

## **BIOSORPTION OF IRON FROM AQUEOUS SOLUTION BY DRIED BIOMASS OF ACTIVATED SLUDGE**

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### **ABSTRACT**

This study was conducted to investigate the removal of iron by dried biomass of activated sludge. Dried activated sludge, prepared as a powder, was tested as a sorbent for the removal of iron from aqueous solutions. The effects of various experimental parameters including initial iron concentration, mass of biomass and contact time were examined and optimal experimental conditions were obtained. The equilibrium time for iron adsorption onto biomass was determined as 150 min. The rate of iron removal was directly correlated to biomass amount and contact time. Increasing contact time from 0.5h to 2.5h resulted in 25% improvement in iron removal efficiency. When the weight of the biomass increased from 0.1g to 0.9 g, the iron removal efficiency increased from 62% to 95%. High initial iron concentration had an adverse effect on iron removal efficiency. Increasing initial iron concentration from 2 to 10 (mg/L) caused declining the iron removal efficiency from 70% to 56%. Freundlich, Langmuir, Dubinin-Radushkevich and Temkin isotherm models could describe the adsorption equilibrium of the iron onto the activated sludge. Meanwhile, Freundlich isotherm represented a better correlation compared to other isotherms ( $R^2 > 0.999$ ). Also Elovich kinetic models fits well with experimental data ( $R^2 > 0.997$ ).

**Key words:** Biosorption, Dried Activated Sludge, Iron, Adsorption

### **INTRODUCTION**

The production of metals has rapidly increased since the industrial revolution. Some metals usually form compounds that can be toxic, carcinogenic or mutagenic, even in very low concentrations (Manriquez *et al.*, 1977; Kazemian and Mallah, 2008 ; Picardo *et al.*, 2009). Iron is commonly found in rocks and soil. Under proper conditions, iron will leach into the water resources from rock and soil formations. Exceeding iron concentration greater than 0.3 mg/L causes water staining that adversely affect plumbing fixtures, dishware and clothes and produce a yellow to reddish appearance in water. These levels may also impart taste and odor of drinking water. The US Environmental Protection Agency (USEPA)

has established a secondary drinking water regulation of 0.3 mg/L for iron (EPA, 2000; Ncibi *et al.*, 2007).

Conventional techniques for metal removal from aqueous solutions include chemical precipitation, ion exchange, adsorption process, membrane process, crystallization and electrochemical treatment. Considering the cost, technical complexity and limitations of the conventional methods make the alternative biological methods more appealing (Volesky, 1990; Schiewer and Volesky, 1995). Biological process for metal ions removal from aqueous solutions can be divided into three general categories: (a) sorption of metal ions onto the surfaces of a microorganism, (b) intracellular uptake of metal ions, and

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(c) chemical transformation of metal ions by microorganisms. The latter two processes require live organisms to proceed (Veglio and Beolchini, 1994; Mamisahebei *et al.*, 2007).

The adsorption of metals onto various types of biomass has been introduced as a cost-effective method in multiple studies. In this method, in addition to direct use of microbial biomass, their by-products including biosorbent may be applied as a low cost method of metal-removal process. In addition to fermentation processes, production of biosorbent can be considered by employing simple and inexpensive culture propagation techniques (Kuyucak, 1990; Gupta and Rastogi, 2008; Mata *et al.*, 2008; Pejic *et al.*, 2008; Vitor and Corso, 2008; Yurtsever and Sengil, 2008; Dang *et al.*, 2009; Quintelas *et al.*, 2009). Sorption of heavy metals by various biological materials has been proposed as an efficient and potentially cost effective tool for metal enriched industrial effluents. Biosorption is a property of certain types of inactive, dead microbial biomass to bind and concentrate heavy metals from very dilute aqueous solutions (Gaur and Dhankhar, 2009).

Capability of microorganisms to bind heavy metals in aqueous solutions has long been of scientific interest (Aksus and Gulen, 2002; Sen and Ghosh Dastidar, 2007). Mapolelo *et al.* (2005) showed that yeast is a viable trace metal enrichment agent that can be used freely by suspending in solution to enrich metal ions at a relatively low concentration (Mapolelo *et al.*, 2005). Biosorption of uranium by filamentous fungus *Uromyces* has been documented in another study (Guibal *et al.*, 1999). Activated sludge from wastewater treatment processes is probably the most abundant source of mixed biomass. The ability of activated sludge biomass to remove and accumulate heavy metals and organics has been recognized in different studies (Al-Asheh and Duvnjak, 1995).

The use of biomass or its derived products has carried the advantages of reducing dissolved metals related toxicity, improving operating conditions, and cost-benefit utility maintenance including nutrient supply (Hua-Jun *et al.*, 2008). The aim of this study was to investigate the adsorption rate and kinetics of removing iron by using dried activated sludge (DAS) biomass.

## MATERIALS AND METHODS

### *Preparation of dried activated sludge*

Activated sludge was obtained from a food industrial wastewater treatment plant in Hamedan city of Iran. The activated sludge was dried at 105°C for 12 h to a constant weight and sieved through a 205 µm diameter mesh. All glass and other instruments were washed with detergent and distilled water to be free from any precontaminations.

### *Preparation of iron solution*

Solution of 500 mg/L of iron was prepared as stock solution and diluted to 2, 3,4,5, and 10 mg/L for the experiments.

### *Batch adsorption experiments*

Erlenmeyer flasks were filled with 10 mg/L of iron solution and different amount of biomass (measured as 0.1, 0.3, 0.5, 0.7 and 0.9 g) mixed to 100mL volume. Five Erlenmeyer flasks filled with dried activated sludge and deionized water and a control flask containing only iron with no adsorbent were used to determine the sludge initial iron and extent of iron removal via non-adsorptive mechanisms. The control flasks contained similar solutions, but no activated sludge. The flasks were charged with different concentrations of DAS and allowed to shake for 1– 4 h. Upon completion of shaking, (HANA-HI 190 model), the samples were withdrawn at 30, 60, 90, 120 and 150 minutes and their iron concentration was analyzed. The effect of different retention times (30, 60, 90, 120 and 150 min) with variation of initial concentration (2, 3, 4, 5 and 10mg/L) at constant pH and temperature on removal of iron by dried mass was considered in batch system. All experiments were carried out at pH= 7±0.2 and temperature =20±0.2°C.

### *Analytical methods*

For separation of activate sludge from the solution, samples were centrifuged (Model-301 Sigma) at 3800 rpm for 15 min. The iron concentration in the supernatant was measured using a spectrometer( UV-1700 Pharma Spect Shimadzo) at a 510 nm wavelength and computing from the calibration curves(Lenore *et al.*, 1998). The amount of iron adsorbed by biomasses was calculated using the

following equation.

$$q_e = \frac{(C_0 - C)V}{M} \quad (1)$$

Where:  $q_e$  (mg/g) is the amount of iron adsorbed by biomass;  $C_0$  and  $C$  (mg/L) are the initial and equilibrium liquid-phase concentrations of iron, respectively;  $V$ (L) represent the initial volume of iron solution, and  $M$ (g) the mass of the biomass. The performance of DAS adsorption was evaluated in terms of its removal efficiency as  $RE(\%)$ , estimated by the following equation:

$$RE(\%) = \frac{(C_0 - C_t)}{C_0} \times 100$$

Where  $C_t$  is the iron concentration at time  $t$  (Xing *et al.*, 2008).

#### Adsorption isotherm

Adsorption isotherm is the basic requirement for designing any adsorption system. Isotherm express the relation between the amounts of adsorbate removed from liquid phase by unit mass of adsorbent at constant temperature. Since adsorption is one of the fundamental surface phenomena, it is important to have a satisfactory description of an equilibrium state in order to

successfully represent the kinetic adsorption behavior of any species from the fluid to the solid phase (Passos *et al.*, 2008). Langmuir, Freundlich, Dubinin-Radushkevich and Temkin adsorption isotherms were used to analyze the biosorption isotherms of iron.

#### Adsorption kinetics

In order to determine the potential rate-controlling steps involved in the adsorption process, kinetic models should be established (Zhi-Yan *et al.*, 2008). Therefore, the first order, pseudo-second-order kinetics and Elovich were used to analyze the biosorption kinetics of iron.

## RESULTS

### Influence of initial iron concentration

The influence of initial iron concentration on the biosorption of iron is shown in Table.1. Considering steady values of other parameters, the initial iron concentration was increased from 2 to 10 (mg/L) which caused a removal efficiency decline from 70% to 56%. Adsorption capacity of dried activated sludge increased with increasing the initial iron concentration.

This was due to an increase in the driving force of the concentration gradient, as an increase in the initial iron concentration.

Table 1: Effect of initial iron concentration on iron removal efficiency by biomass (contact time= 2.5 h, amount of biomass = 0.9 g , pH = 7±0.2 and temperature= 20±0.2°C )

Initial iron concentration (mg/L)	Final iron concentration (mg/L)	Removal efficiency (%)
2	0.62	70
3	1.12	63
4	1.55	61
5	2.12	58
10	4.52	56

### Influence of contact time

The influence of contact time on the biosorption of iron is shown in Table. 2. Increasing contact time from 0.5h to 2.5h caused a removal efficiency

improvement from 56% to 89%. The association between contact time and iron removal efficiency was statistically significant ( $p < 0.05$ ).

Table 2: Effect of contact time on iron removal efficiency by biomass (initial iron concentration= 10mg/L, amount of biomass = 0.9 g, pH= 7±0.2 and temperature= 20±0.2°C)

Time (h)	Initial iron concentration (mg/L)	Final iron concentration (mg/L)	Iron removal rate (mg/h)	Removal Efficiency (%)
0.5	10	4.52	10.96	56
1	10	3.2	2.64	68
1.5	10	2.55	1.3	75
2	10	2.01	1.08	80
2.5	10	1.02	1.98	89

*Influence of the mass of biomass*

The influence of mass of the biomass on the iron biosorption is shown in Fig 1. When the weight of the biomass was increased from 0.1g to 0.9 g, the iron removal efficiency increased from 62% to 95%. Nearly 150 min was required for the equilibrium adsorption for iron. Therefore, the equilibrium time was set conservatively at 150 min for further experiments. The variations of iron removal with initial DAS mass for 10 mg/L iron at the end of 150 min incubation period are shown in Fig.1. Iron removal by adsorption improved by increasing DAS concentrations for

all samples with different iron concentrations and reached a maximum level at 0.9 g DAS mass.

*Adsorption isotherm*

Langmuir, Freundlich, Dubinin-Radushkevich and Temkin adsorption isotherms were employed to determine the preference of one to another. Plot for the nonlinear Langmuir Freundlich, Dubinin-Radushkevich and Temkin equation are shown in Fig.2. Table 3 gives the values of the isotherm constants.

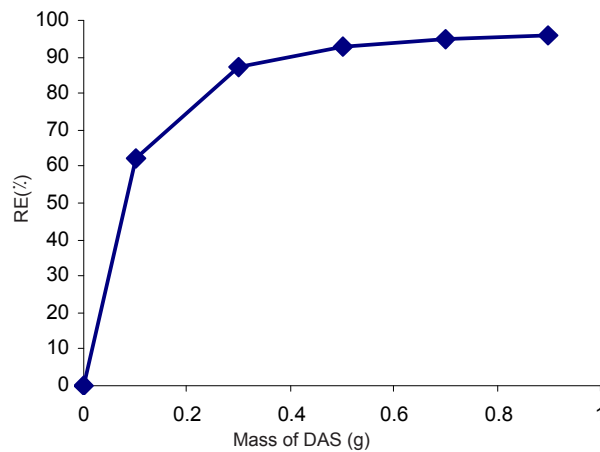


Fig.1: Effect of biomass amount (g) on iron removal efficiency by biomass (contact time: 2.5 h, initial iron concentration: 10 mg/L, pH 7±0.2 and temperature 20±0.2°C)

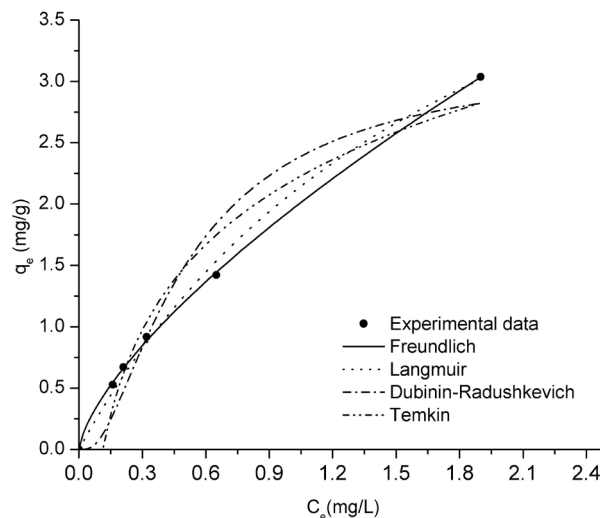


Fig. 2: Langmuir, Freundlich, Dubinin-Radushkevich and Temkin isotherms for the adsorption of iron by DAS

Table 3: Langmuir, Freundlich, Dubinin-Radushkevich and Temkin isotherm constants

Isotherms	Equation	Constant	Value	Standard error	R <sup>2</sup>
Langmuir	$q_e = \frac{q_m b C_e}{1 + b C_e}$ (Langmuir, 1918)	q <sub>m</sub>	6.0388	0.5781	0.9945
		b	0.52518	0.08377	
Freundlich	$q_e = k_f C_e^{1/n}$ (Freundlich, 1906)	K <sub>f</sub>	1.94638	0.0132	0.99952
		n	1.4454	0.01889	
Dubinin-Radushkevich	$q_e = Q_s \text{Exp}(-B \varepsilon^2)$ $\varepsilon = RT \ln \left( 1 + \frac{1}{C_e} \right)$ Dubinin and Radushkevich, (1947)	q <sub>d</sub>	3.15117	0.33376	0.92751
		B <sub>d</sub>	2.54E-04	5.01E-05	
Temkin	$q_e = \frac{RT}{b_T} \ln(K_T C_e)$ (Temkin and Pyzhev, 1940)	A	8.81611	1.59	0.95447
		b <sub>t</sub>	2431.585	277.33134	

The essential characteristics of the Langmuir isotherm can be expressed in terms of dimensionless constant separation factor or equilibrium parameter, R<sub>L</sub>, which is defined by Eq. (3) (Hall, 1996):

$$R_L = \frac{1}{1 + bC_0} \quad (3)$$

Where: C<sub>0</sub> (mg/L) is the highest initial iron concentration. According to the value of R<sub>L</sub> the isotherm shape can be interpreted as presented in Table 4 (Langmuir, 1918):

Table 4: Langmuir isotherm constant parameter, R<sub>L</sub>

R <sub>L</sub> value	Type of isotherm
R <sub>L</sub> >1	Unfavorable
R <sub>L</sub> =1	Linear
R <sub>L</sub> = 0	Irreversible
0< R <sub>L</sub> <1	Favorable

The Freundlich constant, n, should have values lying in the range of 1 to 10 for classification as favorable adsorption.

#### Adsorption kinetics

To find the potential rate-controlling steps involved in the process of biosorption of iron onto DAS, first order, Elovich and pseudo second-order kinetic models were tested to fit the experimental data at various DAS. Adsorption kinetics of iron first- order model, pseudo second-order model and Elovich models shown in Fig 3, and Table 5 shows the comparison of first-order, pseudo second-order and Elovich kinetics constants.

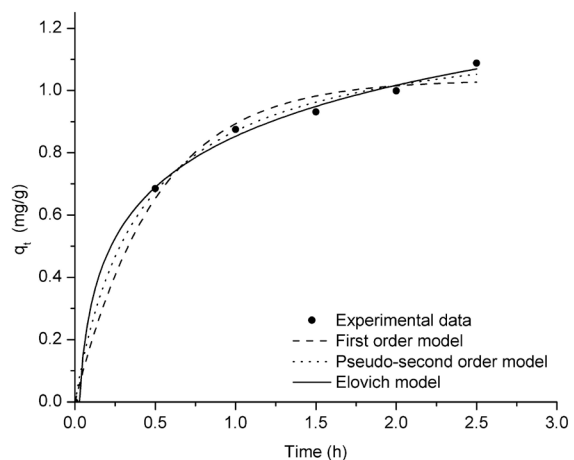


Fig.3: Kinetic experimental data and first- order, pseudo second-order and Elovich models (pH =7±0.2 and temperature =20±0.2°C)

Table.5: Comparison of first-order, pseudo second-order and Elovich kinetic constants

Models	Constants	Value	Standard Error	R <sup>2</sup>
First-order	k <sub>1</sub>	1.99642	0.25921	0.9875
	q <sub>e</sub>	1.03401	0.03262	
Pseudo second-order	k <sub>2</sub>	2.01469	0.33186	0.99577
	q <sub>e</sub>	1.22298	0.0394	
Elovich	a	8.75299	1.63888	0.99769
	B	4.23498	0.27083	

The isotherm and kinetic models parameters were determined using the trial-and-error nonlinear method by MATLAB software.

## DISCUSSION

A high correlation was represented for four Freundlich, Langmuir, Dubinin-Radushkevich and Temkin isotherms. However regarding the correlation coefficient (R<sup>2</sup>), the Freundlich adsorption isotherm (R<sup>2</sup> > 0.999) was found to represent the equilibrium adsorption data with much better fitting compared to Langmuir isotherm (R<sup>2</sup> > 0.994), Dubinin-Radushkevich (R<sup>2</sup> > 0.927) and Temkin (R<sup>2</sup> > 0.954). Calculated R<sub>L</sub> for Langmuir isotherm was 0.16; so, Langmuir isotherm was favorable. Calculated *n* for Freundlich isotherm was 1.4454; hence, Freundlich isotherm was favorable for experimental data. The correlation coefficients for the second-order kinetic model were close to (R<sup>2</sup> > 0.995) for all cases. The correlation coefficients for the Elovich kinetics was (R<sup>2</sup> > 0.997) and for first-order model was (R<sup>2</sup> > 0.987). The theoretical values of q<sub>e</sub> also agreed well with the experimental data. The theoretical q<sub>e</sub> values calculated from the first-order kinetic model did not give reasonable values with obvious deviation from the experimental ones. This may imply that the biosorption of iron on DAS concentration follows Elovich rather than pseudo second-order kinetics.

In another study the kinetics of heavy metals biosorption by inactive biomass of the marine algae *Sargassum* were fast, reaching 60% of the total biosorption capacity in ten minutes. All charts were linear (their regression line was acceptable) and all of the data obtained for isotherm constants in different experiments were in a same range and on echelon type after equilibrium time (Barkhordar and Ghiasseddin, 2004). The rate constants of the pseudo second-

order equation for Pb<sup>2+</sup> biosorption were obtained as 1.86, 0.83 and 0.52 gm/mol/min at initial Pb concentrations of 5, 1 and 0.5 mM, respectively (Nabizadeh *et al.*, 2005). Both Freundlich and Langmuir isotherm models could describe the adsorption equilibrium of the reactive dye onto the activated sludge with the Langmuir isotherm showing better agreement. Second-order kinetic models confirmed the agreement (Ju *et al.*, 2008). In another study the pseudo second-order and first-order models described the biosorption kinetics of As(V) with good correlation coefficient (R<sup>2</sup> > 0.93) and better than the other equations. The data obtained from the experiment of biosorption isotherm were analyzed using the Freundlich and Langmuir isotherm models. The equation described the isotherm of As(V) biosorption with relatively high correlation coefficient (R<sup>2</sup> > 0.93) (Mamisahebei *et al.*, 2007).

Considering constant values of other parameters, an increase in initial iron concentration resulted in declining iron removal efficiency but improving dried activated sludge adsorption capacities. This may be due to an increase in the driving force of the concentration gradient, as an increase in the initial iron concentration. This study showed that DAS has an equilibrium adsorption capacity for iron. The iron removal rate at the end of the 2.5 h incubation period increased and the effluent iron concentrations decreased with increasing DAS concentrations and decreasing initial iron concentration.

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