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The Effect of dissolved air flotation (DAF) assisted by ultrasonic waves on heavy metal removal during industrial recycled sludge thickening

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Abstract

Management of excess sludge generated in wastewater treatment plants, particularly industrial sludge containing heavy metals, is a major environmental and public health challenge. In this study, the efficiency of dissolved air flotation (DAF) assisted by ultrasonic waves was investigated for the removal of selected heavy metals, including chromium, lead, nickel, and copper, during the thickening of recycled sludge. Sludge samples were collected from the Amir Kabir Industrial Town wastewater treatment plant in Kashan, Iran, and experiments were conducted at the laboratory scale under batch conditions. To optimize operational parameters, a jar test was performed to determine the optimal pH and ferric chloride coagulant dosage, which were found to be 7.5 and 200 mg/L, respectively. The effects of operating pressure (3, 5, and 7 atm), flotation time (5 and 10 min), ultrasonic power (75 and 150 W), and coagulant addition on heavy metal removal efficiency were evaluated. The results demonstrated that operating pressure had the most significant effect on heavy metal removal. The highest removal efficiencies for copper (81.37%), nickel (67.59%), and chromium (85.86%) were achieved at a pressure of 5 atm, while the maximum removal of lead (72.31%) occurred at 3 atm. Variations in flotation time and ultrasonic power did not result in statistically significant changes in removal efficiency, whereas coagulant application significantly enhanced the removal of copper and chromium. Overall, the findings indicate that the DAF process is an effective method for reducing heavy metal concentrations in industrial sludge, with operating pressure identified as a key parameter for optimizing system performance.

Keywords: Dissolved Air Flotation (DAF), Ultrasonic Waves, Heavy Metal Removal, Sludge Thickening, Industrial Wastewater, Coagulation, Ferric Chloride

Introduction

With the rapid development of urban areas and industrial sectors, the generation of domestic and industrial wastewater has significantly increased, consequently requiring extensive treatment and resulting in considerable sludge production [1-3].

Therefore, management of the excess sludge generated in wastewater treatment plants has become a critical priority [4]. Sludge handling is a major and costly component of wastewater treatment and should be directed toward reducing sludge volume as well as the associated pollutants, including heavy metals, organic compounds, microorganisms, and toxic substances, through appropriate thickening and dewatering processes [3, 5]. Numerous studies have reported the presence of various heavy metals in sludge, which may be discharged into surface waters, air, and soils [6-8]. Thus, the presence of heavy metals, especially in industrial wastewater and its sludge, poses a serious global threat to the environment and public health [9, 10]. The recommended concentration limits [11, 12] and some of the environmental impacts of heavy metals considered in this study are summarized in Table 1.

Table 1. Recommended concentrations and some effects of heavy metals (Cu, Ni, Cr, and Pb)

Heavy metal	**Recommended concentrations (WHO)**		**Human health effects**	**Environmental effects**
	Drinking water usage (mg.L^{-})	Agricultural wastewater usage (mg.L^{-})		
Cu	2	0.2	Gastrointestinal effects [11]- Oxidative damage and death of cells- Lymphoma cancer- Cognitive disorder [13]	Reduced hydroponic growth of some plants such as alfalfa and lettuce- Changing nutritional and enzymatic content of plants- Cilantro phosphorus reduction [14] Reduced growth- swimming capacity and reproduction in fishes [15]
Ni	0.07	0.2	Skin dermatitis- Carcinogenic potential- Damage and death of cells- Indirect effect of tumorigenic on cells- Allergic response- Headache- Malaise- Diarrhea- Fever- Arthralgia [16]	Morphological and histopathological damages in fishes- Reduction in egg production, respiratoty and swim performance in fishes [15]
Cr	0.05	0.1	Stomach and respiratory disorders- Skin rashes- Reduction level of immune system- Kidney and liver damages- DNA damage- digestive tract and lungs cancer [17-19]	Mutagenic and carcinogenic effects for animals- Mutagenic effects for bacteria [20]
Pb	0.01	5	Nervous effects- Mortality caused by mainly cardiovascular problems- Kidney disorder- Blood pressure- Pregnancy disorders [11]	Disorder in plants photosynthesis and respiration- Imbalance of plants enzyme activity [21]

Dissolved air flotation (DAF) is one of the widely used mechanical methods in water and wastewater pretreatment, treatment, and sludge thickening processes [22-24]. However, previous studies indicate that the application of DAF specifically for sludge thickening has been less common compared with other uses. Compared with conventional processes, DAF offers several advantages, among which a high surface overloading rate (SOR) is the most significant [25]. In addition, its other benefits include resistance to cold water conditions, low space requirements, high operational flexibility, increased dissolved oxygen, and relatively low capital and operational costs [26, 27]. These characteristics have encouraged the application of DAF for the removal of various pollutants such as heavy metals, ammonium, sulfate ions, and disinfection by-products [25, 26, 28] . For example, one study reported that the DAF process using chitosan as a coagulant was able to remove heavy metals such as Cd, Ni, Mn, and Pb by 29%, 27%, 31%, and 29%, respectively [29].

Flotation performance can also be enhanced by other factors that improve its efficiency, among which sonication is an important one used in advanced flotation processes [30]. Another study demonstrated that sonication increased copper removal efficiency by up to 3.5% during the flotation process [31].

The main objective of this study is to evaluate the efficiency of the DAF process assisted by ultrasonic waves in the removal of selected heavy metals (chromium, lead, nickel, and copper) during the thickening of recycling sludge produced at the Amir Kabir Industrial Town wastewater treatment plant in Kashan, Iran.

Materials and methods

Sampling

Amir Kabir Industrial Town comprises several food and chemical manufacturing units that discharge a combination of industrial and partially domestic wastewater to a centralized wastewater treatment plant employing an activated sludge process, with a daily treatment capacity of 350 m³. The required sludge volume was collected once from the aeration tank, near the return activated sludge influent. The collected sample was then transported to the Research Laboratory of the School of Public Health, Kashan University of Medical Sciences. Subsequently, the physicochemical characteristics of the sludge were analyzed, and the sample was stored at 4 °C until further examination.

Analytical methods

During the experiments, standard APHA procedures [32] were employed to determine total suspended solids (TSS) and total solids (TS). Heavy metals, pH, and electrical conductivity (EC) were also measured using an Inductively Coupled Plasma (ICP) analyzer (Optima 2100 DV, USA), a pH meter (Tajhizat Sanjesh, pH 262, Iran), and a conductometer (Metrohm 644, UK), respectively.

Statistical method

SPSS version 16 and Microsoft Excel were used for data analysis. Following data collection, statistical analyses including ANOVA, independent t-tests, and post-hoc comparisons were performed.

Apparatus and procedure

A laboratory-scale batch dissolved air flotation (DAF) system was employed to investigate the removal of heavy metals from the supernatant of flotated sludge. The DAF system consisted of three main components: a saturation tank, a flotation tank (10 × 10 × 35 cm), and an air compressor. The flotation tank was mounted on an ultrasonic transducer, where coagulation, flocculation, flotation, and sonication were performed sequentially. A jar test was conducted to optimize pH and coagulant dosage (ferric chloride).

In this study, the air-to-solids ratio (A/S) was used to determine the required volumes of sludge and saturated water in the flotation tank. A/S is a key design parameter in DAF systems, typically ranging from 0.005 to 0.06 mL/mg for sludge thickening. Equation (1) was used to calculate the A/S ratio.

$$\frac{A}{S} = \frac{1.3\,Sa(fP-1)r}{Su.v} \qquad (1)$$

Where Sa, f, P, and rrr represent the air solubility (18.7 mL/L at 20 °C), the ratio of wastewater air solubility to water air solubility (0.5), the operating pressure (atm), and the volume of saturated water (mL) in the flotation tank, respectively. Additionally, SuS_uSu is the suspended solids concentration (mg/L), and vvv is the volume of sludge in the flotation tank (mL) [33]. The ratio r/vr/vr/v corresponds to the saturated water recycling rate, with the total volume of sludge and saturated water maintained at 3000 mL.

For the experiments, a predetermined volume of water was pressurized in the saturation tank at 3, 5, and 7 atm for a specified duration. Simultaneously, based on the calculated r/vr/vr/v, a defined volume of sludge was introduced into the flotation tank, followed by coagulation (180 rpm for 1 min) and flocculation (30 rpm for 20 min) steps, both with and without the addition of coagulant. The saturated water was then released into the flotation tank to reach a total volume of 3000 mL. After flotation periods of 5 and 10 minutes, sonication was applied at power levels of 75 and 150 W.

Subsequently, the required volume of supernatant for heavy metal analysis was collected via a valve located near the bottom of the flotation tank. The samples were then digested, filtered, and analyzed using inductively coupled plasma (ICP) spectroscopy in accordance with ISO 11885 standards [34]. Finally, the removal efficiency of each heavy metal by the DAF process was calculated using Equation (2).

$$C = \frac{A - B}{A} \times 100 \qquad (2)$$

Where A represents the heavy metal concentration in the diluted sludge sample (i.e., the mixture of sludge and saturated water based on the calculated r/v), B is the heavy metal concentration in the effluent supernatant after flotation, and C denotes the removal efficiency. To ensure accuracy, all experiments were performed in triplicate.

Results and discussion

pH and coagulant dose optimization

In this study, ferric chloride (Merck Co., Germany) was selected as the coagulant due to its widespread use and effectiveness in removing suspended solids and heavy metals from wastewater. A jar test was conducted to determine the optimal operational conditions for coagulation. During the jar test, various pH values and coagulant doses were tested to evaluate their impact on sludge settling and supernatant clarity. Based on these experiments, the optimum pH was found to be 7.5, which provided the best coagulation performance, and the optimal ferric chloride dose was determined to be 200 mg/L, ensuring maximum removal of suspended solids and heavy metals while minimizing chemical consumption.

Sludge properties

As described above, after sludge collection, its physicochemical properties were determined, and the results are summarized in Table 2.

r/v ratio

The ratio was calculated for each operating pressure using Equation (1), with the air-to-solids ratio (A/S) set at 0.005 mL/mg. Based on the determined parameters, the calculated ratios for pressures of 3, 5, and 7 atm were 400%, 135%, and 87%, respectively.

Table 2. Sludge sample contents

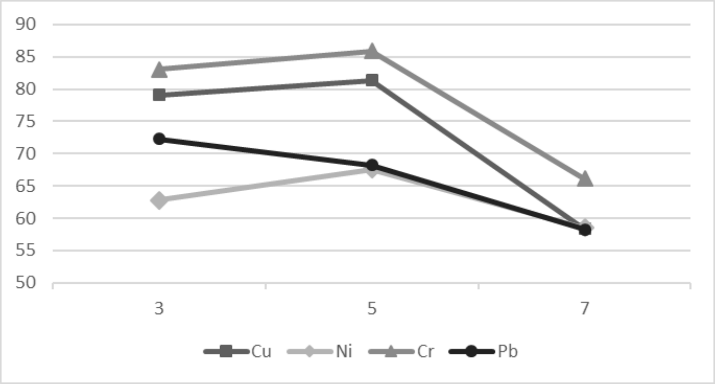
Component	Unit	Content
Turbidity	NTU	4860
pH	-	7.2
TSS	mg/L	9854
TS	mg/L	11560
EC	µS/cm	4815
Cr	mg/L	4.690
Cu	mg/L	2.434
Pb	mg/L	0.174
Ni	mg/L	0.378

Heavy Metals Removal

Pressure effect

As shown in figure 1, the maximum removal efficiencies of Cu, Ni, and Cr were 81.37%, 67.59%, and 85.86%, respectively, at an operating pressure of 5 atm, while the maximum removal of Pb (72.31%) was achieved at 3 atm. Furthermore, ANOVA results indicated that, at a significance level of $p < 0.05$, all heavy metals except Ni showed a statistically significant relationship with operating pressure. A similar significant relationship between heavy metal removal and pressure has been reported by Palaniandy et al. [35].

Figure 1. The Effect of pressure on heavy metals removal efficiency



Flotation Time effect

During the flotation periods of 5 and 10 minutes, the variations in the removal of all heavy metals were not statistically significant. The maximum removal efficiencies of Cu, Ni, and Pb were 74.48%, 63.93%, and 68.24%, respectively, at 5 minutes, whereas Cr exhibited its highest removal efficiency of 81.52% at 10 minutes.

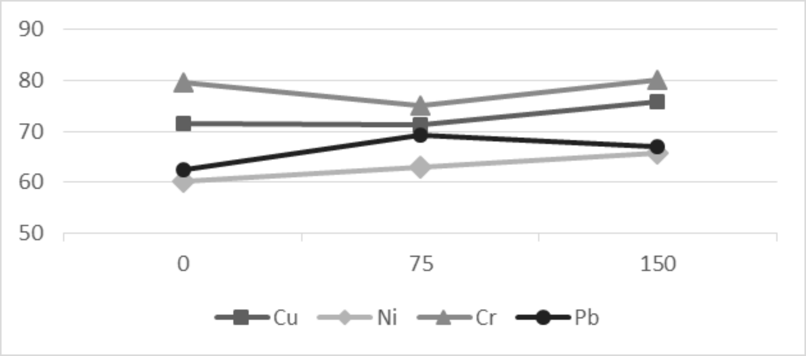
Sonication effect

No significant correlation was observed between the removal efficiencies of the heavy metals and the application of ultrasonic waves. As shown in figure 2, the maximum removal efficiencies of Cu, Ni, and Cr were 75.86%, 65.64%, and 80.24%, respectively, at an ultrasonic power of 150 W, whereas the highest removal efficiency for Pb (69.27%) was achieved at 75 W.

Cogulation effect

The effect of coagulation was significant only for Cu and Cr, with higher removal efficiencies observed when the coagulant was applied (78.22% and 80.55%, respectively). In contrast, Ni and Pb exhibited removal efficiencies of 63.83% and 67.03%, respectively, with no significant changes observed due to coagulation.

Figure 2. The Effect of sonication on heavy metals removal efficiency



Conclusion

This study was conducted to evaluate the combined effects of coagulation, dissolved air flotation (DAF), and sonication on the removal of heavy metals during sludge thickening. The results indicated that, on average, Cr achieved the highest removal efficiency among the studied metals, which may be related to its relatively lower atomic weight and higher mobility in the aqueous phase, facilitating its separation during the flotation process. Cu and

Pb showed moderate removal efficiencies, while Ni exhibited the lowest removal, likely due to differences in chemical speciation and interactions with the coagulant. Among the operational parameters investigated, applied pressure had the most pronounced effect on heavy metal removal, suggesting that increasing the air-to-solids ratio in the DAF system enhances the flotation and separation of metal-laden particles. In contrast, other factors such as flotation time, ultrasonic power, and coagulant addition had variable or less significant impacts depending on the specific metal. These findings highlight the importance of optimizing system pressure as a key parameter in designing efficient DAF-based sludge treatment processes for heavy metal removal.

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