Research Article

Efficiency of ozonation process with calcium peroxide in removing heavy metals (Pb, Cu, Zn, Ni, Cd) from aqueous solutions

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Abstract
Heavy metal ions have deadly effects on all forms of life, and through the disposal of industrial wastewater, they enter water resources and, eventually, the food chain. Advanced oxidation processes can remove hazardous and non-degradable organic pollutants in aqueous solutions. Variables examined were initial concentrations of calcium peroxide and heavy metals, contact time, pH, and ozonation rate. Maximum removal rate of heavy metals by ozonation with calcium peroxide under optimal conditions (contact time = 90 min, pH = 3, heavy metal concentration = 25 mg/L, calcium peroxide concentration = 0.025 mg/L and ozonation rate = 1 g/min) in synthetic and real samples were respectively 89.8% and 64.6% for Pb, 92.1% and 73.9% for Cu, 90.4% and 69.7% for Ni, 86.9% and 59.1% for Cd, and 93.4% and 78.8% for Zn. Maximum COD removal rates in synthetic and real samples were 88.1% and 69.9%, respectively. Removal rates of heavy metals and COD under optimal conditions on wastewater from the Isfahan electroplating industry and steel company were determined. The use of the ozonation process with calcium peroxide can be recommended as a good, coefficient method for the removal of heavy metals in wastewater treatment.

Keywords Ozone · Heavy metals · Calcium peroxide · Aqueous solutions · Advanced oxidation

1 Introduction

Heavy metal ions have harmful effects on many forms of life. Through the disposal of industrial wastewater, they enter water resources and, eventually, the food chain. Due to being non-biodegradable, these metals accumulate and increase in the food chain. Among the various metal ions, lead, mercury, cadmium, and chromium (VI) top the toxicity list [1]. Physical and chemical processes are commonly used to remove heavy metals from wastewater. Several of the most common methods are ion exchange [2], membrane processes [3], coagulation and flocculation [2], adsorption and electrochemical purification [4]. Advanced oxidation is the most effective technology for the decomposition and removal of hazardous, resistant, and non-biodegradable organic pollutants and heavy metals from aqueous solutions [5–13]. They have been used widely and have taken an important place in the treatment of water and wastewater during the past decades. The main mechanism of these processes is based on the production of hydroxyl radicals (OH^-) that can oxidize most organic compounds quickly and accidentally [14, 15]. Calcium peroxide (CaO_2) can be dissolved in water slowly and release oxygen molecules. It is decomposed in water and converted to hydrogen peroxide and calcium oxide [16]. Among the advantages of CaO_2, it is compatible with the

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environment, easily transported, low in cost compared to other substances, and has long-term, high-impact effects [17].

Researchers have been successful in removing heavy metals from wastewater. For example, Mehrasbi et al. [18] in Iran used modified banana peel, Malakootian et al. [19] in Iran used dried activated sludge, Malakootian et al. [20] in Iran used nanofiltration, Mirzaienia et al. [21] in Iran used a microbial desalination cell, Hamidian et al. [22] in Iran used chitosan–clay nanocomposites, Yue et al. [23] in China used extracellular polymer, Harraz et al. [24] in Egypt used Rapid synthesis of titania–silica nanoparticles photocatalyst by a modified sol–gel method, Ray and Chen [25] in Singapore used semiconductor photocatalysis and Alatieh et al. [26] in Saudi Arabia used advanced carbon nanotubes. Upadhyay and Srivastava [27] in India survey Application of ozone on remove of heavy metals from wastewater. Zhai and Jiang [28] in China used calcium peroxide for removal of heavy metals. Massalimov et al. [29] in Russia used calcium peroxide for removal of heavy metal.

The combination of these two methods has a synergistic effect on the removal of heavy metals. Ozone was reacted with calcium peroxide and produced H_2O_2 and then radical hydroxyl. Also, calcium peroxide produced OH^- according to Eqs. (1, 2, 3, 4, 5, 6). Radical hydroxyl and OH^- reacted with heavy metals and omitted them from aqueous solutions.

$$OH^- + H_2O_2 \rightarrow H_2O + HO_2^o \tag{1}$$

$$OH^o + OH^o \rightarrow H_2O_2 \tag{2}$$

$$HO_2^o + OH^o \rightarrow H_2O + O_2 \tag{3}$$

$$OH^o + H_2O_2 \rightarrow O_2^o + H^+ + H_2O \tag{4}$$

$$OH^o + H_2O_2 \rightarrow O_2^o + OH^o + H_2O \tag{5}$$

$$M^{n+} + OH^- \rightarrow M(OH)n \downarrow \tag{6}$$

The aim of this study is to determine the efficiency of Ozonation Process with Calcium Peroxide in Removing Heavy Metals (Pb, Cu, Zn, Ni, Cd) from Aqueous Solutions.

2 Materials and methods

This empirical research was accomplished in the second half of 2017 at the Research Center of Environmental Health Engineering in Kerman University of Medical Science. Stock solutions with concentrations of 1000 ppm of heavy metals were prepared daily. The amount of ozone was determined to be 1 mg per minute. The calibration curves for pH of 3, 5, 7, 9, and 11 were drawn. Samples of the stock solution in concentrations of 25, 50, 75, and 100 mg/L were taken, mixed with concentrations of 0, 0.025, 0.05, 0.075, and 0.1 mg/L of calcium peroxide and stirred by a shaker. Next, the solution was ozonized at times of 30, 60, and 90 min. Then, the solution was centrifuged and filtered. The schematic figure for the experimental set-up is shown in Fig. 1. The gas output from the ozone generator was passed for 10 min through two containers containing 2% solution of potassium iodide (250 ml). After 10 min of ozonation, 200 ml of the potassium iodide solution was taken. Then, 10 ml of 2 N sulfuric acid was added, and the solution was titrated using 0.005 N sodium thiosulfate until the yellow color of iodine disappeared. Thereafter, 1–2 drops of starch were added and titration was continued until the blue color disappeared. Finally, the volume of consumed sodium thiosulfate was recorded. The ozone produced was determined using Eq. (7).

The real sample was prepared with wastewater from Isfahan electroplating industry and steel company. All tests were performed on the real sample. All experiments were carried out at laboratory temperature (22 ± 1 °C) and with three replicates, and the results are reported with the mean. Data was analyzed using descriptive statistics. Ultimately, the heavy metal removal rates were calculated with Eq. (8) [34]:

$$\text{Ozone concentration}\left(\frac{\text{mg}}{\text{L}}\right) = \frac{(A + B) \times N \times 24}{T(\text{min})} \tag{7}$$

A: Consumption of sodium thiosulfate for the first container (ml), B: Consumption of sodium thiosulfate for the second container (ml), T: Ozonation time (min), N: Normality of sodium thiosulfate

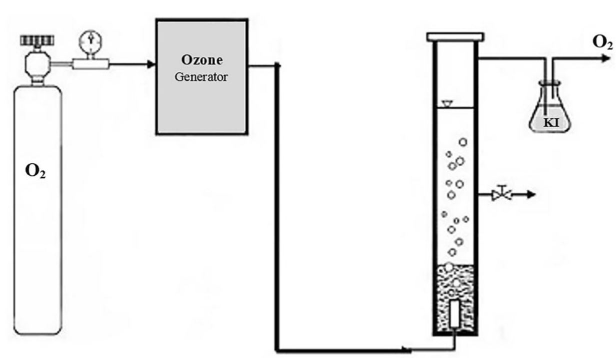

Fig. 1 Schematic figure for experimental set-up

$$\text{Removal efficiency}(\%) = \frac{Ci - Cf}{Ci} \times 100 \quad (8)$$

C_i: The initial concentration of heavy metals (mg/L), C_f: The residue concentration of heavy metals after centrifuge (mg/L)

Heavy metals (lead, copper, nickel, cadmium, zinc), calcium peroxide 98%, sulfuric acid, and sodium hydroxide were purchased from Merck (Germany). Ozone generator devices (ARDA, model MOG-5G/H), air compressor (model FL25), pH meter (model EDT-R357), flow meter, centrifuge (model-150), and atomic adsorption (model: Younglin AAS 8020, YL Instrument Company, South Korea) were used.

2.1 Real sample

Tests in this research were first performed using synthetic solutions. After determining optimal conditions, tests were done on actual wastewater from the Isfahan electroplating industry and steel company. The results regarding the quality of the wastewater from the Isfahan Steel Company are shown in Table 1.

3 Results and discussion

3.1 Effect of pH on removal rate

The results of tests for the effect of pH on removal rate of heavy metals from aqueous solutions are shown in Fig. 2.

Maximum removal rates with a contact time of 90 min and pH = 3 in synthetic samples were 89.8% for lead, 92.1% for copper, 90.4% for nickel, 86.9% for cadmium, and 93.4% for zinc. Removal rate was reduced by increasing pH; the removal rates with a contact time of 90 min and pH = 11 in the synthetic samples were 48.3%, 67.6%, 62.7%, 43.5%, and 70.9% for lead, copper, nickel, cadmium, and zinc, respectively.

Table 1 Quality of turbidity, COD, BOD_5, TSS, EC, pH, VFS, EC, and heavy metals of wastewater from Isfahan electroplating industry and steel company

Parameter	Amount
COD	50 mg/L
BOD_5	97 mg/L
TSS	62 mg/L
VFC	481 lg/L
pH	7.4
Turbidity	55 mg/L
EC	20.7 ds/m
Cu	0.06 mg/L
Cd	0.33 mg/L
Zn	0.12 mg/L
Pb	0.02 mg/L
Ni	0.038 mg/L
Hg	0.41 mg/L
Cr	0.1 mg/L

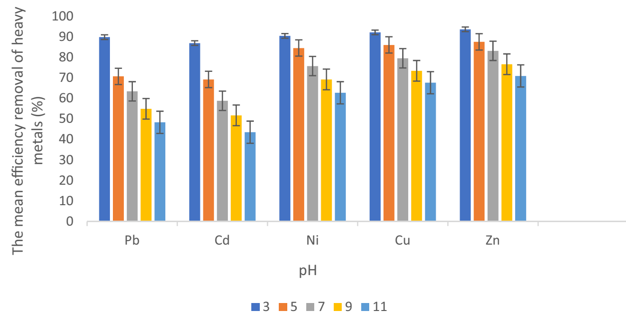

Fig. 2 Effect of pH on removal rate of heavy metals (lead, copper, nickel, cadmium, and zinc) from aqueous solutions by the ozonation process with calcium peroxide

Calcium carbonate and hydroxyl radicals produced from calcium peroxide in acid solution. These compounds oxidize heavy metals. Calcium peroxide reaction in an acid solution is carried out according to Eqs. (9, 10, 11). The mechanism of ozone decomposition with hydrogen peroxide done according to Eq. (3), that cause to product of hydroxyl radicals.

$$CaO_2 + 2H^+ \rightarrow Ca^{2+}(aq) + 2H_2O_2 \quad (9)$$

$$Ca\,(OH)_2 + 2H^+ \rightarrow Ca^{2+}(aq) + 2H_2O \quad (10)$$

$$Ca\,(OH)_2 + CO_2 \rightarrow CaCO_3(s) + H_2O \quad (11)$$

Calcium peroxide has stability and high solubility in low pH [30]. Hydrogen peroxide from the ozonation process and calcium peroxide are very effective in the oxidation of organic compounds [31]. Heavy metals may be present in the water and wastewater in both free form and in complex compounds. Ozone oxidizes the transition metal to their higher oxidation state in which they usually form less soluble oxides; easy to separates by filtration. Other metals like cadmium, chromium, cobalt, copper, lead, manganese, nickel, zinc etc. can be treated in a similar way. Broadly speaking, ozone oxidizes these heavy metals to form metallic oxides or hydroxides, which precipitate off and can easily be removed from water. Ozone is used for eliminating heavy metals from the effluent produced by many types of industries, a typical case being that of mineral extraction plant [27]. Then with filtration the heavy metals were separated.

Removal rate was decreased by increasing pH. The results of this study were consistent with the results of studies by Malakootian et al. [32] in Iran on removing reactive red 198 by calcium peroxide, Qian et al. [33] in China on removing toluene by calcium peroxide, Northup and Cassidy [16] in the USA on the modification of the Fenton method by calcium peroxide, and Rezaee et al. [34] in Iran on reactive blue 19 by UV/H_2O_2 process. The production of hydrogen peroxide and the rate of dissolution of calcium peroxide increases in acidic pH [30].

3.2 Effect of calcium peroxide concentration on removal rate

The results of tests for the effect of calcium peroxide concentration on the removal rate of heavy metals from aqueous solutions are shown in Fig. 3.

The removal rates of heavy metals with a calcium peroxide concentration of 0.025 mg/L in synthetic samples were 89.8%, 92.1%, 90.4%, 86.9% and 93.4% for lead, copper, nickel, cadmium, and zinc, respectively. Removal rate was reduced when the calcium peroxide concentration was increased; removal rate with a calcium peroxide concentration of 0.1 mg/L in synthetic samples were 74.7%, 54.4%, 52%, 45%, and 57.3% for lead, copper, nickel, cadmium, and zinc, respectively.

The mechanism of heavy metals removal from water by calcium peroxide was attributed to the formation of metal oxides and hydroxides precipitation from the reaction of metal ions with H_2O_2 and OH^- generated from calcium peroxide, Eq. (6) [28]. The study of Liu et al. proved that calcium peroxide was more efficient than lime or limestone to remove Cu, Ni, Zn, Cd and Pb from acid mine drainages [35].

$$\text{Equation 6}: \quad M^{n+} + OH^- \rightarrow M(OH)n \downarrow$$

Removal rate was decreased when the calcium peroxide concentration was increased. The results of this study were consistent with those of Honarmandrad et al. [36] in Iran on the use of the ozonation process with calcium peroxide for the removal of metronidazole antibiotic from aqueous solutions, Malakootian and Honarmandrad [37] in Iran on use of the ozonation process with calcium peroxide for removal of reactive blue dye from textile wastewater and Rahmani et al. [31] in Iran on ciprofloxacin removal by the ozonation process with calcium peroxide from aqueous solutions. A high presence of oxidant in the environment reduces radicals and makes that part of the oxidant radicals convert to intermediate substances and other compounds [36]. In excess concentration of hydrogen peroxide, hydroxyl radicals that are less active are formed Eq 1, and in excess concentration of radical hydroxyl are transformed into hydrogen peroxide through dimerization Eq. 2. Due to the reaction of Eqs. 3, 4 and 5 hydroxyl radicals, did not participated in the oxidation reaction and under other reactions, they been decompose into anions of OH and oxygen [38, 39].

$$\text{Equation 1}: \quad OH^- + H_2O_2 \rightarrow H_2O + HO_2^o$$

$$\text{Equation 2}: \quad OH^o + OH^o \rightarrow H_2O_2$$

$$\text{Equation 3}: \quad HO_2^o + OH^o \rightarrow H_2O + O_2$$

$$\text{Equation 4}: \quad OH^o + H_2O_2 \rightarrow O_2^o + H^+ + H_2O$$

$$\text{Equation 5}: \quad OH^o + H_2O_2 \rightarrow O_2^o + OH^o + H_2O$$

3.3 Effect of initial concentrations of heavy metals on removal rate

The results of tests for the effects of heavy metal concentrations on the removal rate of heavy metals from aqueous solutions are shown in Fig. 4.

The initial concentration of a heavy metal is one of the most important parameters affecting the removal of heavy metals from aqueous solutions. Heavy metal removal

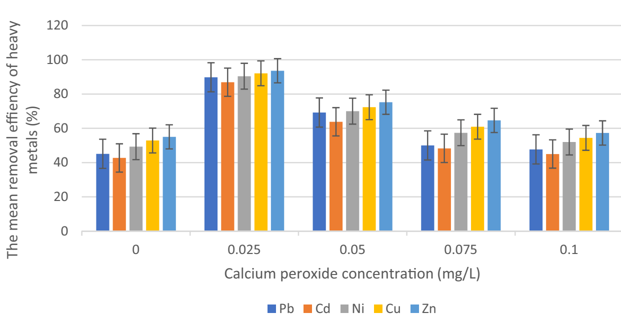

Fig. 3 Effect of calcium peroxide concentration on removal rate of heavy metals (lead, copper, nickel, cadmium, and zinc) by the ozonation process with calcium peroxide from aqueous solutions

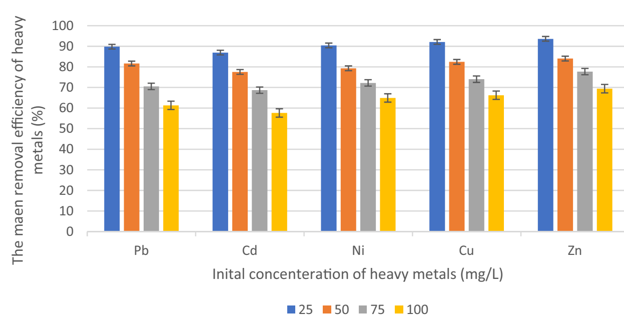

Fig. 4 Effect of initial heavy metal concentrations on removal rate of heavy metals (lead, copper, nickel, cadmium, and zinc) by the ozonation process with calcium peroxide from aqueous solutions

rates with a heavy metal concentration of 25 mg/L in the synthetic samples were 89.8% for lead, 92.1% for copper, 90.4% for nickel, 86.9% for cadmium, and 93.4% for zinc. removal rate was reduced when the initial heavy metal concentration was increased; removal rates with a heavy metal concentration of 100 mg/L in synthetic samples were 61.3% for lead, 66.2% for copper, 64.9% for nickel, 57.6% for cadmium, and 69.4% for zinc.

Removal rate decreased when the initial heavy metal concentration was increased. The results of this study were consistent with those of studies by Ghaneian et al. [40] in Iran on reactive blue 19 by Jujube Stems powder and Olyaie et al. [41] in Iran on the removal of arsenic contamination from aqueous solutions by calcium peroxide nanoparticles. By increasing the concentration of pollutants, oxidizing substances such as the ozone molecule and radical hydroxyl were increased. When the concentration of pollutants was lower, the removal rate was increased, pollutants were more completely decomposed, and fewer intermediate materials were produced [31].

3.4 Effect of contact time on removal rate

The results of tests for the effect of contact time on the removal rate of heavy metals from aqueous solutions are shown in Fig. 5.

Removal rates of heavy metals with a contact time of 90 min in the synthetic samples were 89.8%, 92.1%, 90.4%, 86.9%, and 93.4% for lead, copper, nickel, cadmium, and zinc, respectively. The results showed that removal rate decreased when contact time was reduced; removal rates with a contact time of 30 min in the synthetic samples were 66.5%, 71.9%, 68.4%, 59.3%, and 71.9% for lead, copper, nickel, cadmium, and zinc, respectively.

Removal rate increased when contact time was increased. The results of this study were consistent with the results of studies by Bahrami et al. [42] in Iran on the removal of metronidazole from aqueous solutions using the ozonation process, Malakootian et al. [32] in Iran on removing reactive red 198 by calcium peroxide, and Malakootian et al. [20] in Iran on removing Pb, Cd, Cr and Cu by nanofiltration. With increases in contact time, the production of radical hydroxyl and ozone molecule were also increased, and consequently, removal rate was increased.

3.5 Effect of ozonation alone and ozonation with calcium peroxide on removal rate

The effect of ozonation without calcium peroxide and ozonation with calcium peroxide on the removal of heavy metals are shown in Figs. 6 and 7. The maximum removal rates by, ozonation with calcium peroxide 89.8%, 92.1%, 90.4%, 86.9% and 93.4% for lead, copper, nickel, cadmium, and zinc and ozonation without 45.1%, 52.9%, 49.3%, 42.7% and 55% were respectively. The removal rate of ozonation with calcium peroxide was greater than the other two methods. According to Eqs. (12 to 15), calcium peroxide produced hydrogen peroxide and reacted with ozone to produce radical OH°, and radical OH° has a great effect on heavy metal oxidation [31, 43].

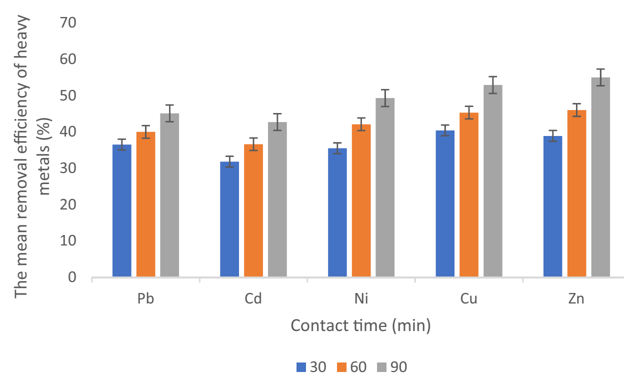

Fig. 6 Effects of ozonation alone on the removal of heavy metals

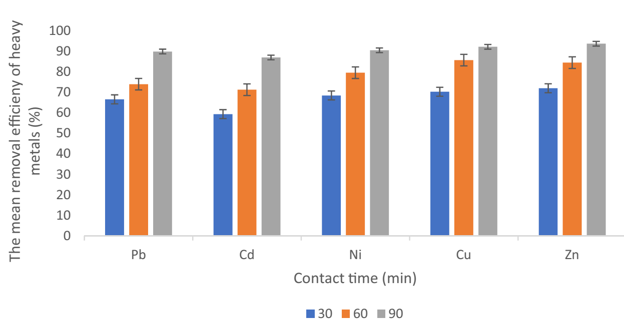

Fig. 5 Effect of contact time on removal rate of heavy metals (lead, copper, nickel, cadmium, and zinc) by the ozonation process with calcium peroxide from aqueous solutions

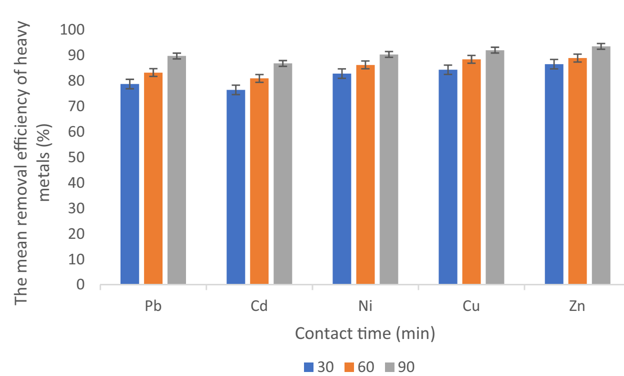

Fig. 7 Effects of ozonation with calcium peroxide on the removal of heavy metals

$$CaO_2 + 2H^+ \rightarrow Ca_2 + 2H_2O_2 \tag{12}$$

$$OH^o + H_2O_2 \rightarrow H_2O + HO_2^o \tag{13}$$

$$OH^o + HO_2 \rightarrow H_2O + O_2 \tag{14}$$

$$HO_2^o + HO_2 \rightarrow H_2O_2 + O_2 \tag{15}$$

3.6 Effect of optimal conditions on removal rate of real sample

The results of tests determining the quality of the wastewater from the Isfahan electroplating industry and steel company are shown in Table 1.

Under optimal conditions (pH = 3, calcium peroxide concentration = 0.025 mg/L, heavy metals concentration = 25 mg/L, and contact time = 90 min), the removal rate for Cu 92.1%, Pb 89.8%, Ni 90.4%, Zn 93.4%, and COD 88.1% in the synthetic sample were 100% respectively, and in the real sample, removal rates were 73.9% for Cu, 64.6% for Pb, 69.7% for Ni, 78.8% for Zn, and 69.9% for COD. The removal rates for Cu, Pb, Ni, Zn, and COD were 92.1%, 89.8%, 90.4%, 93.4%, and 88.1%, respectively, in the synthetic sample and 73.9%, 64.6%, 69.7%, 78.8%, and 69.9%, respectively, in the real sample.

The reduced removal rate of heavy metals and COD in the real sample compared to that of the synthetic samples can be explained by the existence of organic substances such as turbidity, TSS, and sulfate in the wastewater from the Isfahan electroplating industry and steel company. The reduction in removal rate in the real wastewater solution was caused by interferences, including the cations and anions present in the wastewater.

Maximum removal rate of heavy metals by the ozonation process with calcium peroxide under optimal conditions (contact time = 90 min, pH = 3, concentration of heavy metals = 25 mg/L, and calcium peroxide concentration = 0.025 mg/L) in the wastewater of the Isfahan electroplating industry and steel company was decreased. This decrease in removal rate was caused by interferences, including the cations and anions present in the wastewater, turbidity, TSS, sulfate, and ammoniac. The sulfate ion is combined with free radicals of hydroxyl and converts them to inorganic hydroxyl radicals. Due to the fact that the power of the inorganic hydroxyl radical is lower than that of the free radical hydroxyl, the removal rate of heavy metals and COD in the real sample was less than the synthetic sample.

4 Conclusions

Maximum removal rate of heavy metals by the ozonation process with calcium peroxide under optimal conditions (contact time = 90 min, pH = 3, concentration of heavy metals = 25 mg/L, concentration of calcium peroxide) in synthetic and real samples 0.025 mg/L calcium peroxide in synthetic and real samples were 89.8% and 64.6% for lead, 92.1% and 73.9% for copper, 90.4% and 69.7% for nickel, 86.9% and 59.1% for cadmium, and 93.4% and 78.8% for zinc. Maximum removal rate of COD in synthetic and real samples were 88.1% and 69.9%, respectively. The use of the ozonation process with calcium peroxide is a good method because of its relatively high efficiency in removing heavy metals, its strong oxidization, and the decomposition of resistant organic compounds. It can be recommended as a coefficient method for the removal of heavy metals from industrial wastewater.

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Compliance with ethical standards

Conflict of interest We have no conflict of interest to declare.

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