Available online at http://ijcpe.uobaghdad.edu.iq and www.iasj.net
Iraqi Journal of Chemical and Petroleum Engineering
Vol. 24 No. 4 (December 2023) 151 – 159
EISSN: 2618-0707, PISSN: 1997-4884

Flotation of Chromium Ions from Simulated Wastewater Using Air Microbubbles

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Abstract

A microbubble air flotation technique was used to remove chromium ions from simulated wastewater (e.g. water used for electroplating, textiles, paints and pigments, and tanning leather). Experimental parameters were investigated to analyze the flotation process and determine the removal efficiency. These parameters included the location of the sampling port from the bottom of the column, where the diffuser is located to the top of flotation column (30, 60, and 90 cm), the type of surfactant (anionic, SDS, or cationic, CTAB) and its concentration (5, 10, 15, and 20 mg/L), the pH of the initial solution (3, 5, 7, 9, and 11), the initial contaminant concentration (10, 20, 30, and 40 mg/L), the gas flow rate (0.1, 0.2, 0.3, and 0.5 L/min), and the contact time (5, 10, 15, 20, 25, 30, and 35 min). The experimental results revealed that the highest removal efficiency (95%) was achieved in 20 min with a pH of 7, a flow rate of air 0.5 L/min, an SDS surfactant concentration of 15 mg/L, and a pollutant concentration of 30 mg/L at a sampling port height of 30 cm. The use of microbubbles in comparison to normal bubbles, resulted in a 56% improvement of the removal efficiency. The flotation process follows a first-order kinetics.

Keywords: Flotation, Microbubble, Heavy metal, Chromium ion, Surfactant.

Received on 15/05/2023, Received in Revised Form on 01/10/2023, Accepted on 04/10/2023, Published on 30/12/2023

https://doi.org/10.31699/IJCPE.2023.4.15

1- Introduction

Due to water poisoning, heavy metals entering the ecosystem are now a global problem. The main culprits for this are modern chemical industries like mining, battery production, and tanneries [1]. Heavy metals like lead, arsenic, cadmium, chromium, and zinc can negatively impact health, leading to high blood pressure, exhaustion, sleep issues, mood swings, and memory loss [2]. Heavy metals' solubility in water facilitates their absorption by living organisms and their accumulation in the human body when they enter the food chain. Exceeding the permitted concentration level can be harmful [3]. Chromium (VI), a hazardous metal, comes from industries like canning, steel production, and paint manufacturing, leading to water pollution [4]. Elevated Cr (VI) levels can damage the liver, andkidneys, and cause stomach cancer, bronchitis, and skin irritation [5]. Preventing toxic ion release is crucial. Research on efficient water purification methods is growing due to limitations and costs of traditional approaches for managing metal ion concentrations ranging from 1.0 to 100.0 mg/L [6]. Heavy metals reach water through air deposition, erosion, sewage, and mining [7]. Water quality is vital for aquatic life and human well-being [8]. the World Health Organization (WHO) permissible limit for chromium (Cr) ions is 0.05 mg/L, based on toxicity [9]. Chromium occurs naturally alongside elements like ferric chromite, crocoite, and chrome ochre. As the sixth most abundant transition metal in the Earth's crust, it is highly prevalent [10]. Cr (III) being more beneficial to human health and less harmful than Cr (VI) [11]. The toxic compound chromium (VI) is often found in industrial wastewater, causing health problems [12]. Chromium finds applications in textiles, metal finishing, leather tanning, and electroplating [13]. The Environmental Protection Agency (EPA) sets 0.1 mg/L limit for hexavalent chromium in drinking water [14]. Traditional water treatment techniques, such as absorption, have been assessed for water and wastewater treatment [15], ultrafiltration [16], coagulation and direct microfiltration [17], electrochemical method [18], reverse osmosis [19], solvent extraction [20], using raw scrap with zero-valent iron and zero-valent aluminum [21] and ion-exchange methods [22]. Conventional water treatment methods remove contaminants but face issues such as energy use, complexity, and hazardous waste [23].

Flotation is a highly effective separation method used in various industries. Currently, water treatment uses micro-sized air and oxygen bubbles because they are more effective than conventional techniques [24, 25, 26]. Using microbubble-assisted traditional flotation with 30-100 μm

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*Corresponding Author: Name: Rossen Sedev, Email: r.sedev@ecu.edu.au microbubbles effectively recovers sub-13 µm mineral particles and separates pollutants. Microbubbles excel in boosting separation efficiency, especially for sub-5 µm ore particles, by reducing the bubble size distribution and increasing bubble surface flux for improved fine particle capture [27]. To remove heavy metals from simulated wastewater by flotation, this method involves adding surfactants to the solution and injecting compressed air to pass through a diffuser, generating microbubbles and creating a dynamic interface between the gaseous and liquid phases, thus causing the microbubble interface to contain a complex of contaminated material. The mineral particles bind to the surfactant molecules with their different charges gathered at the bubble interface [28]. The outermost layer of the bubble is referred to as the liquid phase. Depending on the type of operation being performed, this liquid phase may consist of the same material as the bubble's shell or it may contain a surfactant [29]. To capture the metal particles, an anti-charged surfactant is chosen. Due to the positive surface charge of the material being tested, an anionic surfactant (sodium dodecyl sulfate) was chosen for the experiments [30]. Ion flotation is a multi-component, multi-phase process involving interactions between metal ions and surfactant molecules, surfactant molecules, and air bubbles under complex hydrodynamic conditions [31]. Particle-bubble interaction in flotation starts with collisions and is influenced by fluid hydrodynamics. The movement of bubbles, particles, and liquid flow within a flotation cell governs this process [32]. Particle and bubble characteristics greatly affect attachment and stability. Successful attachment relies on quick water film drainage during impact. The efficiency of flotation strongly depends on bubble/particle interactions [31].

This study presents an innovative method for treating contaminated water from industries like electroplating, textiles, paints and pigments, tanning leather, and battery manufacturing. It uses controlled compressed air from an air compressor to introduce microbubbles via a premium micro-porous ceramic diffuser in a flotation column containing a simulated industrial wastewater with added surfactant. This process creates complexes with the heavy metal contaminants, which are carried to the surface by the microbubbles, effectively removing the pollutants. Regular sampling from the column allows researchers to track changes in heavy metal concentration over time and calculate removal efficiency. This approach holds promise for addressing contamination issues in these industries, offering an eco-friendly wastewater treatment solution.

2- Experimental Section

2.1. Materials

Some of the properties of the materials used in this study are listed in Table 1.

2.2. Equipment

The experimental setup is depicted in Fig. 1. The vertical column is where flotation happens. There are 11 sampling ports along the height of the column. The equipment used is listed in Table 2.

Table 1. Details of the Materials Used

Property	**Sodium Dodecyl Sulfate (SDS)**	**Cetrimonium Bromide (CTAB)**	**Potassium Dichromate**	**Hydrated Chromium Sulfate**
Appearance	white powder	white powder	red-orange crystalline solid	violet crystalline solid
Chemical formula	$C_{12}H_{25}SO_4Na$	$C_{13}H_{33}N(CH_3)_3Br$	$K_2Cr_2O_7$	$Cr_2(SO_4)_3{\cdot}18H_2O$
Molecular weight (g/mol)	288.38	364.45	294.18	700.33
Supplier	THOMAS BAKER (Mumbai, India)	AVONCHEM LIMITED (UK)	BDH (England)	CDH (India)

Table 2. Equipment Used in this Research

Devices	**Specifications**
Microbubble diffuser (MBD)	D = 10 cm, W = 909 g, Flow = 1.6L/min, P = 3.4 bar, Point Four TM diffuser, Aqua cave Company, Canada
Microbubble Flotation Column (MBFC)	Made of acrylic material with dimensions (I.D = 12 cm, O.D = 13 cm and 200 cm in height), has 11 ports
Air compressor (AC)	Ingco industrial, 220-240V, ~50Hz, AC25508, P=0-8 bar, capacity 50 liters, China
Flow Meter (FM)	(YYZX, instrument company), range (0 - 1.0 L/min)
pH Meter	(HANNA, instrument, pHep HI98103), range (0.00 - 14.00)
Pressure Gauge (PG)	(BS EN 837 offered by Brannan, UK), range (0 – 5 bar)
Pump (P)	(TYP 2600 DA, diaphragm booster pump, china), flow rate is 0.75 LPM
Tanks (T)	Two tanks 20-liter plastic
Taps and Valves (T & V)	Qua liquid inlet valve only 1/4" di vertor ball valve on/off (11 taps, China)

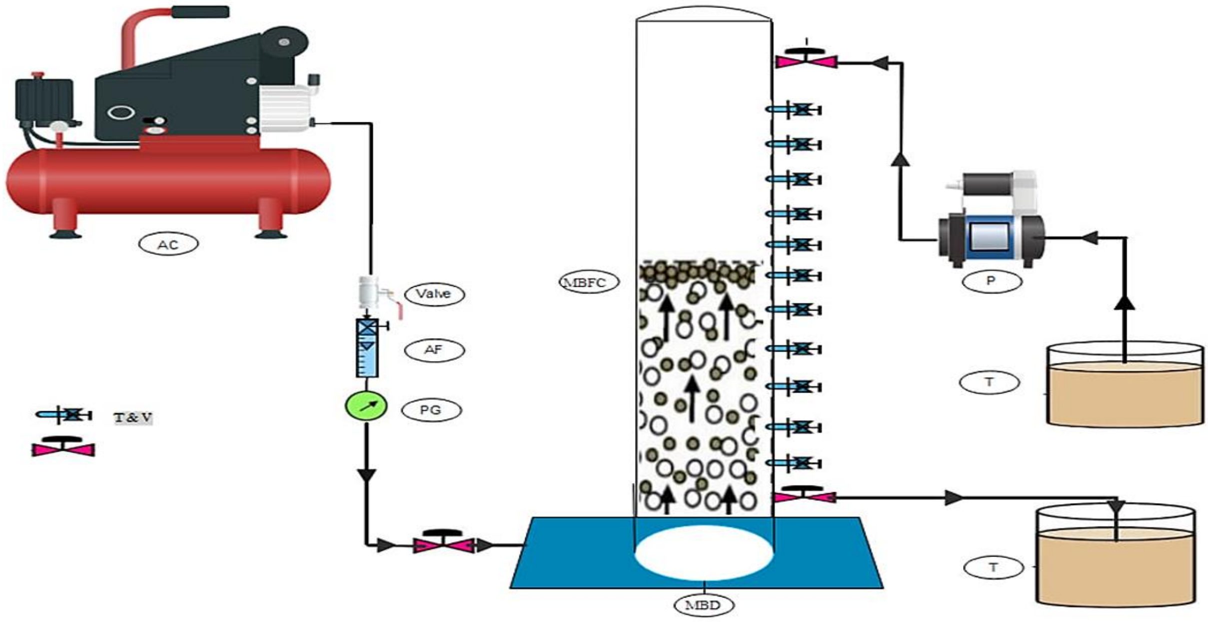


Fig. 1. Schematic Diagram of the Experimental Setup [Define the abbreviations seen in the figure]

2.3. Procedure

A stock solution of 1000 ppm contaminant was prepared by dissolving 1.0 g of chromium-containing contaminant in 1000 ml distilled water and mixing well until complete homogeneity. Further concentrations were prepared daily from the stock solution by dilution. The concentrations 10, 20, 30, and 40 mg/L, were obtained using the equation below:

$$V_1 \times C_1 = V_2 \times C_2 \tag{1}$$

Where: C_1= Concentration of the stock solution (mg/L). C_2= Working concentration to the polluter solution (mg/L). V_1= volume of stock solution (ml). V_2= Needed volume to the polluter solution (ml).

In all experiments, SDS (sodium dodecyl sulfate) was used as an anionic (negatively charged) surfactant. The effect of SDS was compared with CTAB (Cetyltrimethylammonium bromide) a cationic (positively charged) surfactant. Both surfactants have a hydrophilic head (carrying the charge) and a hydrophobic tail. When pumping air bubbles in the surfactant solution, the surfactant molecules attach to the surface of the bubble. The hydrophobic tail is mostly inside the bubble while the hydrophilic head remains in the aqueous solution. As an anionic surfactant, SDS contains a negatively charged head group (sulfate) and a long hydrophobic tail (dodecyl). In the presence of positively charged cadmium metal particles, these are attracted to the negatively charged SDS molecules located on the surface of the bubble. The buoyancy force lifts the bubble to the surface of the solution resulting in the removal of the cadmium ions. The micrometer size of the air bubbles provides a higher surface area compared to normal bubbles, leading to an increased removal rate.

All experiments were conducted at a room temperature. Samples were taken from the solution every 5 min after starting the experiment and their concentrations were measured using a flame atomic absorption spectrophotometer (, Model AA-7000, Shimadzu, Japan). The initial pH of the solution was adjusted using sodium hydroxide (0.4 N NaOH) and hydrochloric acid (0.1 N HCl). The removal efficiency was calculated according to the equation:

$$\text{Removal Efficiency, \%} = \frac{Co - Ct}{Co} \times 100 \tag{2}$$

Where Co and Ct were the initial and current concentrations of metal ions in mg/L.

3- Results and Discussion

3.1. Effect of pH

pH is an important factor in the flotation process. A range of pH values (3, 5, 7, 9, and 11) were studied to see how the removal efficiency of chromium ions in a MBF system is influenced by the pH of the solution. The other factors, including (concentration of SDS 15 mg/L, flow rate of air 0.5 L/min, chromium ion concentration 30 mg/L, 2nd port 30 cm) were kept constant. This effect of pH is illustrated in Fig. 2. The removal efficiency went up during the initial 10 minutes, then increased slower until 25 minutes when the final value was reached. The maximum removal efficiency of 94% was achieved at pH 7. The efficiency decreased for pH values below 7 due to the competition between H^+ and chromium ions for SDS. Furthermore, in basic media, heavy metals can form complexes with hydroxide ions. These complexes can be less reactive or less accessible to the removal medium, thus reducing the efficiency of the removal process [27, 33, 34].

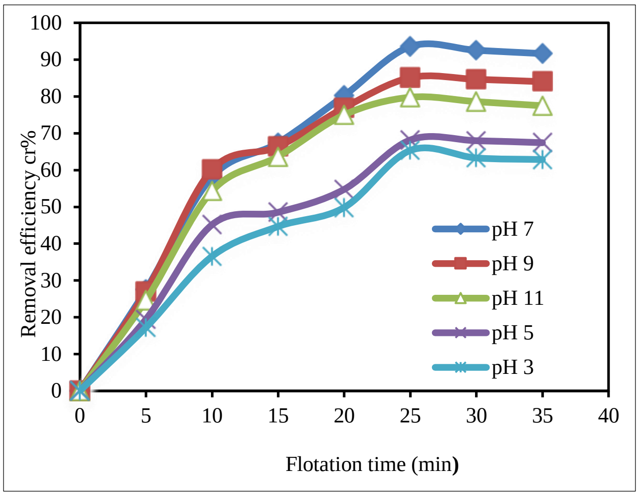


Fig. 2. Removal Efficiency vs. Time at Different pH Values

3.2. Effect of Initial Concentration of Chromium Ions

Four different initial concentrations of chromium ions were examined (10, 20, 30 and 40 mg/L). The other factors were kept constant (flow rate of air 0.5 L/min, pH 7, C_{SDS} 15 mg/L and Sp 30 cm). The findings, illustrated in Fig. 3, show that when the concentration of chromium ions increased from 10 to 30 mg/L, the removal efficiency at 20 min also increased: 65%, 78%, and 94%. Extending the flotation time from 20 to 30 min did not affect removal efficiency significantly. At the higher concentration of 40 mg/L, the removal efficiency decreased down to 84%. This is consistent with previous findings [34, 35].

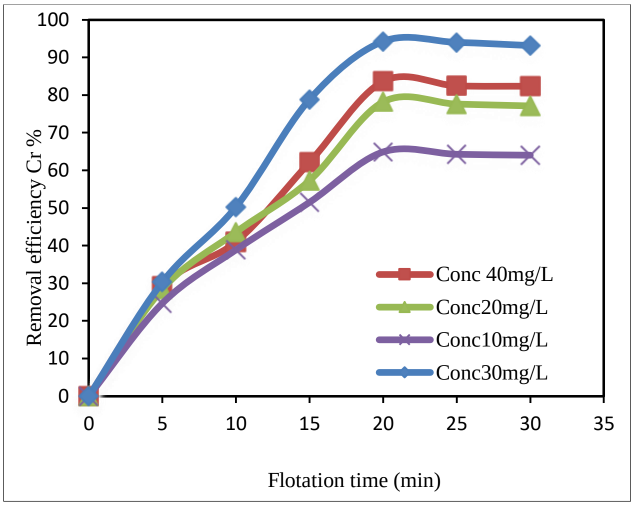


Fig. 3. Removal Efficiency vs. Flotation Time for Different Initial Concentration of Chromium Ions

3.3. Effect of Surfactant Type

Sodium dodecyl sulfate (SDS), is a negatively charged in solution while cetyltrimethyl ammonium bromide (CTAB) is positively charged. The performance of the two surfactants is compared in Fig. 4. Other parameters were kept fixed (flow rate of air 0.5 L/min, chromium ion concentration 30 mg/L, pH 7, and Sp 30 cm, surfactant concentration 15 mg/L). The removal efficiency of chromium ions reached 95% and 58% at 20 minutes when using SDS and CTAB, respectively. Clearly the negatively charged surfactant SDS is more efficient than the positively charged surfactant CTAB [34, 37].

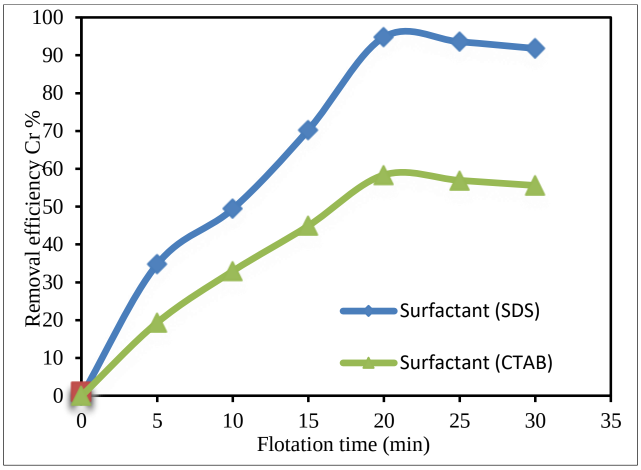


Fig. 4. The Removal Efficiency Values for Different Types of Surfactants

3.4. Effect of Surfactant Concentration

Different SDS surfactant concentrations (5, 10, 15 and 20 mg/L) were used, while other parameters were kept fixed (flow rate of air 0.5 L/min, chromium ion concentration 30 mg/L, pH 7, and Sp 30 cm). The removal efficiency of chromium ions reached a maximum of 95% after 20 minutes for SDS 15 mg/L as shown in Fig. 5. However, when the SDS concentration was increased to 20 mg/L, the removal efficiency of chromium ions decreased to 63%. This is due to the competition for bubble surface locations between the metal-collector complex and free collector ions. Bubbles attach to the free collector ions more than the metal-collector complexes, and the removal efficiency decreases. The excess of collector can lead to the formation of micelles, which may carry some collector in the effluent [27]. The chromium ions adsorb on the micelles, which themselves do not float due to their hydrophilic surfaces. Thus flotation is affected when the surfactant concentration is increased beyond the critical micelle concentration [37, 38].

3.5. Effect of Flow Rate of Gas (Air)

The effect of this parameter was studied using different gas flow rates (0.1, 0.2, 0.3, and 0.5 L/min), while other parameters (chromium ion concentration 30 mg/L, C_{SDS} 15 mg/L, pH 7, and Sp 30 cm) were kept fixed. The effectiveness of the chromium ion removal in the microbubble flotation column is shown in Fig. 6.

It can be noticed when the gas (air) flow rate reaches 0.5 L/min after 20 minutes of flotation, removal efficiency gradually rises to the peak value of 94%. When reducing the flow rate to a value of 0.3 L/min, removal is lesser (90%). Increased fluid activity (stress) at the bottom part, early bubble detachment, bubble coalescence, and (mainly) bubble breakup are all caused by an increased gas flow rate. With the low gas flow rate, higher retention times were needed [38]. The efficiency of removal at the optimum flow rate of 0.5 L/min was 94% compared with low flow rate 0.1 L/min where removal was 75%. The size and number of the bubbles grow in direct proportion to the flow rate. An increased gas flow rate leads to an increase in the amount of surface area accessible for metal collector adsorption [27].

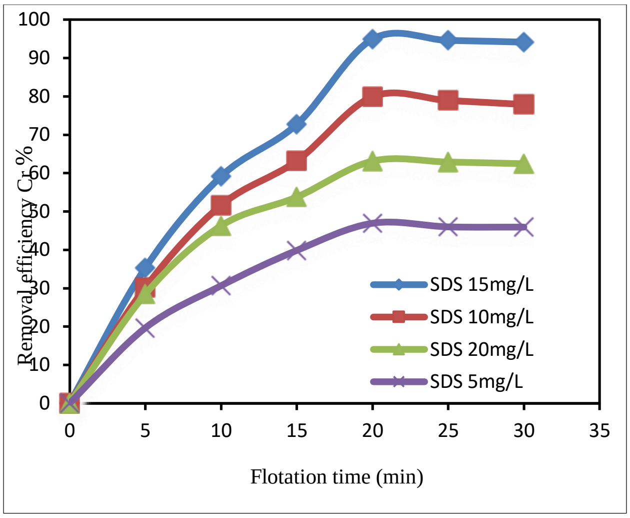


Fig. 5. The Removal Efficiency Values for Different (SDS) Surfactant Concentration

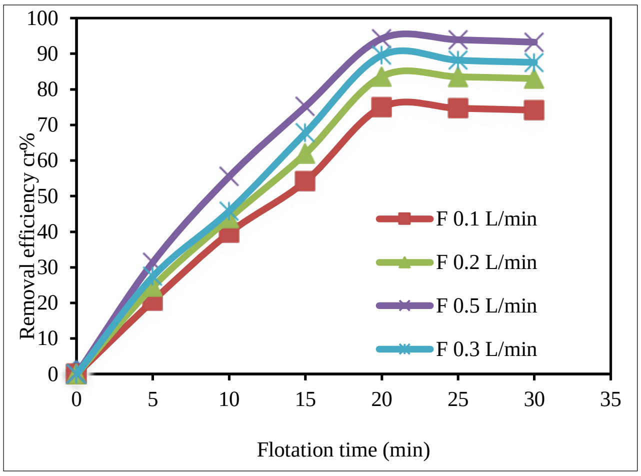


Fig. 6. The Removal Efficiency Values for Different Flow Rate

3.6. Effect of Sampling Port Location

Several ports were installed on the flotation column, with the first port near the diffuser from the bottom of the float shaft to the top. The ports were spaced 15 cm apart (see Fig. 1), and three ports (Sp1 at 30 cm, Sp2 at 60 cm, and Sp3 at 90 cm) were chosen to evaluate the gradient in removal efficiency. Other parameters remained constant (chromium ion concentration 30 mg/L, C_{SDS} at 15 mg/L, pH 7, and flow rate of air 0.5 L/min). After 20 minutes, the removal efficiency stabilized as follows: Sp1 at 93%, Sp2 at 83%, and Sp3 at 72% respectively – Fig. 7. It can be concluded that the first port, located near the diffuser at a height of 30 cm, is the optimal sampling port. One contributing factor to this finding is the decrease in internal pressure and density of bubbles as they move away from the diffuser. Consequently, the accessible bubble surface area decreases, resulting in lower flotation efficiency [39].

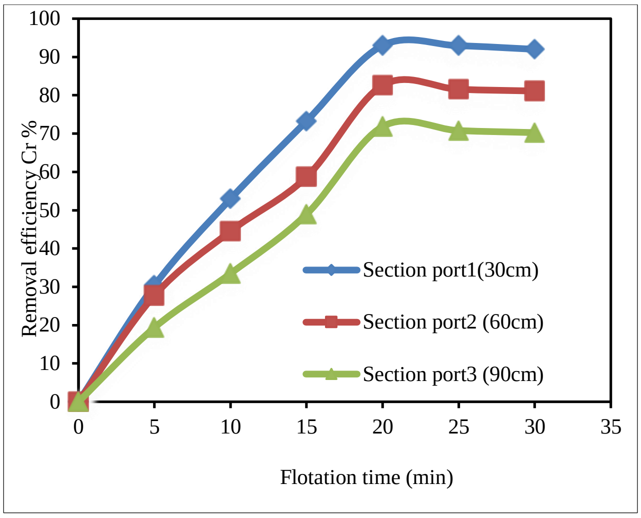


Fig. 7. Removal Efficiency vs. Flotation Time for Different Sampling Port Location

3.7. Effect of Microbubbles

To assess the advantages of using microbubble technology into the pollutant flotation column, several tests were carried out to remove chromium ions with and without the MB diffuser (i.e., with conventional bubbles by a regular diffuser), and its effect on the efficiency of flotation, while other parameters were kept fixed (flow rate of air 0.5 L/min, chromium ion concentration 30 mg/L, C_{SDS} 15 mg/L, pH 7 and Sp 30 cm). The result is shown in Fig. 8.

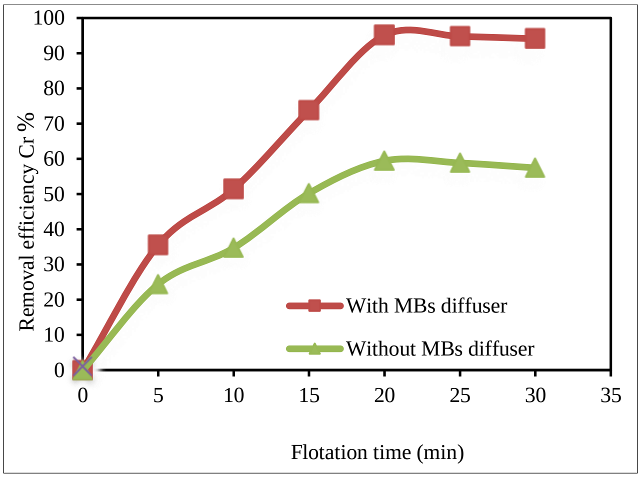


Fig. 8. The Removal Efficiency Values at MBs Diffuser and Without

After 20 minutes of flotation time, the removal efficiency of microbubble air flotation was 95%. This is significantly higher than the removal efficiency of bubbles by a regular diffuser, which was 59%. The better performance of microbubbles in comparison to normal bubbles matches previous results [8, 25].

3.8. Flotation Kinetics

Flotation kinetics describes how the concentration of the floated material changes over time. This is beneficial for understanding the process' mechanism and may be applied as a predictive tool for implementing flotation technology [35]. The rate of flotation is equivalent to the rate at which the concentration of floatable material in the cell alters.

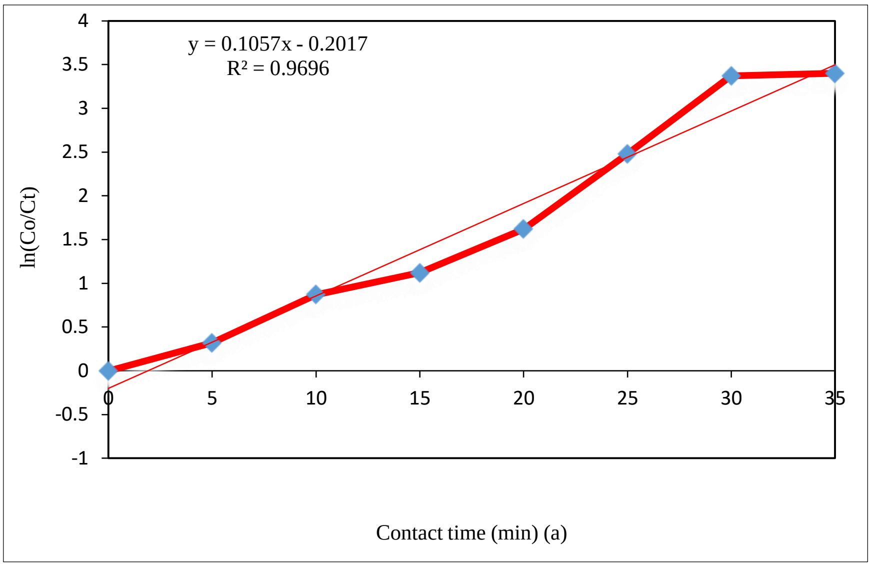
The equations used to estimate the kinetics of flotation are:

$$Ct = C_O \exp(-k_1 t) \text{ for first order.} \qquad (3)$$

$$Ct = C_O / (1 + C_O k_2 t) \text{ for second order.} \qquad (4)$$

Where C_O (mg/L) is the pollutant's initial concentration at time 0, Ct (mg/L) is the contaminant concentration at time t, and the rate constants for the kinetics of the first and second orders, respectively, are k_1 [1/min] and k_2 [l/mg/min]. To determine the values of the rate constants for each order of reactions, and the values of the rate constant, the optimal conditions for the $C_{Cr\ (VI)}$ removal experiments (flow rate of air 0.5 L/min, pH 7, chromium ion concentration 30 mg/L, C_{SDS} 15 mg/L and Sp 30 cm), the data given in Fig. 9 (a; b), respectively, were obtained by applying the findings of this experiment to the above equations. Table 3. Contains the information for the rate constants and correlation coefficients.

The information in Table 3. indicates that the first-order kinetics model fits better the results. We note that a higher rate constant shows that the species floats quicker [27, 34]. By comparing the first-order equation with the second-order equation, the larger correlation coefficient obtained for the first-order equation (R^2) is greater and consistent with previous findings [40].



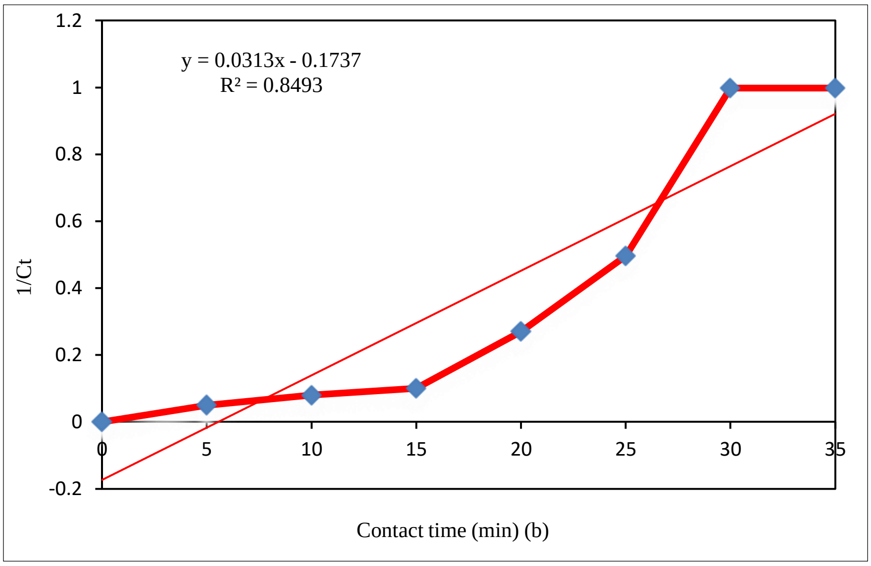


Fig. 9. Time Dependency of the Estimated and Experimental Values of $C_{Cr(VI)}$, (a)1st order, (b) 2nd order

Table 3. Rate Constant and Correlation Coefficient for 1st and 2nd order Reactions

N	**Rate Constant and Correlation Coefficient for Chromium Ions**		
	Units	k_n	R^2
1	1/min	0.1057	0.9696
2	l/mg.min	0.0313	0.8493

4- Conclusion

The technique of flotation with air micro bubbles was used to remove chromium ions from simulated wastewater. A removal rate of 95% was achieved within 30 minutes at pH of 7, a flow rate of air 0.5 L/min, an SDS concentration of 15 mg/L, and a chromium ion concentration of 30 mg/L using the sampling port at 30 cm. From these results, we conclude that the pH range of 6.5 - 7.5 is the most appropriate and that increasing the percentage of chromium ions and the flow rate of gas (air) enhances the removal rate at a 1:2 ratio of surfactant to chromium ions. Additionally, it was observed that anionic surfactants are more suitable for these conditions than cationic ones. The kinetic of flotation follows a first-order pattern.

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تعويم أيونات الكروم من مياه الصرف بواسطة فقاعات الهواء الدقيقة

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الخلاصة

استخدمت هذه الدراسة تقنية تعويم الهواء بالفقاعات الدقيقة لإزالة أيونات الكروم من مياه الصرف الصحي المحاكاة. تم فحص المعلمات التجريبية لتحليل عملية التعويم وتحديد كفاءة الإزالة. تضمنت هذه المعلمات موقع منفذ أخذ العينات (٣٠، ٦٠، ٩٠ سم)، نوع الفاعل بالسطح (أنيوني، SDS، أو كاتيوني، CTAB وتركيزه (٥، ١٠، ١٥، ٢٠ مجم / لتر)، الرقم الهيدروجيني للمحلول الأولي (٣، ٥، ٧، ٩، ١١)، تركيز الملوث الأولي (١٠، ٢٠، ٣٠، ٤٠ مجم / لتر)، معدل تدفق الغاز (0.1−٥.٠٠ لتر / دقيقة)، ووقت الاتصال (٥، ١٠، ٢٠، ٢٥، ٣٠، ٣٥ دقيقة). أظهرت النتائج التجريبية تحقيق أعلى كفاءة إزالة (٩٥,١١٪) في ٣٠ دقيقة مع درجة حموضة ٧، ومعدل تدفق ٠,٥٠ لتر / دقيقة، وتركيز خافض للتوتر السطحي SDS 15 مجم / لتر، وتركيز ملوث ٣٠ مجم. / لتر عند ارتفاع منفذ أخذ العينات ٣٠ سم. علاوة على ذلك، عند مقارنة استخدام الفقاعات الدقيقة بالفقاعات العادية، أظهرت النتائج تحسنًا بنسبة ٥٦,٥٪ في كفاءة الإزالة عند استخدام الفقاعات الدقيقة. بالإضافة إلى ذلك، حددت الدراسة أن الترتيب الحركي للطفو، والذي كان تقريبًا من الدرجة الأولى.

الكلمات الدالة: عملية التعويم، الفقاعات الدقيقة، المعادن الثقيلة، أيونات الكروم، المادة الخافضة للتوتر السطحي.