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A critical study on the treatability of metal plating industry wastewater and real scale adaptation

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Abstract

Water pollution is one of the major problems for humankind. Various pollutants could be detected in wastewater because of human activities such as industrialization, agriculture, domestic waste and etc. Removal of pollutants such as heavy metals, dyes, oils and pesticides are of great importance which affects human life negatively. Many methods have been extensively used to provide "clean water" for environment and human. Heavy metals are important industrial pollutants that need to be quickly removed from wastewater due to their high toxicity and non-biodegradable structure. In this paper, the heavy metals including copper, nickel and zinc have been examined in real wastewater from metal plating industry in Bursa, Türkiye. Concentrations of pollutants (Cu, Ni, Zn, chemical oxygen demand (COD) and SO_4) in the effluent as a result of coagulation-flocculation process were determined and their compliance with sewage discharge standards was investigated. Moreover, the removal efficiencies of the pollutants were examined (Cu and Ni: 97-100%; Zn: 82-98%; COD: 32-54%; SO_4: 16-23%) and the effect of the coagulant doses used on the operating cost, sludge quantity were also discussed.

1. Introduction

Water is crucial for human life and ecosystem and only 1% of the available water can be used for drinking and agricultural activities. Various activities such as natural events, wars, accidents, industrial activities, farming and agriculture have led to the discharge of many pollutions which represents a public concern [1]. Wastewater has become the most important environmental problem for human and living organisms due to its hazardous effects on our health and habitat [2,3]. Water can be polluted by dye, heavy metals, oil, pesticides, poly- and perfluoroalkyl substances, personal care products, and so on [4,5].

Global water resources are severely polluted by heavy metal ions and organic compounds from industrial activities [6,7]. Heavy metal-based water pollution is one of the most dangerous pollutions for human life because metals tend to accumulate in the body, not biodegradable and heavy metals with a density exceeding 5 g/m^3 are known to be toxic and carcinogenic [8]. Zinc (Zn), Copper (Cu), Nickel (Ni), Mercury (Hg), Cadmium (Cd), Lead (Pb) and Chromium (Cr) are heavy metals found in industrial wastewater and require special attention in the cleaning of industrial wastewater [9,10]. Heavy metals in wastewater from various industrial activities such as metal plating-smelting, electrolysis and electroplating could pollute soil, water, and air [11]. In metal plating industries, platings are often used to protect metals against corrosion and to improve basic properties such as mechanical or chemical stability which is used in platings beautify objects such as car and aircraft parts, bathroom fixtures, chairs, and tables [12,13]. The metal plating industry produces large volumes of strongly acidic wastewater containing high concentrations of organic and inorganic pollutants, cyanide, heavy metals (chromium, nickel, zinc, and copper), degreasing solvents and particulate matter [14–17]. In metal plating industries, objects are plated in plating baths and their surfaces are cleaned in rinsing baths. Heavy metals in the plating solution migrate to the rinsing baths and the water in the rinsing baths is constantly changed. Rinse waters contain toxic heavy metals (Zn, Ni, Cu, Hg, Cd, Pb, Cr etc.), cyanide and various carcinogenic substances that cause environmental pollution. toxic compounds may be formed by the contact of cyanide with air and metals [18,19].

Zinc is an element necessary for human health, but excessive amounts cause health problems such as abdominal pain, skin problems and nausea [20]. Lead damages the central nervous system which is the second most toxic metal after arsenic [21]. Mercury is neurotoxic and damages the central nervous system. High concentration of mercury could cause deterioration in kidney function, chest pain and shortness of breath. It is well known that removing such pollutants from wastewater is of great importance for human health and environment that we live in [22,23]. Untreated discharge of rinse water into sewage systems causes serious problems due to high toxicity and can negatively affect the microbial activity of activated sludge in the wastewater treatment plant [24]. The content of the wastewater varies depending on the process applied, the type of plating and the amount. Wastewater from metal plating plants can be both alkaline and acidic. Cyanide-containing wastewater or water used in rinsing baths has a very high pH, while wastewater from chromium, copper and nickel baths is acidic [16,25].

A variety of techniques have been devoted in developing quick, effective, and cheap way to remove heavy metals from heavy metals. These methods include coagulation, flocculation adsorption, membrane filtration, reverse osmosis, ion exchange, and precipitation [26,27]. Even all methods have been utilized for water treatment, each has its own limitations such as time consuming, require complicated procedure, low efficiency, high cost etc. Among them, Coagulation-Flocculation process is the most effective, easy-to-implement and economical method for the treatment of heavy metals and inorganic compounds [28,29]. Since the pH of metal plating industry wastewater is highly acidic, it is necessary to bring the pH to the neutral range before discharge. Furthermore, the increase in wastewater pH requires high costs for the use of chemical reagents and treatment of the metal oxide precipitates [30,31]. The basis of coagulation-flocculation processes is to adjust the pH in wastewater. Chemical coagulants such as aluminum or iron salts are added to the wastewater to remove pollutants in colloidal form [32,33]. Polymerized forms of metal coagulants such as aluminum sulfate (alum), ferric chloride, and PAC (poly-aluminum chloride) are compounds that provide high pollutant and organic matter removal and can promote lower volume sludge formation [34]. Inam et al. [35] have studied the removal of As (III) and As (V) from aqueous solution by coagulation-flocculation process using $FeCl_3$ as coagulant, organic ligands (humic acid and salicylic acid) and the removal efficiencies were determined as 99% and 92%, respectively. Ag removal from industrial wastewater using coagulation-flocculation process was investigated. 46.36 mg/L PAC was used as coagulant and 0.318 mg/L anionic polyelectrolyte (Praestol 2640) was used as flocculant and Ag removal efficiency was 99% [36]. Cd(II) removal from aqueous solution by coagulation-flocculation process was investigated and CaO/PAC was used as coagulant, anionic polyacrylamide was used as flocculant. Cd(II) removal efficiency was founded as 94% [37]. Cr and Ni removal were investigated using flocculation process in real chromic acid lotion electroplating wastewater. Functionalized carboxylated chitosan flocculants with xanthate and sulfonic acid groups have been used. Cr and Ni removal efficiencies were 95% and 99%, respectively [38]. Cu(II) removal was investigated in flocculation process from aqueous solution and xanthated chitosan was used as flocculant. In the study, Cu(II) removal efficiency was founded as 97% [39].

In this study, COD and (Cu, Ni, Zn) heavy metals were investigated with the coagulation-flocculation process in the effluent concentrations and removal yield and it was aimed to supply the discharge standard to the sewage in real wastewater from the metal plating industry in Bursa. High concentrations of SO_4 are formed due to the use of H_2SO_4 in rinsing baths in the metal plating industry. Besides COD and Heavy Metal (Cu, Ni, Zn) removal, SO_4 removal has also been investigated. Coagulants (Caustic (NaOH), lime ($Ca(OH)_2$), iron III chloride ($FeCl_3$), barium chloride ($BaCl_2$) and anionic polyelectrolyte ($(NH_4)_2S_2O_8$)) operating costs used were calculated. The outcomes have demonstrated that the wastewater is integrated into the wastewater treatment plant and the system is operated.

2. Method

2.1. Sample collection

The wastewater used in the experiments were obtained from the metal plating industry in Bursa Demirtaş Organised Industrial Zone (Türkiye). The facility has a copper bath with a capacity of 5 m^3, a nickel bath with a capacity of 8 m^3 and a rinse bath with a volume of 20 m^3. Wastewater from plating tanks such as nickel and copper and rinsing bath, enters the balancing tank for homogenously mixed. Figure 1 shows the sampling point taken from the balancing tank in the metal plating industry. Since the plant has a standard production capacity, wastewater is formed at standard flow and characterization. Samples were taken twice a week and a total of 4 liters of composite samples were created, half a liter sample per hour. The obtained wastewater from the balancing tank in metal plating and stored at 4 °C in refrigerator before the experiments.

2.2. Coagulation-flocculation jar test procedure

Coagulation-flocculation study was carried out under laboratory conditions (24 °C) using a jar test device. During the research, 250 mL wastewater volume was used in each trial set. Each coagulation experiment was started by adding coagulant to the reactor containing wastewater, and then the appropriate initial pH was adjusted using HCl and NaOH. In the coagulation step, the reaction time was limited to 3 minutes and the process was continued at a stirring speed of 120 rpm [40]. The flocculation process was carried out at a mixing speed of 30 rpm with a contact time of 10 minutes [41]. The precipitation step was determined as 45 minutes and at the end of the process, samples were taken from the upper phase and analyzed for COD, Cu, Ni, Zn, SO_4.

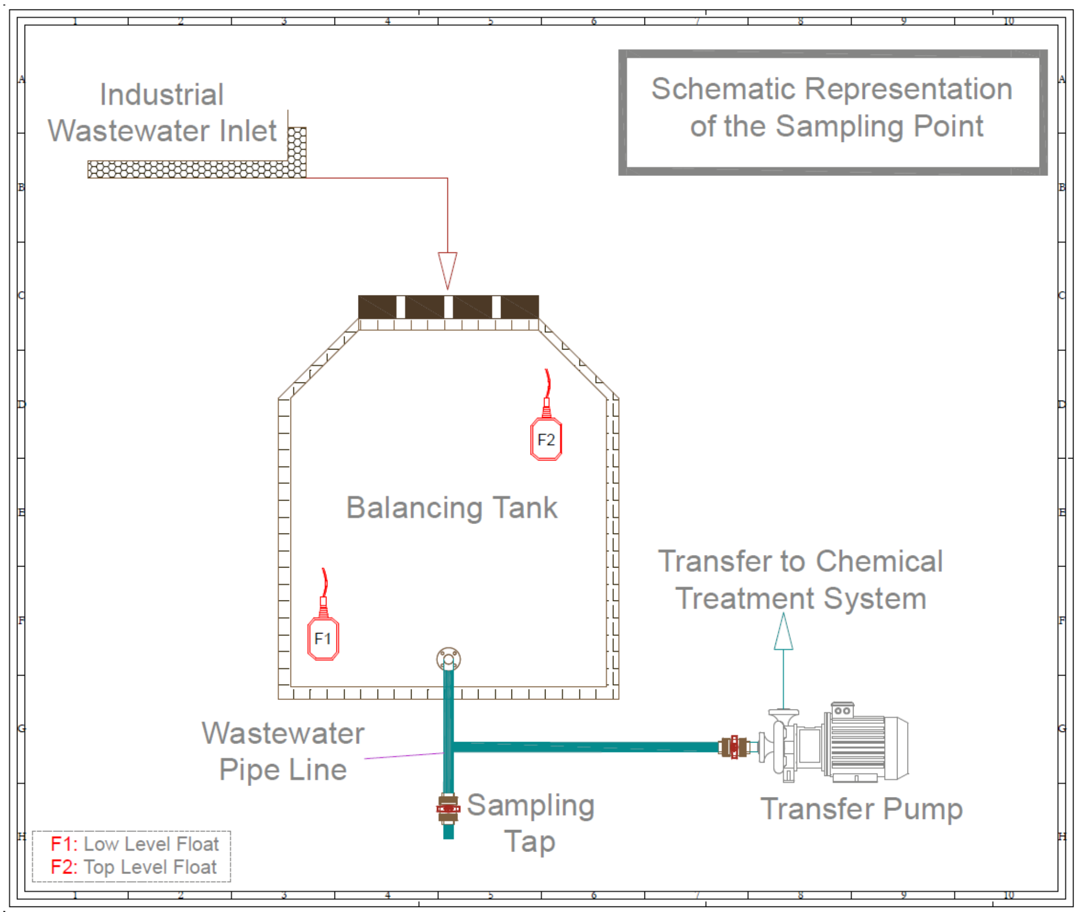

Figure 1. Metal plating industry sampling point.

The concentrations of coagulant to be used in the influent wastewater and the optimum pH values were determined at four different sets by Jar test in. Among all jar tests, doses and pH values of Set 4, which can provide limit values related to the discharge standard, were selected. In the metal plating industry wastewater, 45% NaOH, 5% $Ca(OH)_2$, 40% $FeCl_3$ and 0.1% $(NH_4)_2S_2O_8$ was used for coagulation-flocculation.

The aim of the first stage was to find the optimum concentrations in the coagulant doses to be used and the optimum pH value to which this dose was applied. By applying the chemicals and their doses used in Set 4 at the pH values specified in Table 1 and discharge standards for the compounds Cu, Ni, Zn, COD and SO_4 in sewages were provided. Chemicals used: NaOH: 2.75 mL; $Ca(OH)_2$: 11 g; it was used at the specified concentration, $FeCl_3$: 1.45 mL and $(NH_4)_2S_2O_8$: 6.5 g, and at a pH value between 6.4-12.3.

In the second phase of present study, removal efficiencies and effluent concentrations were investigated in order to reach the limit values specified for Cu, Ni, Zn, COD and SO_4 in the sewage and pre-treatment discharge criteria of Demirtaş Organized Industrial Zone [42]. The characterization of real metal plating wastewater is summarized in Table 2.

Table 1. Coagulants used in the study, doses and pH values

Coagulants	Doses				pH values			
	Set 1	Set 2	Set 3	Set 4	Set 1	Set 2	Set 3	Set 4
NaOH (mL)	3.3	3.1	3.1	2.75	11.8	7.6	8.0	6.4
$Ca(OH)_2$ (g)	-	9.5	12.5	11	-	11.7	12.8	12.3
$FeCl_3$ (mL)	1.3	1.2	1.5	1.45	9.5	9.4	8.8	8.8
$(NH_4)_2S_2O_8$ (g)	11	8	6	6.5	9.5	9.4	8.8	8.0

Table 2. Real metal plating industry wastewater characteristics.

Parameter	Present Study Value	Literature	Reference
pH	1.2	2.57	[44]
Conductivity (µS/cm)	240	-	-
COD (mg/L)	1230	159, 600, 4000	[12,44,45]
MLSS (mg/L)	1098	-	-
Cu (mg/L)	88.67	301	[12]
Ni (mg/L)	438.10	16, 51	[12,41]
Oil-grease (mg/L)	10	44	[44]
Zn (mg/L)	3.84	1.1, 145, 200	[41,44,45]
SO_4 (mg/L)	2797	-	-

2.3. Characterization

The conductivity and pH were measured using a pH meter and a conductivity device. COD (LCK-514), Cu (LCK-329), Ni (LCK-337), Zn (LCK-360), and SO_4 (LCK-353) were carried out with Hach test kits. Mixed Liquor Suspended Solids (MLSS) were determined according to Standard Methods (1994) (American Public Health Association, 1995). Oil-grease was analyzed by standard methods [43].

2.4. Operating cost

The operating cost was calculated in EUR (€) per cubic meter (m^3) wastewater volume, taking into account the optimum conditions in which the discharge parameters to the sewage were provided. The Equation 1 used in calculating the cost resulting from chemical consumption was given.

$$\text{Operating Cost} = c \times C_{chemical} \quad (1)$$

In Equation 1, c; the amount of chemicals required to treat 1 m^3 of wastewater (kg); $C_{chemical}$; cost of 1 kg of chemicals (€/kg).

3. Results and Discussion

In the metal plating industry, different compounds and concentrations are encountered depending on the chemicals used and the type of process. Ni concentration was 438.10 mg/L in present study and was higher than the literature in Table 2. While the oil-grease concentration was found to be lower than the value encountered in the literature; the Zn concentration (3.84 mg/L) was among the concentrations stated in the literature.

Additionally, COD concentrations were found at different values of 159 mg/L and 4000 mg/L. In present study, the COD value was determined as 1230 mg/L. In the jar tests performed in four sets which is given in Figure 2, the removal efficiency of the samples taken from the supernatant phase was examined.

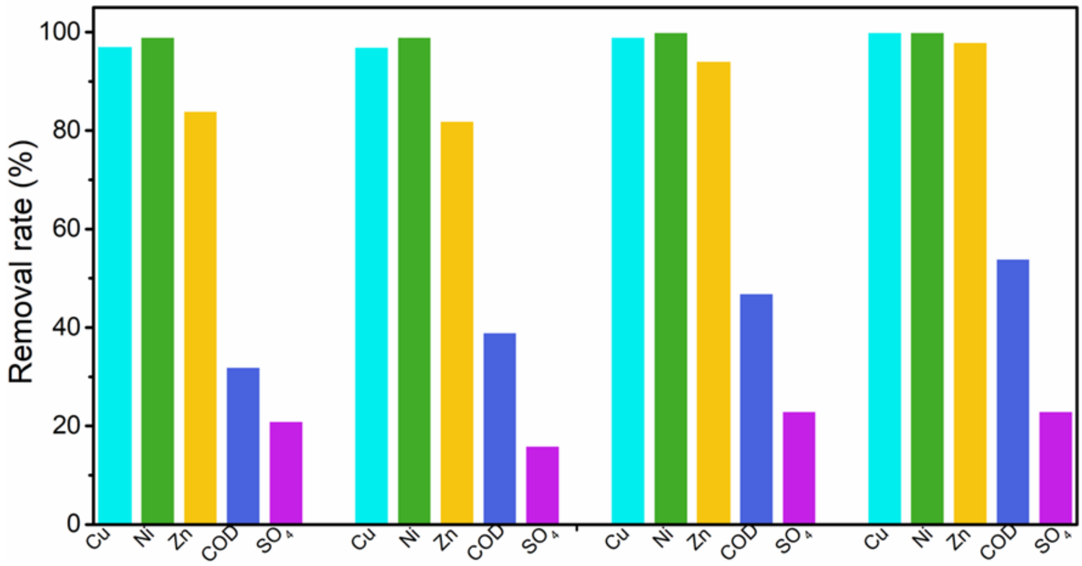

Figure 2. Removal efficiencies of pollutants in result of the jar test.

The pollutants removals were measured based on percent removal efficiency as indicated in the Equation 2 [46].

$$\text{Removal rate (\%)} = (C_0 - C) / C_0 * 100 \quad (2)$$

Where C_0 is the concentration of pollutants in the influent wastewater sample and C is the concentration of pollutants treated wastewater sample. When the chemicals used in Set 1 and Set 2 in Figure 2 were applied at the concentration and pH values specified in Table 1, the removal of Cu, Ni and Zn pollutants were achieved but, the discharge standard into the sewage could not be supplied. Additionally, with Set 1, no active settling occurred after 45 minutes. The flocs remained suspended and in a small structure. With Set 2, treatment was more effective, and it was considered to increase the applied dose slightly. In Set 3, the discharge standard to the sewage was supplied, but in order to reduce the amount of chemicals spent and reduce the cost, the concentrations specified in Set 4 were studied and these values were applied. Yatim et al. [47] investigated the removal performance by coagulation and sedimentation processes in plating industry wastewater. Cu, Zn and Cr^{3+} removal efficiencies by hydroxide precipitation at pH 10.5 using 0.8 mL of $FeCl_3$ and 1 mL of NaOH solution, respectively; it was found to be 86.61%, 99.81% and 99.99%. In the same study, Cu was removed by 93.91%, Zn by 99.37% and Cr^{3+} by 99.99% by sulphide precipitation at pH 10 using 0.2 mL of $FeCl_3$ and 0.8 mL of Na_2S solution. In present study, Cu removal was found to be higher. As can be seen from the Figure 2, Cu and Ni removals were in the range of 97-100% in all sets. Also, Zn removal was similar to [47] and was found to be 98% in set 4. Zn removal from aqueous solution by coagulation process was investigated and anionic polyacrylamide was used. Zn removal was found ~100% [48]. It was found that Zn removal gradually increased from 82% to 98% and the highest Zn removal was found in set 4. Cu removal from aqueous solution by flocculation was investigated and acrylamide and acrylic acid-based co-polymers functionalized with hydroxamic acid groups were used. At the end of the study, 67% Cu removal was founded [49]. Although the wastewater used in present study was real metal plating industry wastewater, Cu was almost completely removed (97-100%). In Table 3, the effluent concentrations of all compounds obtained from four sets and the limit values for discharge to sewage are given.

Figure 2, COD was removed to a limited and its removal remained between 32-54%, but as can be seen from Table 3, COD values met the discharge standard specified in the regulation. Polyaluminum chloride was used as coagulant using automotive wastewater and COD, Fe, Zn and Ni removal efficiencies were found to be 70%, 98%, 83% and 63%, respectively [50]. In the present study, although COD removal was limited to 54% compared to the literature, Zn and Ni removal efficiencies were found ≥97%.

Table 3. Effluent concentrations and limit values obtained from all sets.

Parameter	Effluent concentrations (mg/L)				Limit Values (mg/L) [42]
	Set-1	Set-2	Set-3	Set-4	
SO_4	2220	2344	2143	2150	1500
Cu	3.1	2.24	0,18	0.11	1
Ni	6.03	6	0.41	0.47	3
COD	838	751	654	568	3000
Zn	0.63	0.71	0.23	0.09	5
MLSS	-	-	-	< 10	1

SO_4 concentrations were found to be high due to the H_2SO_4 used in production. For SO_4 where the discharge standard could not be supplied, (5%) $BaCl_2$ (4 g, 8 g, 12 g and 16 g) was added which was the last stage of present study. When $BaCl_2$ was added at 4 g, 8 g, 12 g and 16 g the effluent concentrations were found to be 1618 mg/L, 545 mg/L, 297 mg/L and 153 mg/L, respectively. Since 545 mg/L SO_4 concentrations were reached with the 8 g $BaCl_2$ dose, the limit value (1500 mg/L) was supplied. pH values in the solution affect the efficiency of coagulation and flocculation. Optimum pH values for different metal types are stated as Ni: 10.5-11; Cu: 8-8.2; Al: 11; Cr: 8.5-9; Fe^{3+}: 3-4; Fe^{2+}: 8-9; Pb: 9-10; Zn: 9-10; Ag: 12; Cd: 9-11 [51]. While the pH values that provide the best removal of Cu through coagulation/flocculation processes in plating industry wastewater were ≥8; The best removal efficiency in Ni was at pH ≥10 [52]. In parallel with the literature, in present study, the applied pH value was kept in the range of 11-12 and the removal efficiency of Cu and Ni were 100%.

3.1. Operating cost

In the removal of pollutants, removal efficiency and operating cost are important parameters in the selection of the wastewater treatment process. In the coagulation flocculation process, the operating cost consists of the coagulant cost. As a result of the study, the cost of the chemicals used in Set 4 values and the standard of discharge to sewage was calculated. In Table 4 and 5, the consumption of coagulants added in 250 mL volumes is calculated. Since the chemicals used in set 4 supplied the criteria for discharge to sewage, the cost analysis was calculated over set 4. In addition, the dose of $BaCl_2$ used for reducing SO_4 concentration in Set 4 to supplied the discharge criteria was calculated and given in Table 6.

The amounts consumed for NaOH and $FeCl_3$ were found to be 4.11 and 2.08 g, respectively.

The amounts consumed for $Ca(OH)_2$ and $((NH4)_2S_2O_8$ were 0.55 and 0.0065 g, respectively. Table 6 shows the coagulant doses and costs for 1 m^3 of water. The total cost was found using Equation 1.

Unit Costs were added from the cash purchase price list dated 14.11.2023 of one of the chemical suppliers that the company purchases to operate the treatment plant. It was observed that the highest cost among the chemicals used was due to the use of NaOH due to the high consumption. If it is assumed that the plant works for 6 days and generates 150 m^3 of wastewater per week, 25 m^3 of wastewater will be generated daily. In this case, the cost will be 25*12.74=318.5 € (cost of coagulant used per day.)

Sludge disposal is one of the main cost elements in treatment processes. Sludge disposal methods in treatment plants spend 20-30% of the total construction cost of the plant and 50-70% of the operating cost on sludge disposal. The thickening process increases the solids density in the sludge and reduces the volume of the sludge by removing the water [53]. The chemical treatment sludge coming out of the plant is taken into the thickening process and converted into sludge cake with the help of filter presses to reduce the moisture content.

Using 250 mL sample, it was observed that 67 mL of sludge was formed at the bottom by settling the chemicals applied in set 4. Since the plant flow is 24 m_3/day 6.4 m^3/day of sludge ((24 m^3/day *67 mL)/250 mL) was formed.

Although the amount of dry solids in the sludge varies according to the treatment processes and the difference in the techniques applied, it has been reported that sludges generally contain between 5-12% dry solids, [54]

When the dry solids content is accepted as 5% in present study;

Solid content in sludge:

6.4 m^3/day*(1000 kg/m^3)*(0.05) = 320 kg/day of dry sludge generated in one day due to the chemicals used in the plant.

In filter presses, the dry matter content reaches up to 40% [55]. The sludge transformed into an easily transportable form is sent to sludge incineration plants.

The sludge from the filter press was calculated as:

Sludge from filter press: 320 kg/day / 0.4

800 kg sludge was sent to incineration plants.

Table 4. Consumption of NaOH and $FeCl_3$ according to Set 4 for 250 mL sample.

Coagulants	Concentration (%)	Consumption (mL)	Density (g/cm^3)	Consumption (g)
NaOH	45	2.75	1.495	4.11
$FeCl_3$	40	1.45	1.435	2.08

Table 5. Consumption of $Ca(OH)_2$ and $(NH_4)_2S_2O_8$ according to Set 4 for 250 mL sample.

Coagulants	Concentration/Solution (by mass) (%)	Solution Consumption (g)	Consumption calculation of the chemical used in the solution (g)
$Ca(OH)_2$	5	11	11*(5/100) = 0.55
$(NH_4)_2S_2O_8$	0.1	6.5	6.5*(0.1/100) = 0.0065
$BaCl_2$	0.5	8	8*(5/100) = 0.40

Table 6. Consumption and cost of coagulants used for 250 mL jar test in 1 m^3 wastewater.

Coagulants	Consumption in 1 m^3 Water (kg)	Unit Cost per kg (€)	Total Cost (€)
NaOH	16.44	0.45	7.40
$Ca(OH)_2$	2.2	0.10	0.22
$FeCl_3$	8.32	0.27	2.25
$(NH_4)_2S_2O_8$	0.26	2.75	0.07
$BaCl_2$	1.6	1.75	2.80
Cost of coagulants to be used for 1 m^3 wastewater			12.74 €

4. Conclusion

Coagulation-flocculation process has been investigated in order to operate the removal of heavy metals and COD from real wastewater in metal plating industry. Cu, Ni and Zn removal efficiencies were above 97% in set 4 by achieved the discharge criteria to sewage. In terms of COD removals, sewage discharge criteria were achieved. It was found that high concentration of SO_4 is detected due to the use of sulphuric acid in rinsing baths. SO_4 concentrations were also reduced by using $BaCl_2$.

The operating cost of the procedure has been studied. The total cost of the chemicals used in terms of the treatment plant is 318.5 € per day. The outcomes show that wastewater in metal plating industry have been successfully studied and heavy metals from wastewater has been removed and integrated to the wastewater treatment plant. No other coagulant other than $FeCl_3$ was used in present study. It will be more advantageous to make comparisons using different coagulants in the metal plating industry.

Sludge disposal is an important step in wastewater treatment processes, accounting for more than half of the total cost. Sludge disposal and management is therefore a serious challenge. Although the coagulation-flocculation process is one of the most widely used treatment processes in industrial wastewater due to its easy applicability and high efficiency, sludge generated as a result of the coagulation-flocculation process contains high concentrations of heavy metals can cause problems in the sludge treatment-disposal stage. Moreover, synthetic polymer flocculants can create large amounts of highly toxic sludge. For these reasons, the sludge causes serious problems in terms of environment and health. Also, heavy metals and toxicity are major disadvantages in the conversion of waste into valuable resources.

Green materials such as waste-based products and renewable materials can be used to produce environmentally friendly and biodegradable coagulants. Thus, negative impacts on the environment can be avoided and minimized and the cost of treatment can be significantly reduced.

Author contributions

Inci Karakas: Data curation, Methodology, Writing-Original draft preparation, Investigation, Writing-Reviewing and Editing.
Soner Kızıl: Writing-Original draft preparation, Investigation, Writing and Reviewing.

Conflicts of interest

The authors declare no conflicts of interest.

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