and coagulant agents were evaluated with CF-S conditions fixed at settings as explained in the section ‘Sampling and CF-S process’. For this purpose, each of the PAC and FeCl₃ coagulants were first added to the jar test dishes with concentrations of 5, 15, 25, 35, 45, and 55 mg L⁻¹ at rapid mixing step, and then an equal quantity of 2 mg L⁻¹ of C-150 polymer was added to each of the six jar test containers at the slow velocity. Then, before and after each CF-S process, the amounts of turbidity, TSS, COD, BOD₅, color, pH, electrical conductivity (EC), FC of different samples were measured.

At the second stage of this part of the study, the effects of the simultaneous application of coagulant agents and returned chemical sludge as a modification were evaluated with CF-S conditions fixed at settings as explained in the section ‘Sampling and CF-S process’. At the modification stage, PAC and FeCl₃ coagulants were added to the jar test dishes at concentrations of 5, 15, 25, 35, 45, and 55 mg L⁻¹ at rapid mixing step, and then half of the settled sludge in the earlier steps was added to each of the containers at slow velocity (sludge deposited within jar test at each coagulant concentration was kept in a measuring cylinder and half of it was added to a corresponding concentration of coagulant in the following step). The sludge was collected from the lower half of the measuring cylinder which had a higher density and used as the returned sludge. To enhance the accuracy, all CF-S processes were repeated three times for FeCl₃ and PAC coagulants. The amounts of turbidity, FC, color, TSS, COD, BOD₅, pH, and EC before and after each CF-S process were measured and an average of the three CF-S processes for each coagulant was considered for presenting the results.

Figures 5–8 illustrate the removal efficiency of BOD₅, COD, TSS, and turbidity using PAC and FeCl₃ coagulants alone, each with half of the sludge returned, and each with C-150 polymer. Figure 5 illustrates FeCl₃ and PAC turbidity removal efficiencies. As can be seen from this figure, the highest turbidity removal efficiencies belong to the conditions that half of the sludge was returned for both coagulants. Further, the turbidity removal efficiency was
higher in cases where 2 mg L\(^{-1}\) of C-150 polymer was used compared to the cases in which the coagulants were used alone. Generally speaking, in all six cases, turbidity removal efficiency by PAC coagulant shows higher efficiency especially when half of the sludge is returned. Research has shown that coagulation can be induced in the presence of inorganic metal ion salts (Renault et al. 2009). It can be hypothesized that the returned sludge might increase the amount of metal ions by recycling unused coagulants, and dense sludge as condensation nuclei, for improving removal efficiency in the CF-S process. A combination of coagulant and polymer has been found to enhance flocculation and sedimentation processes while slightly reducing chemical consumption (Väänänen 2014).

The results of TSS removal efficiency using FeCl\(_3\) and PAC coagulants are presented in Figure 6. In both coagulants, the better TSS removal efficiency, especially at low coagulant concentrations, is obtained when half of the sludge is returned. In general, among the six states illustrated in Figure 6, the TSS removal efficiency is the highest when PAC is used along with half of the produced sludge. Using a coagulant with high removal efficiency of turbidity and TSS has also shown to be an effective way for the removal of many other contaminants that can be adsorbed by colloids, such as metals, toxics, organic matter, viruses, and radionuclides (Stechemesser & Dobiáš 2005).

Figure 7 shows BOD\(_5\) removal efficiencies obtained under six conditions using FeCl\(_3\) and PAC coagulants. The highest BOD\(_5\) removal efficiency obtained was when half of the sludge was recycled to the CF-S processes regardless of the coagulants used. Generally, BOD\(_5\) removal efficiencies in three cases of CF-S processes using PAC coagulant were higher compared to the corresponding three cases where FeCl\(_3\) coagulant was used. These results are in agreement with the findings of a previous study (Aziz et al. 2007), which concluded that the removal efficiency of color and BOD\(_5\) increased intensively with an increase in PAC dosage. According to another study (Yang et al. 2012), BOD\(_5\) removal by CF-S processes, as an enhanced
wastewater treatment technology, had a direct correlation with elimination of other pollutants, such as most endocrine disrupting chemicals, pharmaceuticals, and personal care products.

The results of COD removal efficiency using FeCl₃ and PAC are shown in Figure 8. Similar to BOD₅, TSS, and turbidity, the COD removal efficiencies for both coagulants were at the highest levels when half of the sludge was used in CF-S processes for all coagulant concentrations. Furthermore, when PAC coagulant was used along with half of the sludge, the COD removal efficiency was higher compared to the case where FeCl₃ coagulant was used along with half of the sludge. A similar study on wastewater treatment showed that coagulants had the lowest COD removal efficiency when they were used alone. It was found that adding some polyelectrolyte and clay mineral agents to FeCl₃ as coagulant aids could improve COD removal efficiency (Hassan et al. 2009).

The sludge produced by each coagulant in the optimum dosages was deposited in a measuring cylinder and measured after 15 min for all cases. Reductions in sludge volume when half of the sludge was returned and when C-150 polymer was used were calculated and compared with the state of using each coagulant alone. Table 1 shows the volume of sludge deposited by CF-S processes in six cases of PAC and FeCl₃ applications in the states of optimum concentrations. When half of the PAC sludge was returned and when 2 mg L⁻¹ of C-150 polymer was used, the sludge volume reduced, respectively, by 40% and 35% compared with using PAC coagulant alone. For FeCl₃ coagulant, in the corresponding cases, sludge reduction was recorded as 28% and 34%, respectively.

Disinfection process

The CF-S operation effluents for optimum dosages of PAC and FeCl₃ (45 mg L⁻¹ PAC and 55 mg L⁻¹ FeCl₃) were disinfected by 1 mg L⁻¹ chlorine in all cases, including each coagulant along with half of the returned sludge, each coagulant in addition to 2 mg L⁻¹ of C-150 polymer, and each coagulant alone. The amount of residual chlorine and FC were measured after 20 min. The results of disinfection processes are presented in the last two rows of Table 2. The residual chlorine ranged from 0.1 to 0.3 mg L⁻¹.

Optimum dosages for irrigation reuse

Table 2 compares the values of TSS, COD, BOD₅, pH, EC, FC, color, and turbidity of Yazd-ICEAS effluent before and after the CF-S processes in optimum dosages of PAC (45 mg L⁻¹) and FeCl₃ (55 mg L⁻¹) in three cases, including each coagulant with half of the returned sludge, each coagulant with 2 mg L⁻¹ of C-150 polymer, and each coagulant alone. To be appropriately reused, discharged effluent must match the standards such as those set by the EPA (Al-Jasser 2011). Table 2 also compares the quality of six different CF-S operation effluents with 2012 EPA irrigation guidelines and the standards set by the regional employer company (Yazd Water and Wastewater Company). The obtained results show that at the optimum dosages, the EPA standards related to irrigation of ‘processed type’ crops were met in CF-S processed effluents except for the cases using FeCl₃ coagulant alone. However, some of the parameters of CF-S process effluent, such as COD, did not meet the EPA standards required for irrigation of ‘non-processed type’ crops. Moreover, when half of the sludge was returned to CF-S processes in PAC optimum dosages, all recommendations set by Yazd Water and Wastewater Company and 2012 EPA standards of irrigation (mentioned in Table 2) were met for both ‘processed and non-processed type’ agricultural crops.

The final quality of treated effluent using CF-S process in optimum dosages of PAC and FeCl₃ were indicated in Table 3 for some important parameters in agricultural use. Also the value of these parameters in final effluent were in concordance with 2012 EPA irrigation guidelines (EPA Guidelines 2012).

<table>
<thead>
<tr>
<th>Chemical coagulants</th>
<th>Sludge volume (mL L⁻¹)</th>
<th>Percentage of sludge reduction</th>
</tr>
</thead>
<tbody>
<tr>
<td>FeCl₃</td>
<td>35 ± 5</td>
<td>0</td>
</tr>
<tr>
<td>PAC</td>
<td>30 ± 4</td>
<td>0</td>
</tr>
<tr>
<td>PAC + 50% returned sludge</td>
<td>18 ± 4</td>
<td>40</td>
</tr>
<tr>
<td>PAC + 2 mg L⁻¹ C-150</td>
<td>20 ± 3</td>
<td>33</td>
</tr>
<tr>
<td>FeCl₃ + 50% returned sludge</td>
<td>25 ± 2</td>
<td>28</td>
</tr>
<tr>
<td>FeCl₃ + 2 mg L⁻¹ C-150</td>
<td>23 ± 5</td>
<td>34</td>
</tr>
</tbody>
</table>
Table 2 | Removal efficiency of each CF-S process compared to 2012 EPA and agricultural irrigation guidelines recommended by the regional employer

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Effluent process/Unit</th>
<th>Yazd-ICEAS effluent</th>
<th>FeCl₃ (55 mg L⁻¹)</th>
<th>PAC (45 mg L⁻¹)</th>
<th>2112 EPA Guideline</th>
<th>Regional recommended employer</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>CF-S effluent in optimum dosage³</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>FeCl₃ alone</td>
<td>FeCl₃ + 2 mg C150</td>
<td>FeCl₃ + 50% returned sludge</td>
<td>PAC alone</td>
</tr>
<tr>
<td>Turbidity</td>
<td>NTU</td>
<td>13 ± 5</td>
<td>3.4 ± 1</td>
<td>2.5 ± 0/5</td>
<td>2 ± 0.3</td>
<td>3 ± 0.4</td>
</tr>
<tr>
<td>TSS</td>
<td>mg L⁻¹</td>
<td>28 ± 7</td>
<td>4 ± 1</td>
<td>3 ± 1</td>
<td>2 ± 0</td>
<td>3 ± 1</td>
</tr>
<tr>
<td>Color</td>
<td>PT-CO</td>
<td>55 ± 10</td>
<td>35 ± 6</td>
<td>33 ± 4</td>
<td>35 ± 7</td>
<td>34 ± 4</td>
</tr>
<tr>
<td>COD</td>
<td>mg L⁻¹</td>
<td>53 ± 12</td>
<td>24 ± 2</td>
<td>28 ± 3</td>
<td>21 ± 2</td>
<td>26 ± 3</td>
</tr>
<tr>
<td>BOD₃</td>
<td>mg L⁻¹</td>
<td>35 ± 12</td>
<td>14 ± 3</td>
<td>12 ± 2</td>
<td>10 ± 2</td>
<td>11 ± 2</td>
</tr>
<tr>
<td>pH</td>
<td>–</td>
<td>7 ± 1</td>
<td>6.8 ± 0.2</td>
<td>6.9 ± 0.25</td>
<td>6.7 ± 0.2</td>
<td>6.8 ± 0.17</td>
</tr>
<tr>
<td>EC</td>
<td>µS/cm</td>
<td>1,200</td>
<td>1,241</td>
<td>1,240</td>
<td>1,248</td>
<td>1,246</td>
</tr>
<tr>
<td>Mean FC without disinfection MPN/100 mL</td>
<td>10⁷–10⁸</td>
<td>14 × 10³</td>
<td>26 × 10³</td>
<td>8 × 10³</td>
<td>82 × 10²</td>
<td>36 × 10³</td>
</tr>
<tr>
<td>Mean FC after disinfection in 20 min³ MPN/100 mL</td>
<td>14 × 10⁴</td>
<td>2</td>
<td>4</td>
<td>0</td>
<td>2</td>
<td>3</td>
</tr>
</tbody>
</table>

³The most appropriate dosage determined by TSS, turbidity, BOD, and COD.
¹Not specified.
¹¹mg L⁻¹ obtained with 2 mg L⁻¹ calcium hypochlorite Ca(OCl)₂.
³Dependent on the crops.
³Fecal coliform.
³Yazd Water and Wastewater Company.
CONCLUSION

An advanced technique of treatment including CF-S was used to investigate water reuse of secondary effluent of Yazd-ICEAS wastewater for irrigation purposes. Physical, chemical, and microbiological parameters were determined at the inlet and outlet of both secondary Yazd-ICEAS and CF-S effluents. Based on the results of the present study, the following conclusions can be drawn from the experiments:

- In the Advanced treatment of Yazd-ICEAS secondary effluent (at the concentrations of 5 to 55 mg L\(^{-1}\)), FeSO\(_4\) and K\(_2\)FeO\(_4\) coagulants had low removal efficiencies; however, PAC and FeCl\(_3\) coagulants had higher removal efficiencies for BOD\(_5\), COD, TSS, and turbidity.
- Using PAC and FeCl\(_3\) each with half of the produced sludge as a modification or each with 2 mg L\(^{-1}\) of C-150 polymer, resulted in higher removal efficiencies of BOD\(_5\), COD, TSS, and turbidity compared with using each coagulant alone. Moreover, both coagulants, especially PAC, showed the highest removal efficiency when half of the produced sludge was returned compared with using 2 mg L\(^{-1}\) of C-150 as flocculant along with the coagulants.
- At the optimum dosage (45 mg L\(^{-1}\)), when PAC was used along with half of the returned sludge, the CF-S process effluent parameters of BOD\(_5\), COD, TSS, turbidity, FC, and pH were, respectively, 7 ± 1, 18 ± 1, 1 ± 0, 1 ± 0 mg L\(^{-1}\), 0, and 6.7 ± 0.2 which were higher than all other cases.
- At the optimum dosage, when half of PAC sludge was returned or 2 mg L\(^{-1}\) of C-150 flocculant was used, respectively, 40% and 30% of the sludge volume was reduced as compared with using PAC alone, while in the corresponding cases, these amounts for FeCl\(_3\) were calculated as 28% and 34%, respectively.
- At the optimum dosages, except for using FeCl\(_3\) alone, in the other five cases the 2012 EPA standards related to irrigation of ‘processed type’ crops were met after disinfection using 1 mg L\(^{-1}\) chlorine in 20 min retention time. Also, when half of the sludge was returned to the PAC coagulant CF-S process and after disinfection using 1 mg L\(^{-1}\) chlorine in 20 min retention time, all recommendations set by Yazd Water and Wastewater Company and 2012 EPA standards of irrigation were met for both ‘processed and non-processed type’ agricultural crops.

Investigations on the processes of combination of coagulation/flocculation and filtration would be of interest to evaluate operational experience. Also, it is recommended to examine the process of a combination of coagulation/flocculation and multi-oxidants to study the development of tertiary treatment. Some natural coagulants, such as chitosan, are more favorable in wastewater treatment due to their environmental friendly characteristics, thus it is recommended that they be investigated.

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