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Micro-Bubble Flotation for Removing Cadmium Ions from Aqueous Solution: Artificial Neural Network Modeling and Kinetic of Flotation

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Abstract

In this work, microbubble dispersed air flotation technique was applied for cadmium ions removal from wastewater aqueous solution. Experiments parameters such as pH (3, 4, 5, and 6), initial Cd(II) ions concentration (40, 80, and 120 mg/l) contact time(2, 5, 10 , 15, and 20min), and surfactant (10, 20and 40mg/l) were studied in order to optimize the best conditions .The experimental results indicate that microbubbles were quite effective in removing cadmium ions and the anionic surfactant SDS was found to be more efficient than cationic CTAB in flotation process. 92.3% maximum removal efficiency achieved through 15min at pH 5, SDS surfactant concentration 20mg/l, flow rate250 cm^3/min and at 40mg/l Cd(II) ions initial concentration. The removal efficiency of cadmium ion was predicted through 11 neurons hidden layer, with a correlation coefficient of 0.9997 between ANN outputs and the experimental data and through sensitivity analysis, pH was found to be most significant parameter (25.13 %).The kinetic flotation order for cadmium ions almost first order and the removal rate constant (k) increases with decreasing the initial metal concentration.

Keywords: cadmium ions, flotation, microbubbles, ANN model, kinetic flotation rate.

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1- Introduction

The presence of heavy metals in water is a universal environmental concern from landfills and industrial. [1]**.** Among various organic and inorganic pollutants, heavy metal ions are very toxic and carcinogenic in nature. Heavy metals enter into the food chain through the disposal of wastes into water bodies **[2]** and possibly cause severe health problems, when their concentration exceeds their permissible, also they are very difficult to eliminate naturally from the environment as it was non-biodegradable pollutants [3].

Cadmium is a highly toxic element and its exposure may results which called “cadmium blues” results from respiratory damage after few weeks. Highly exposures causes tracheo-bronchitis, pneumonitis, diarrhea, stomach pains and severe vomiting, bone fracture, reproductive failure and possibly infertility. Permissible limits of Cd (II) ions in drinking water is 0.005 mg/L and above this level may cause serious infection [4].

Many methods are available to remove heavy metals concentration from water, such as precipitation, flotation, ion exchange, solvent extraction, adsorption, and cementation onto iron, membrane processing and electrolytic methods [5].

The selection of a particular treatment depends on a number of factors such as waste type, contaminant concentration, level of cleanup required and economics [6]**.** Flotation is one of the most effective separation techniques from the above processes .Nowadays, many industrial applications use microbubbles of air and oxygen in water treatment application due to its superior efficiency, compared with conventional methods [7]**, [8].**

Flotation processes utilize microbubbles are very effectively for removing low-density particulate matter from water [9] , which act as carriers for fine particles, which are lifted up from the bottom of the column.

Different flotation techniques processors available depending on the microbubble generation process, like: dissolved-air flotation, dispersed-air flotation or electro-flotation [10], [11].

Traditional flotation assisted with microbubbles (30–100 μm) was used in the recovery of fine mineral particles (<13 μm), as well as separation to remove pollutants. The microbubble has advantage of improving the separation efficiencies as compared with coarse bubbles, particularly those for the ultrafine (<5 μm) ore particles. By decreasing the bubble size distribution (through the injection of small bubbles), the bubble surface flux will be increased and the fines capture.

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Dissolved air flotation with microbubbles, treating water, wastewater and domestic sewage is known for a number of years and is now gradually entering in the mining environmental area **[12].** Though there are several studies on the removal of heavy metal by dispersed air flotation such as those applied by**[13][14][15],** more of them used frother in order to reduce the size of the air bubbles in a flotation cell to present sufficient surface area for collection.

Artificial Neural Network are powerful techniques used in modeling complex systems that seeks to simulate human brain behavior by treatment of data on the basis of trial and error. ANN has been identified as tool to determine and optimize complicated nonlinear relationships between parameters **[16]**.

Numerous studies have been conducted in which ANNs were used in different wastewater processes such as flotation **[17]**, advanced oxidation process**[18]** power generation by microbial fuel cell**[19],** adsorption**[20]** , and sorptive flotation **[21]**. The scope of this study was to investigate the effect of micro- bubbles in the removal efficiency of Cd(II) ions from aqueous solution at different parameters , ionic strength , flotation kinetic and use the artificial neural network (ANN) to describe this behavior.

2- Flotation Kinetics

The variation of floated concentration with time will be studied by flotation kinetics, which are useful in the elucidation of the mechanism of the process, and serve as predictive tools in the implementation of flotation technology [**22**]

The flotation rate is equal to the rate of change of concentration of floatable material in the cell, is

$$\frac{dc}{dt} = -kc^n \qquad (1)$$

Where, k is the flotation rate constant and the value of (n) denotes the order of the equation.
By integration Eq. (1) with n= 1 (first order) gives:

$$C = C_o\, e^{-kt} \qquad (2)$$

Where C_o= initial concentration, and C = the final concentration of valuable material remaining at time t. Taking the ln of Eq.(2):

$$\ln \frac{C}{C_o} = -kt \qquad (3)$$

A high rate constant shows that certain species floats quickly while a low rate constant indicates slow flotation**[23]**.

3- Experimental Work

The flotation experiments were carried out in a glass tank with 33cm in length, 19cm in height and 8 cm in width as shown in Fig. **1** The tank was operated at batch mode for the liquid phase, and continuous flow with respect to air which was provided by a compressor (type: SAMTONG) in which it was compressed from1 bar to up to 7 bar and then passed into the microbubble generator (MBG) which consists of a ceramic micro-porous diffuser (Point Four TM diffuser) (Riverforest Corporation, USA, AS MK-III). The dissolution of gas into the solution was achieved inside the MBG by applying a high pressure.

The microbubbles (mean diameter= 100µm) were generated by the release of pressure, and then entered to the tank through a pre-calibrated rotameter (0-2.5cm^3/min). The desired concentration of cadmium ions was prepared by dissolving the calculated amount of $Cd(NO_3)_2.4H_2O$ in distilled water. The main physicochemical properties of the cadmium ions was shown in Table **1** .Cd(II) ions feed gently from the top of the tank. Simultaneously, the tank was pressurized so as to prevent liquid to spew from the holes**[24],** then samples were drawn through port at the center of the surface (19cm high and 8 cm) of the tank at different periods (2, 5, 10, 15 and 20 minutes) . Firstly, 2ml of solution was drained from the port before withdrawing each sample in order to reduce the entrainment of air bubbles. The concentration of the Cd(II) ions was determined by using flame atomic absorption spectrophotometer (AAS; SHIMADZU, Model: 7200, Japan).

The initial pH of the working solutions was adjusted by the addition of 1 mol/l NaOH or HCl using a pH meter (WTW, inoLab 720, Germany) and all experiments were carried out at room temperature. HCL was used to clean the tank between experiments and then washed twice with distilled water.

Table 1. Main physicochemical properties of cadmium ion

Property	Cadmium nitrate tetrahydrate
Appearance	White colorless crystals
Chemical formula	$Cd(NO_3)_2.4H_2O$
Molecular weight (g/mole)	308.7
Atomic weight (g/mole)	112.7
Density (g/cm^3)	2.45
Solubility in water g/100 ml at 20 °C	136
Hydrated ionic radius, Å	4.26
Electronegativity	0.95
Company	BDH England

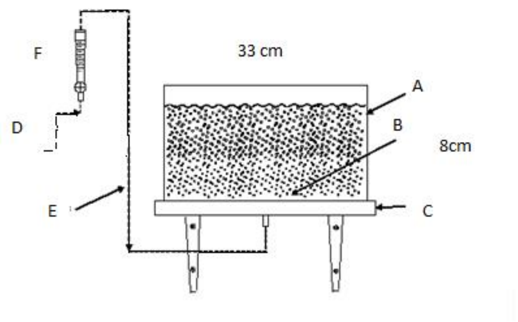

Fig. 1. Experimental flotation apparatus, with(A= operating tank ; B= microbubble diffuser ; C= Diffuser bed ; D=Air bubble supply ; E = Pipe ; F=Flow meter

4- Results and Discussion

4.1. ANN Model Develop

ANN model was developed using Levenberg–Marquardt backpropagation (LMA) training algorithm for correlating the removal efficiency of Cd(II) ions from aqueous solution.

This algorithm was determent using Matlab program version 78.2.0.701 (R2013b). Randomly divide the subset of 100 experimental data into; 60 %, 20% and 20% as training set, validation set, and testing set, respectively.

Fig. **2** indicate the best topology for ANN for Cd(II) ions removal and the variation of parameters was calculated at (6:18:1) depending on the RMSE of the prediction and training sets , which was the highest at hidden neurons no. 2 then decreased significantly to reach the minimum values 1.062 at 11 hidden neurons as can be seen in Fig. **3**, the dependence between RMSE and the neuron number for the LMA algorithm.

Fig. **4** shows that the training was disabled after epoch 15 when the best validation performance was 0.77385 and the best regression for training, validation and testing for the Levenberg-Marquardt algorithm was set in Fig. **5**, with correlation coefficients of 0.99725, 0.99393, 0.95795 and 0.99357for training, validation, testing and all data.

The correlation coefficient between predicted and experimental data approves that the ANN model can efficiently simulate the removal efficiency with correlation coefficient 0.9997as can be seen in Fig. **6**.

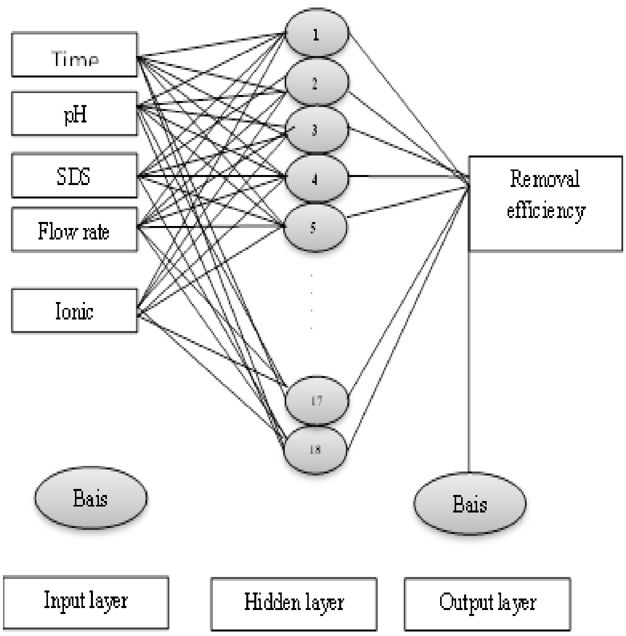

Fig. 2. ANN best architecture model

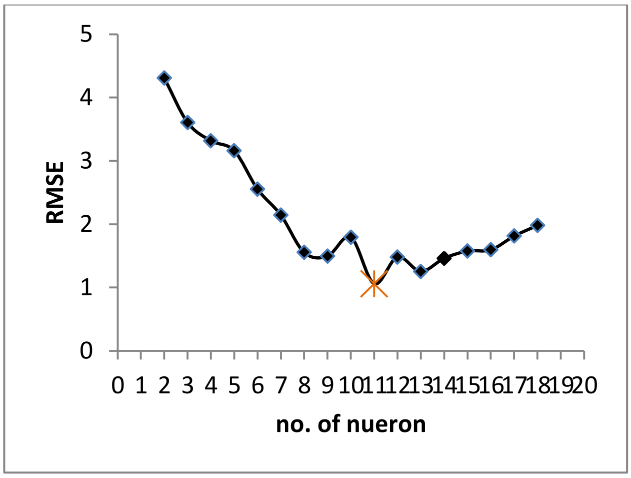

Fig. 3. RMSE at different hidden neurons no

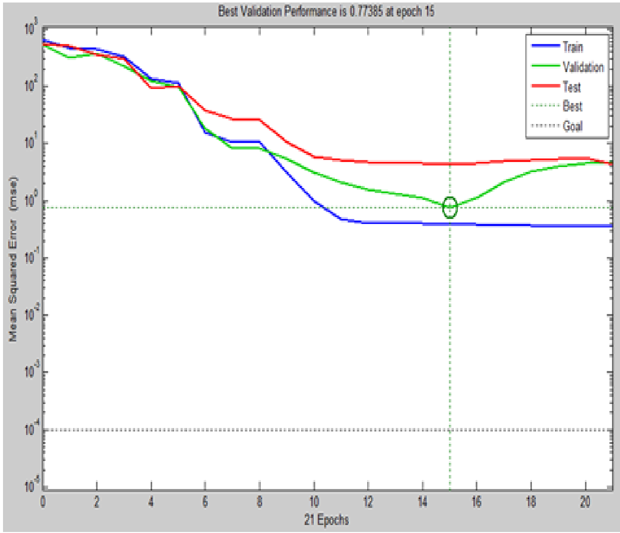

Fig. 4. Mean square errors training, validation, and testing for the Levenberg–Marquardt algorithm

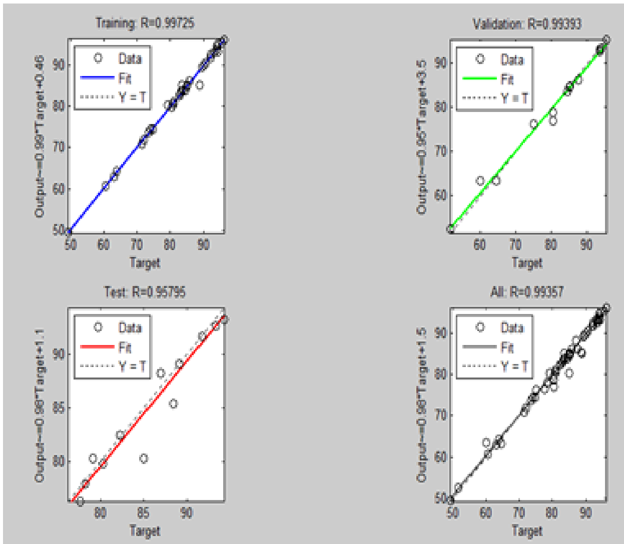

Fig. 5. regression analysis for the Levenberg-Marquardt algorithm

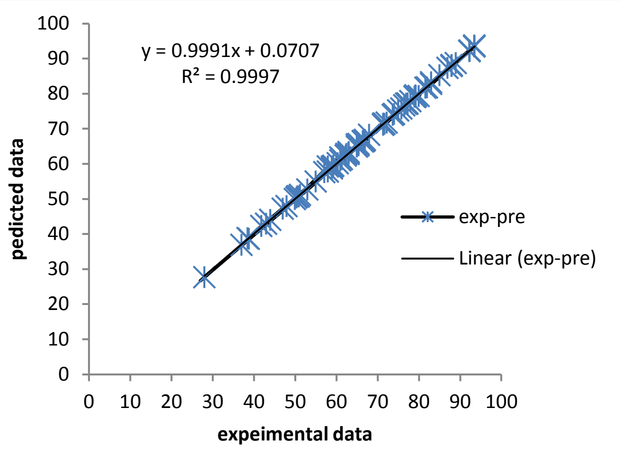

Fig. 6. Relationship between predicted and experimental values of the output

4.2. Effect of Surfactant Type

Fig. **7** presents a comparison between two types of surfactants ;sodium dodecylsulfate surfactant (SDS) as anion surfactant and Cetyltrimethyl ammonium bromides (CTAB) as cation surfactant, at the same concentration (10 mg/L), while all other parameters were kept constant pH 6.8, air flow rate 2.5 ml/min and Cd(II) ion concentration= 40 mg/l) , It can be seen that the removal of Cd(II) ions reached 85% and 60% by using SDS and CTAB surfactant, respectively. That's meant the anionic surfactant (SDS) is more efficient than the cationic surfactant (CTAB) and will be used in the next experiments.

The desired collector type depends upon pH value of a solution. Generally, an anionic collector is required for pH (1-7) due to that metal ion is on cationic shapes. On higher pH, cationic collectors could be required when metal ion was existing as an anion **[25][26].**

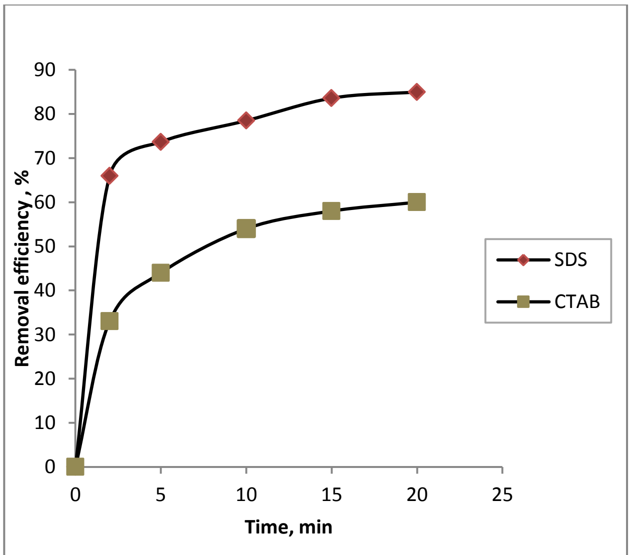

Fig. 7. Influence of surfactant type on the removal of Cd(II) ions

4.3. Influence of pH

In order to investigate the influence of solution pH on the separation efficiency of cadmium ions towards the collector used in microbubble dispersed air flotation system, different pH values (3,4,5and 6) were tested while keeping other parameters constant (SDS concentration= 10mg/l, air flow rate 250 cm^3/min and Cd(II) ion concentration= 40 mg/l), Fig. **8** shows that the removal efficiency increased suddenly at the first 2 minute, then it began to increased slowly with time due to decreasing SDS concentration with time. The maximum removal efficiency achieved at pH5 was (79.2%), while it decreased for pH less than 5 because the competition between Cd(II) ions and H ions for collector, This results agree with the finding of[**13**][**22**] also a good agreement between the experimental and predicted data can be seen from the figure. Table **2** presents the removal rate constant (k) at various pH , for this table it can be noticed that the removal rate constant increased with measuring pH value and it reached 4.248*10^$^{-12}$ min at pH6

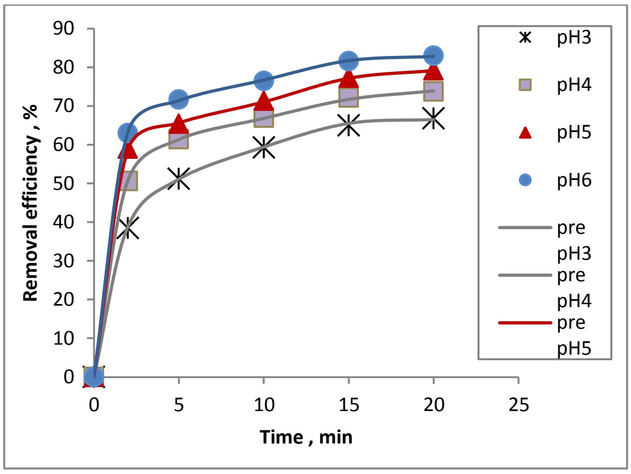

Fig. 8. Removal efficiency of Cd (II) ions as a function of pH

Table 2. Kinetic flotation at various pH

	Effect of pH		
	pH=4	pH=5	pH=6
$k\times10^2$ (min^{-1})	3.378	3.834	4.248

4.4. Influence of SDS Surfactant Concentration

Different sodium dodecylsulfate surfactant (SDS) concentrations (0, 10, 20 and 40mg/l) were used while other parameters were kept constant (pH 5, flow rate =250cm^3/min, Cd(II) ion concentration = 40mg/l).

Fig. **9** shows that the removal of Cd (II) ion reached (92.3%) at 15 min at SDS concentration of was 20 mg/l and by increasing SDS concentration to 40 mg/l the removal efficiency of Cd(II) ion was deceased to (60.2%) due to competition between the metal-collector complex and free collector ions for bubble surface sites at excessive collector amount as well as micelle formation , the potential toxicity of residuals amounts of collector in the effluent and cost [**23**], also this figure confirms that the ANN model could be effectively predicting the experimental results.

To theoretically examine, if the amount of SDS represent the optimum value for highly removal efficiency, ANN model was used at different concentrations of SDS was varied around the best value (15, 18, 20, 22, 25, 28, 30 and 35 mg/L) as shown in Fig. **10**. It can be seen from this figure that there is a good agreement between the experimental data and the predicted data, in addition through ANN model, the optimum value of SDS concentration was 18 mg/l.

Table **3** shows that, the removal rate constants increased with increasing SDS concentration. The result is in agreement with the finding of **[26].**

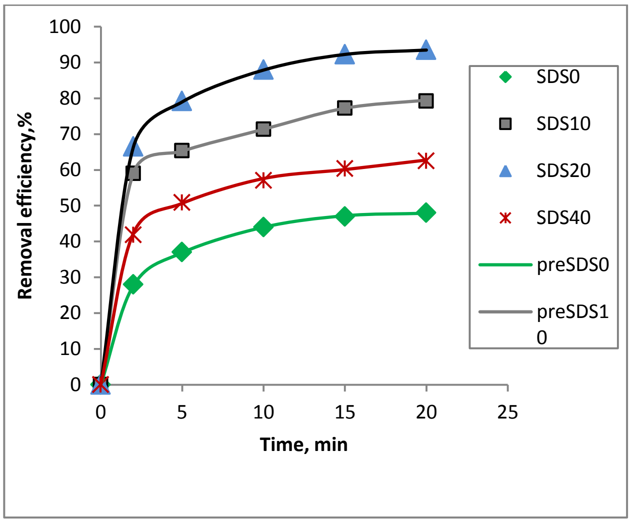

Fig. 9. Influence of SDS on the Cd(II) ions removal efficiency

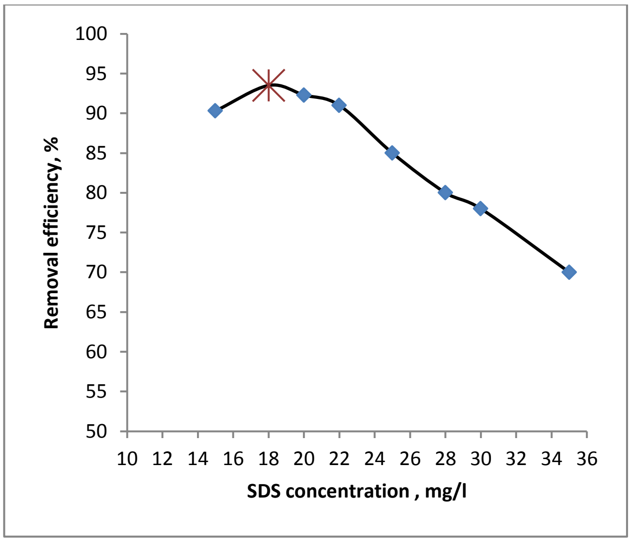

Fig. 10. Removal efficiency predicted by ANN model at different SDS concentration

Table 3. Rate constant at various SDS concentrations

	SDS concentration		
	SDS10	SDS20	SDS40
k×10^2 (min^{-1})	1.883	4.505	1.155

4.5. Influence of Flow rate

Different air flow rate (100, 200 and 250cm^3/min) were tested to examine their effect on the removal efficiency of Cd(II) ion in the microbubble dispersed air flotation column was investigated.

The other parameters were remained constants (pH=5, cadmium conc. = 40 mg/ l and SDS = 10 mg/l), Fig. **11** indicated that the removal efficiency was highly affected by the gas flow rate, the large number of small bubbles leads to increase the surface area available for metal-collector adsorption.

By increasing gas flowrate, the removal efficiency increased. Increasing gas flow rate causes early bubble detachment, large fluid activities (stress) at the bottom section and bubble coalescence and (mostly) break up **[27]** also, these results are in good agreement with the proposed ANN model.

To theoretically examine, if this value represents the best flow rate for maximum removal efficiency, Fig. **12** presents the influence of different air flow rate around the best value on the removal rate of Cd(II) ions (2, 2.2, 2.5 , 2.7 and 3 cm^3/min) it can be noticed from this figure that 2.7 cm^3/min was the best predicted value.

Table **4** presents the removal rate constants (k) at different air flow rate, which shows that the(k) increased by gas flow rate increased.

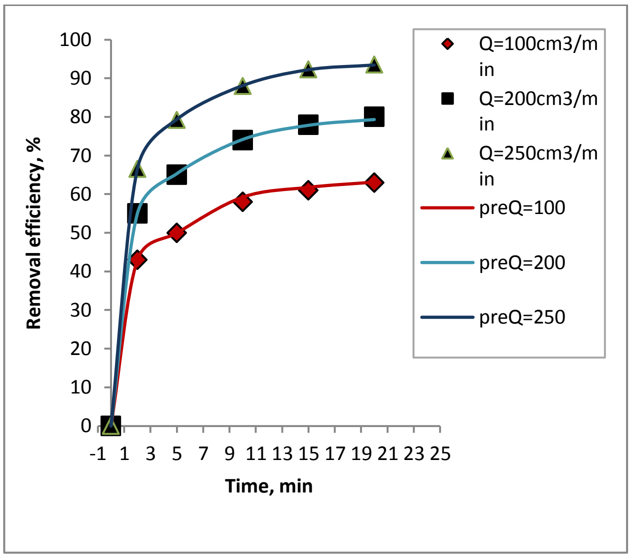

Fig. 11. Removal efficiency of Cd(II) ions at different air flow rate

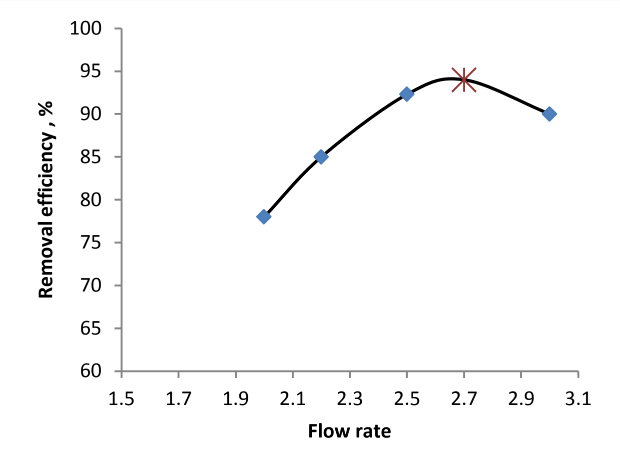

Fig. 12. Predicted removal efficiency at different air flow rate

Table 4. Flotation kinetic at different air flow rates

	Air flow rate(cm^3/min)		
	Q=100	Q=200	Q=250
$k\times10^2$ (min^{-1})	2.375	4.456	9.156

4.6. Influence of Initial Concentration of Cd(II) Ions

Three different initial Cd(II) ions concentrations were tested in this study (40 , 80 and 120 mg/l) while keeping other parameters constant (pH=5, SDS = 10 mg/l and flow rate = 250 cm3/min) and their results presented in Fig. **13**, which indicating that the removal rate decreased with increasing Cd(II) ions concentration from 40mg/l to 120mg/l , owing to the fact that the floatability of Cd(II) ions reduced at their higher concentrations, the removal efficiency decreased from (92.3%) to (63%) for Cd(II) ions by increasing the initial metal ions concentration from 40 to 120 mg/L respectively at 15min.

This is in agreement with the finding of **[28][22],** they concluded that by increasing metal ion concentration ,more collector are required at low pH for the same percent removal. So, the removal efficiency decreased at higher Cd(II) ions concentrations, also this figure confirms that the neural network model could be effectively predicting the experimental results.

To theoretically examine this value for maximum removal efficiency, Fig. **14** present the influence of different initial metal concentration around the optimum value (30, 35, 40, 45, 50, and 55) on the removal rate of Cd(II) ions using ANN model , the ANN model reveals 40 mg/l concentration was the best predicted value as it shows an agreement between predicted and results.

A concentration of (40 mg/l) was predicted to be the best value. Table **5** shows higher removal of Cd(II) ions occurred at the lowest initial concentration.

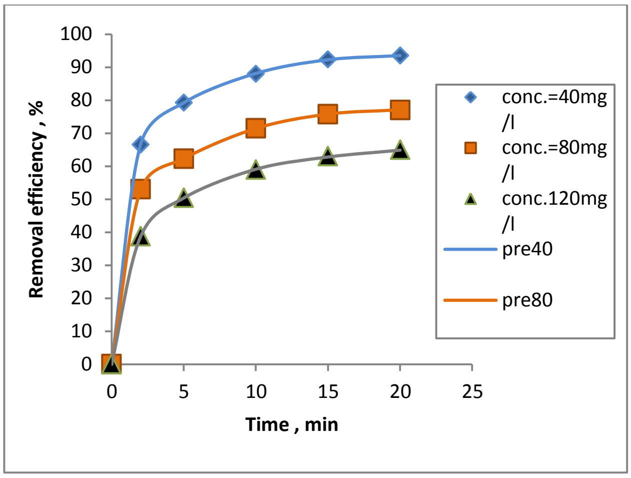

Fig. 13. Removal efficiency as a function of initial metal ions concentration

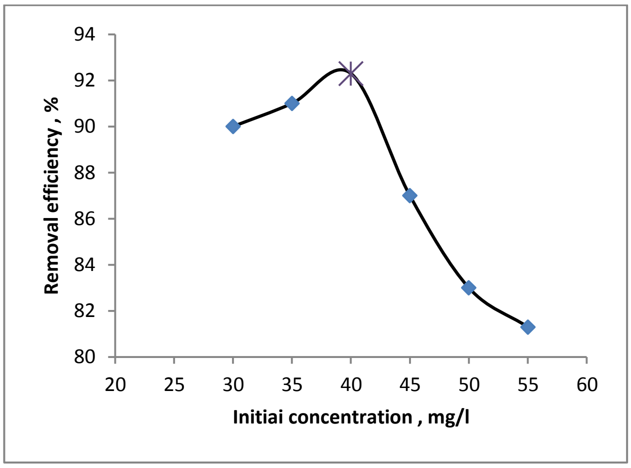

Fig. 14. Effect of Cd(II) ions concentration predicted around the best value on the removal efficiency

Table 5. Kinetic flotation at different initial Cd(II) ions concentration

	Co=40mg/l	Co=80 mg/l	Co=120 mg/l
$k\times10^2$ (min^{-1})	9.256	3.998	2.994

4.8. Influence Ionic Strength

Fig. **15** shows the influence of NaCl addition to the cadmium ions removal at different concentration (0, 10 and 25 mg/l), while all other parameters were kept constant (pH5, initial cadmium ions concentration40mg/l, flow rate 250cm^3/min and SDS concentration 10mg/l), the results of this figure explain that the removal rate reduced significantly with increasing NaCl concentration.

The reduction of cadmium ion in the presence of NaCl could be attributed to the competitive effect between Cd(II) ions and Na from the salt so the metal ions cannot find enough SDS surfactant molecules to attach to [**29**], this is in agreement with the finding of[**27**] Fig.15 also, shows the agreement between the experimental data and the predicted.

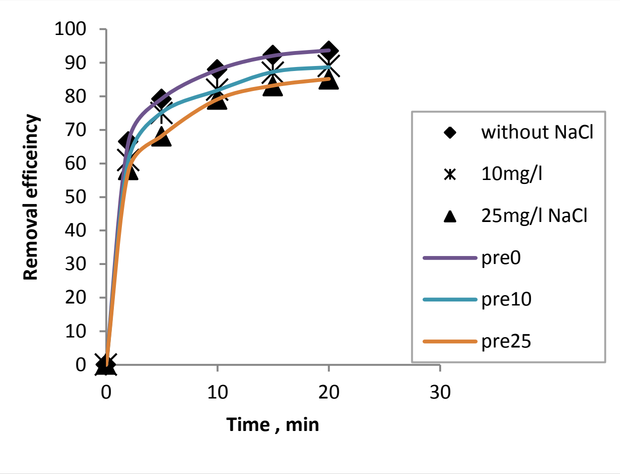

Fig. 15. Effect of salt concentration on the Cd(II) ions

4.9. Turbidity

An important aspect of water quality is turbidity, it is estimated as the liquid cloudiness and caused by colloidal matter and suspends like clay, silt, inorganic matter and finely divided organic, plankton and other microorganisms [**30**].

Fig. **16** shows that the efficiency of turbidity removal was increased from 90.23% to 96.88% by increasing air flow rate from 100 to 250cm^3/min .So, it is concluded that flow rate has a significant influence in reducing turbidity due to increase the bubble rise velocity [**31**].

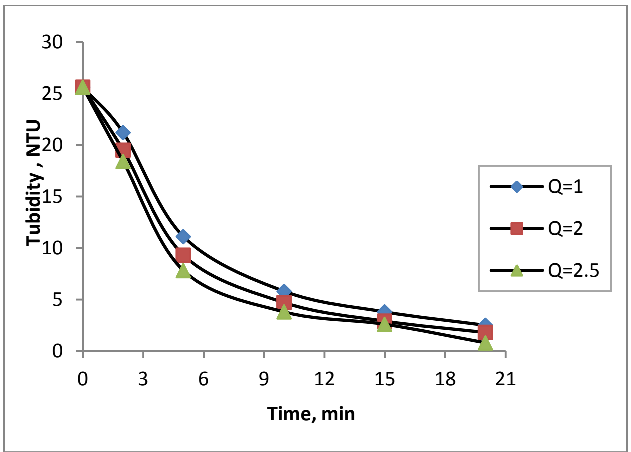

Fig. 16. Removal of turbidity at different flow rate

4.10. Analysis of Sensitivity

Garson suggested an equation assess the relative importance of the input variables depends upon the connection weights partitioning, as can be seen in Eq. (4) [**31**].

$$Ij = \frac{\sum_{m=1}^{m=Nh}\left(\left(\frac{\left|w_{jm}^{ih}\right|}{\sum_{k=1}^{Ni}\left|w_{km}^{ih}\right|}\right)\times\left|w_{mn}^{ho}\right|\right)}{\sum_{k=1}^{k=Ni}\left\{\sum_{m=1}^{m=Nh}\left(\frac{\left|w_{km}^{ih}\right|}{\sum_{k=1}^{Ni}\left|w_{km}^{ih}\right|}\right)\times\left|w_{mn}^{ho}\right|\right\}} \quad (4)$$

Where; Ij is the relative importance of the jth input variable on the output variable, Ni and Nh are the numbers of input and hidden neurons, respectively, the superscripts i, h and o refer to input, hidden and output layers, respectively, Ws are connection weights, and subscripts k , m and n refer to input, hidden and output neurons, respectively.

Fig. 17 presents that the most important parameter is pH with relative importance of 25.13%.It Sensitivity analysis varied depending on reactive material and the type of contaminant **[17].**

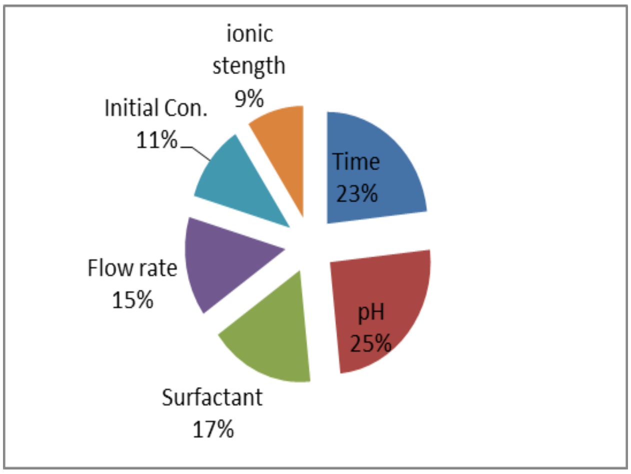

Fig. 17. Piechart for relative importance predicted using ANN model

5- Conclusion

In this work, ANN model was used to examine the performance of microbubble dispersed air floatation method for the removing of Cd (II) ions from simulated wastewater. The maximum correlation coefficient was more than 0.999 which confirmed exact and powerful results experimentally. Hidden neuron of LMA were 11neurons having RMSE 1.062. The sensitivity analysis showed that the reactions were influenced by the pH, input concentration of Cd (II) ions, SDS surfactant concentration, flow rate and ionic strength but pH appeared the most influence parameters with relative importance of 25.13 %. The maximum removal efficiency achieved was 92.3% at pH=5, 20mg/l initial Cd(II) concentration, 10mg/l surfactant concentration and 250cm^3/cm^3 flowrate at 15min contact time . The kinetic flotation order for Cd (II) ions almost first order and the removal rate constant (k) increases with decreasing the initial metal concentration and increasing flow rate.

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التعويم باستخدام الفقاعات المايكروية لازالة ايونات الكادميوم من المحاليل المائية: موديل الشبكة العصبية الاصطناعية والتعويم الحركي

الخلاصة

تهدف هذه الدراسة الى تطبيق تقنية دقائق الهواء المتناهية الصغر في طريقة تعويم الهواء لإزالة أيونات الكادميوم من المحاليل المائية وذلك من خلال دراسة مجموعه من المتغيرات :الرقم الهيدروجيني (3 و 4 و 5 و 6) والتركيز الأولي للأيونات الكادميوم (40و 80 و 120 ملغم / لتر) وقت الاتصال (2 و 5 و 10 و 15 و 20 دقيقة) ، تركيز المواد الخافضة للشد السطحي (10 و 20 و 40 ملغم / لتر) من أجل ايجاد أفضل الظروف وتشير النتائج التجريبية إلى أن دقائق الهواء المتناهية الصغر كانت فعالة جدًا في إزالة أيونات الكادميوم وأن مادة SDS أكثر فعالية من CTAB كمادة خافضة للشد السطحي في عملية التعويم.وبكفاءة عالية بلغت 92.3٪ تحققت خلال 15 دقيقة عند دالة حامضية 5 ، وتركيز مادةSDS (20 ملغم / لتر)و معدل تدفق 250 سم ^{3} / دقيقة وبتركيز اولي (4ملغم/لتر).

كما تضمنت الدراسة تطبيق برنامج ANN من اجل التنبؤ بازالة ايونات الكادميوم من المحاليل المائية خلال 11 طبقة من الخلايا العصبية المخفية ، مع معامل ارتباط 0.9997 بين مخرجات ANN والبيانات التجريبية ومن خلال تحليل الحساسية تبين أن الرقم الهيدروجيني (25.13٪) هو اكثر معامل يؤثر على العملية كماتوصلت الدراسة الى ان كان موديل يمثل النتائج العملية هو موديل التعويم الحركي من الدرجة الثانية ويزيد ثابت معدل إزالة مع انخفاض تركيز المعادن الأولي.

الكلمات الدالة : أيونات الكادميوم ، التعويم ، دقائق الهواء المتناهية الدقة ، ANN، معدل التعويم الحركي.