Desalination and Water Treatment
www.deswater.com

43 (2012) 1–7
April

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doi: 10.1080/19443994.2012.672182

Evaluation of lead and COD removal from lead octoate drier effluent by chemical precipitation, coagulation–flocculation, and potassium persulfate oxidation processes

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Received 12 May 2011; Accepted 19 February 2012

ABSTRACT

A range of treatment processes for the removal of mineral and organic pollutants from industrial wastewater have been investigated. This study was aimed to remove lead, turbidity, and chemical oxygen demand (COD) from lead octoate drier wastewater via potassium persulfate oxidation in the presence of UV and heat, chemical precipitation by sulfuric acid, and coagulation–flocculation processes. $Al_2(SO_4)_3$, poly-aluminum chloride (PAC), and $FeCl_3$ were employed as coagulants. Lime, bentonite, and cationic and anionic polymers were also utilized as coagulant aids. All of the applied methods were successful in lead and turbidity removal. Precipitation by sulfuric acid followed by filtration eliminated 99.0% of lead and 90.0% of turbidity. The most removal efficiency involving coagulation–flocculation processes was 99.7% for lead (by PAC/lime) and 98.3% for turbidity (by PAC). However, COD removal was only achieved by potassium persulfate oxidation. The results indicated that oxidation process by potassium persulfate in exposure to heat was the most promising treatment method, resulting in the removal of COD, lead, and turbidity at the rates of 94.9, 99.9, and 96.6%, respectively.

Keywords: Lead octoate; COD; Lead removal; Potassium persulfate; Oxidation; Coagulation

1. Introduction

Heavy metal ions can be toxic and carcinogenic even at very low concentrations, and hence, usually pose a serious threat to the environment and public health [1–3]. Heavy metal ions are commonly found in industrial effluents and some of them may contain heavy metal ions at high concentrations [4,5]. For example, lead battery-recycling site generates discharge wastewaters derived from the smelting process with lead concentrations that vary from 2 to 300 mg/L depending on discharge sites and operating conditions [6]. The effluent of a radiator manufacturing factory in India includes lead concentration of around 709 mg/L [7].

Lead octoate is a synthetic organic salt employed as a drier in alkyd and other unsaturated paints as well as some other industries [8]. Wastewater source is the tank cleanout accompanied by heating from the process in which lead drier is manufactured. Organic salts of lead are particularly difficult to treat since lead is not free to form precipitate. A number of treatment methods have been utilized for the removal of heavy metals from industrial effluent. The most commonly applied methods include: chemical precipitation, solvent extraction, evaporation, electrodialysis, ultrafiltration, reverse osmosis, ion exchange, ion flotation, and adsorption [9–18].

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Furthermore, various oxidation processes have been successfully employed for the removal of the organic contents of wastewaters. For instance: ferrate technology [19–22], catalytic oxidation of the dye wastewater with hydrogen peroxide [23], various advanced oxidation processes and chemical treatment methods for COD and color removal from a polyester and acetate fiber dyeing effluent [24], ozone and membrane filtration based strategies for the treatment of cork processing wastewater [25], and Fenton oxidation of carpet dyeing wastewater for removal of COD and color [26].

The effect of sulfate radical on degradation of organic contaminants in wastewater treatment application has also been demonstrated by previous studies and some instances include: ultraviolet activated persulfate oxidation of phenol [27], methyl tert-butyl ether degradation by ferrous ion-activated persulfate oxidation [28], trichloroethylene degradation by zerovalent iron-activated persulfate oxidation [29], and effect of persulfate on the oxidation of benzotriazole and humic acid by e-beam irradiation [30]. In this study, conventional methods such as coagulation and flocculation, chemical precipitation as well as oxidation process by potassium persulfate ($K_2S_2O_8$) in the presence of UV and heat were investigated.

2. Materials and methods

2.1. Instruments for analysis and chemicals

COD: Hach DR2000 UV/VIS spectrophotometer, Turbidity: HANNA HI 93703 Turbidity meter, Lead: Unicam 919 atomic absorption spectrophotometer, pH: Metrohm 691digital pH meter, Jar test: Phipps and Bird stirrer 7790-402, UV radiation: Ultraviolet lamp 80 W, ferric chloride, poly-aluminum chloride, alum, cationic polyacrylamide, anionic polymer, bentonite, calcium hydroxide, potassium iodide, potassium persulfate, powdered activated carbon, sulfuric acid, hydrochloric acid.

Samples: Industrial wastewater samples were obtained from a lead drier manufacturing company located in an industrial complex in Tehran, Iran. Samples were taken and preserved in accordance with the Standard Methods for the Examination of Water and Wastewater [31]. The characteristics of the wastewater are shown in Table 1.

2.2. Chemical precipitation, coagulation–flocculation

Experimental studies were designed to obtain maximum lead, turbidity, and COD removal in the region of neutral pH and optimum dosages of the coagulants. First, pH adjustment of the alkaline waste-

Table 1
Test results of precipitation and coagulation processes

No. of process	Treatment condition	pH*	COD (mg/L)	Turbidity (NTU)	Pb^{2+}(mg/L)
–	Raw wastewater	13.8	1,390	60	405.00
1	H_2SO_4 (3 mL/L)/filtration	7.0	1,155	6	4.01
2	Alum (20 mg/L)	7.0	1,455	4	2.64
3	Alum (100 mg/L)	7.0	1,487	3	2.40
4	$FeCl_3$ (20 mg/L)	6.8	1,325	9	2.51
5	$FeCl_3$ (100 mg/L)	6.6	1,362	4	1.15
6	$FeCl_3$/activated carbon 20/4 (mg/L)	6.9	1,385	4	2.21
7	$FeCl_3$/activated carbon 40/10 (mg/L)	6.9	1,394	10	2.13
8	PAC (20 mg/L)	7.0	1,260	4	2.57
9	PAC/KI 20/40 (mg/L)	7.5	1,300	4	2.23
10	PAC/Bentonite 20/400 (mg/L)	8.5	1,125	18	2.12
11	**PAC/Lime 20/500	5.4	1,123	29	1.35
12	**PAC/Lime 20/4,000 (mg/L)	8.5	1,110	7	0.96
13	PAC (40 mg/L)	7.0	1,273	1	1.22
14	PAC/anionic polymer 20/20 (mg/L)	7.0	1,225	4	1.82
15	PAC/anionic polymer 20/100 (mg/L)	7.0	1,234	7	1.53
16	PAC/cationic polymer 20/2 (mg/L)	7.6	1,087	24	2.61
17	PAC/cationic polymer 20/4 (mg/L)	7.5	1,050	36	2.46
18	Cationic polymer (1 mg/L)	7.6	1,101	11	3.51

*pH after settling time.
**pH was adjusted to 4, then PAC and lime were added.

water to neutral point was carried out using concentrated H_2SO_4. In this stage, white precipitates formed and turbidity greatly increased, while the lead content had a considerable decline after filtration (Table 1).

The coagulation processes were placed after the acid treatment step. Jar tests on the laboratory scale were carried out in order to choose the appropriate coagulant and its optimum dosage. $Al_2(SO_4)_3$, poly-aluminum chloride, and $FeCl_3$ of commercial grade were tested as chemical coagulants. Different doses of each coagulant were added to the beakers containing 500 mL samples and mixed at 100 rpm for 30 s and the stirring speed was adjusted to 30 rpm for the next 30 min. The amounts of COD, lead, turbidity, and pH of the samples were measured after 45 min settling time. Lime was added to the beakers containing 500 mL samples in the gentle mixing stage subsequent to the flash mixing with PAC, while the pH value had been previously reduced to 4 using H_2SO_4. Powdered activated carbon was added after the flash mixing of $FeCl_3$ with the neutralized samples. Polyacrylamide (cationic) was utilized separately as a coagulant and also after the flash mixing of the neutralized sample with PAC. Anionic polymer was utilized similarly to the last-mentioned. Potassium iodide (KI) was also used after the flash mixing of the neutralized sample with PAC.

2.3. The development of $K_2S_2O_8$ oxidation process

The $K_2S_2O_8$ oxidation process was carried out on the laboratory scale. For the development of the chemical oxidation method, experiments were performed using potassium persulfate ($K_2S_2O_8$) in the presence of UV and heat. Different dosages of $K_2S_2O_8$ were added to 500 mL of the samples in order to select the optimum quantities of the reagents. It was added in different dosages to the supernatant after settling time following the coagulation of the neutralized wastewater samples by poly-aluminum chloride in order to investigate its effect on COD (Table 2). It was added to the raw wastewater samples as well.

For the development of the oxidation reaction in the presence of heat, the reaction mixture was heated to 90°C. Oxidation started after a few minutes and the organic content oxidized to H_2O and CO_2 (Scheme 2). Bubbles of CO_2 were proper indicators for the reaction monitoring. After an hour, CO_2 formation ended. Heating was continued for additional 15 min. At the end of this period, lead ions (Pb^{2+}) precipitated as $PbSO_4$ and settling concluded by 20 min. In addition, UV radiation instead of heat was employed to activate persulfate to initiate the oxidation reaction. So the samples, as prepared above, were placed in the exposure of an 80 W UV lamp for 72 h. In this case, oxidation process was so slow that CO_2 bubbles were not traceable. The samples were analyzed and the results are summarized in Table 2.

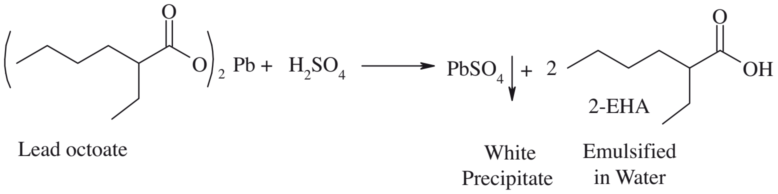
3. Results and discussion

As usual in drier industry, a hot solution of NaOH is utilized for rinsing the reactor of lead octoate so that the remained lead octoate at a temperature of around 90°C dissolves and the consequent effluent is discharged. Thus, apart from a high concentration of lead up to 405.00 mg/L, the wastewater has a pH value of around 13.8. Moreover, it comprises a COD content of about 1,390 mg/L in soluble state. Some treatment methods have been utilized to eliminate them and the results are as follows:

Table 2
Test results of $K_2S_2O_8$ oxidation

No. of process	H_2SO_4/PAC (mL/L)/(mg/L)	pH*	Pb^{2+} (mg/L)*	Turbidity (NTU)*	COD (mg/L)*	$K_2S_2O_8$ (mg/L)	Heat	UV	Time (h)	Pb^{2+} (mg/L)**	COD (mg/L)**	Turbidity (NTU)**	Lime (g/L)	pH**
19	3/20	7.0	2.57	4	1,260	10	√	–	1	0.72	560	1	–	6.8
20	3/20	7.0	2.57	4	1,260	20	√	–	1	0.50	150	1	2.5	7.3
21	–	13.8	405	60	1,390	10	√	–	2	0.65	515	12	–	10.5
22	–	13.8	405	60	1,390	20	√	–	2	0.40	70	2	2.5	7.0
23	–	13.8	405	60	1,390	10	–	√	72	2.84	1,030	17	–	10.9
24	–	13.8	405	60	1,390	20	–	√	72	0.97	880	9	–	8.2

*Before oxidation.
**After the process completion.



Scheme 1.

3.1. Chemical precipitation

The effect of chemical precipitation by sulfuric acid on lead, turbidity, and COD is summarized in Table 1. Concentrated H_2SO_4 was used prior to coagulation–flocculation processes with the purpose of reducing pH. Using H_2SO_4, pH decreased to 7 and the sample took on a whitish color and turbidity increased by a large amount (832 NTU). The increase of turbidity was due to the reaction between sulfuric acid and lead octoate through which Pb^{2+} converted to white suspended particles of $PbSO_4$ and organic salt of octoate converted to 2-ethylhexanoic acid (2-EHA). The reaction is depicted in Scheme 1. Hence, H_2SO_4 resulted in substantial lead deposition which increased turbidity from 60 to 832 NTU. Filtration removed a great deal of lead content and consequently turbidity decreased to 6 NTU.

However, COD had negligible decrease as organic acid of 2-EHA emulsified after pH reduction. Because, organic acids containing more than six atoms of carbon are insoluble in water, moderate carboxylic acids form emulsion with water, and the larger ones make two separated phases. On the other hand carboxylic acids become soluble in water by converting to ionic carboxylate salts in an alkaline medium. In this case, 2-EHA with eight atoms of carbon as a moderate acid, which is soluble in alkaline NaOH solution, emulsified as 2-EHA due to adding H_2SO_4 and decrease of pH. Therefore, the organic content, 2-EHA, was not filtered out effectively and COD reduction was negligible.

3.2. Coagulation–flocculation

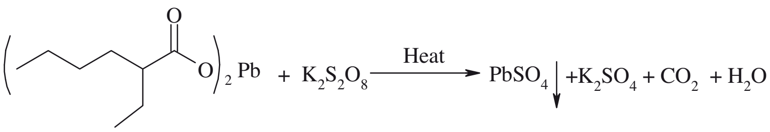
The effect of some coagulants and coagulant aids on the removal of lead, turbidity, and COD was investigated and the results are shown in Table 1. Prior to each coagulation–flocculation experiment, pH was adjusted using sulfuric acid. As the pH decreased, suspended particles of $PbSO_4$ formed and the turbidity increased for the same reason as above. Poly-aluminum chloride (PAC) was able to remove the turbidity and lead due to effective flocculation. It also facilitated further removal of lead probably due to $PbCl_2$ formation. Although the usage of PAC along with H_2SO_4 produced better results for turbidity and lead removal, it was not able to eliminate COD.

As shown in Table 1, the addition of KI in gentle mixing was effective for more lead removal as it converted soluble Pb^{2+} to PbI_2 precipitates, which reduced the lead content to 2.23 mg/L. However, a slight increase in COD value was observed in comparison with the time when PAC had been singly used.

Anionic and cationic polymers were used as coagulant aids in gentle mixing in order to reduce COD. Even though, COD reduction was negligible since they were not able to trap the organic content effectively. The usage of anionic polymer resulted in more removal of lead because of the interaction of Pb cations with the anions of the polymer. The increase of the polymer dosage led to more Pb removal, but its extra amount caused an increase in turbidity from 4 to 7 NTU.

In comparison with anionic polymer, the usage of cationic polymer led to more decrease in COD, but it increased turbidity values. Turbidity even had more increase (36 NTU) when more dosage of the polymer was used, as it gave a colloidal form to the sample. The reduction of COD would be as a consequence of interaction between cations of the polymer and anions of 2-EHA.

Lime was added in gentle mixing, while pH had been adjusted to 4 in order to prevent excess pH increase. Coagulation process with PAC/lime following pH adjustment by H_2SO_4 reduced lead concentration.



Scheme 2.

After the process completion, the pH increased from 4 to 5.4 at which inadequate flocculent settling caused a turbidity rise to 29 NTU. The decrease of COD was negligible. pH increased from 4 to 8.5 with the increase of lime dosage which improved the coagulation process and as a result the turbidity reduced to 7 NTU. It also facilitated the most removal efficiency of lead (99.7%).

Using bentonite in gentle mixing, lead and COD concentration decreased to some extent by means of precipitation. However, turbidity value increased to 18 NTU possibly because of the existence of the extra particles of bentonite which remained suspended in the supernatant.

The effect of $FeCl_3$ on lead, turbidity, and COD is summarized in Table 1. As shown in Table 1., $FeCl_3$ as a conventional coagulant was successful to deposit suspended particles resulted from H_2SO_4 adding and as a result removed a great deal of lead and turbidity. It also decreased lead concentration by $PbCl_2$ formation. COD removal was not achieved by $FeCl_3$. The amount of lead and turbidity decreased using more dosage of $FeCl_3$.

Powdered activated carbon added in gentle mixing was not able to adsorb the organic content. Accordingly it was not able to remove COD. However, it caused more Pb removal. With the increase of its dosage together with the increase of $FeCl_3$ dosage, Pb concentration demonstrated more reduction, whereas the turbidity value increased from 4 to 10 NTU because a number of particles of activated carbon remained in the supernatant.

Alum reduced the amount of turbidity and lead (Table 1). The decrease of lead and turbidity resulted from coagulation of suspended particles of $PbSO_4$ which had been formed subsequent to pH adjustment by H_2SO_4. It was also because of the reaction of $Al_2(SO_4)_3$ with Pb which resulted in more Pb deposition as $PbSO_4$.

The effect of the above processes on turbidity, lead, and COD is depicted in Figs. 1–3. According to Fig. 1, the most removal efficiency of turbidity was obtained by PAC. Fig. 2 indicates that coagulation process by PAC/lime has the most effect on lead removal. As shown in Fig. 3 coagulation by PAC/cationic polymer had comparatively the most removal efficiency of COD, although it was still unsatisfactory.

Despite lead and turbidity removal, the above processes were not efficient for COD elimination. The results indicated that conventional treatment methods such as chemical coagulation and precipitation were not successful for the removal of the organic content of lead octoate drier effluent. 2-EHA or its salts have fewer tendencies to conventional coagulants. So, effective coagulation and separation of organic acids are so difficult. Although 2-EHA is biodegradable [32], biological treatment would be complicated in the presence of Pb^{2+} as an inhibitor. As an alternative method, chemical oxidation by potassium persulfate ($K_2S_2O_8$) was carried out in order to remove the organic content and the results are as follows.

3.3. The oxidation process in the presence of heat

The effect of the oxidation processes on lead, COD, and turbidity is shown in Table 2. In the beginning, the oxidation experiments were carried out subsequent to the coagulation process with PAC. Two samples with the same initial values of turbidity, lead, and COD were experimented. The oxidation process was performed along with heating in order to speed up the oxidation reaction. The values of lead, turbidity, and COD before and after the oxidation process are shown in Table 2. According to the results, oxidation by $K_2S_2O_8$ decreased COD to 560 mg/L through the reaction which oxidized the organic content to H_2O and CO_2 and proceeded 1 h. Bubbles of CO_2 were proper indicators for the reaction monitoring. The increase of $K_2S_2O_8$ dose reduced COD to 150 mg/L. However, it caused a decrease in pH probably because of the reaction between CO_2 and H_2O. So, lime was added to adjust it near neutral pH (Table 2).

In addition to COD reduction, $K_2S_2O_8$ was found to be able to decrease lead and turbidity as a result of $PbSO_4$ deposition. Therefore, the following experiments were carried out with the raw wastewater samples with the same initial values of turbidity, lead, and COD and the results are summarized in Table 2. The oxidation reaction between $K_2S_2O_8$ and lead octoate continued for 2 h and soluble Pb^{2+} ion converted to $PbSO_4$ precipitate. A great deal of lead content precipitated after 20 min settling time. On the other hand, it reduced the COD by oxidizing organic ions of octoate to CO_2 and water. The reaction is shown by Scheme 2. The increase of $K_2S_2O_8$ dosage improved oxidation reaction and led to further removal of COD

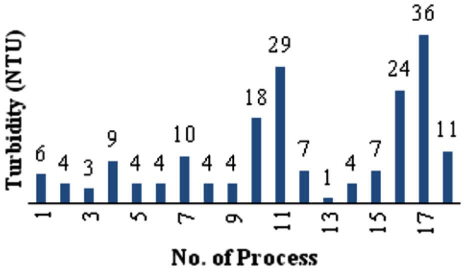


Fig. 1. Turbidity (NTU) after precipitation and coagulation processes.

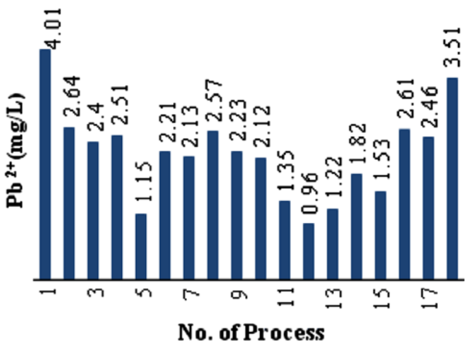


Fig. 2. Pb^{2+} (mg/L) concentrations after precipitation and coagulation processes.

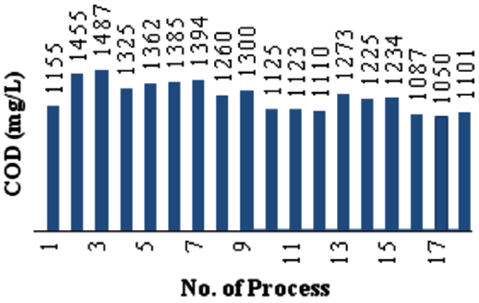


Fig. 3. COD (mg/L) after precipitation and coagulation processes.

and lead. Turbidity also decreased from 12 to 2 NTU with the increase of its dose possibly because lower pH enhanced settling condition.

3.4. The oxidation process in the presence of UV

UV radiation was utilized instead of heat to initiate oxidation reaction. The samples were exposed to UV for 72 h. The reaction was so slow that bubbles of CO_2 were not traceable. COD, lead, and turbidity values were measured after 72 h and the results are summarized in Table 2. The reduction of their values was less than those of the former processes conducted in the presence of heat because UV was not able to activate the oxidation reaction successfully. The increase of the dosage of $K_2S_2O_8$ was not adequately effective in this stage. The upper values of turbidity would be as a result of the upper pH values as well.

4. Conclusion

All of the applied processes were effective for lead and turbidity deposition. In addition to pH adjustment, H_2SO_4/filtration solely reduced lead concentration by a large amount. Coagulation–flocculation processes were highly effective for turbidity and more lead deposition. However, they were not successful in the COD removal. Oxidation process was able to remove the organic content. Potassium persulfate was selected as a versatile oxidant. Because, in addition to its capability to oxidize the organic contents, it was able to convert soluble Pb^{2+} ion to $PbSO_4$ precipitate. The oxidation process by potassium persulfate in the presence of heat was the most promising treatment method resulting in COD, lead, and turbidity removal at the rates of 94.9, 99.9, and 96.6%, respectively. Furthermore, $K_2S_2O_8$ is soluble in water and easy to use in lead drier industry. In this process, heat can be supplied from lead drier manufacturing reactor in which the temperature is about 90°C after the product discharge. The reactor is equipped with a filter press to filter the product (lead octoate drier), by which the precipitated lead salts ($PbSO_4$) can be filtered. Therefore, oxidation by $K_2S_2O_8$ in the presence of heat is cost effective, efficient, and environmentally benign for the treatment of lead drier effluent.

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