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Caspian J. Env. Sci. 2014, Vol. 12 No.1 pp. 15~29

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CJES

Caspian Journal of Environmental Sciences

[Research]

Biosorption of simulated aqueous solution containing acidic dyes by *Azolla filliculoides*

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(Received: Apr. 16. 2013, Accepted: Sept. 05. 2013)

ABSTRACT

Biosorption of acidic dyes using the live fern *Azolla filliculoides* was studied in a discontinuous system. Dye parameters, dye initial concentration and contact time were studied in temperature range of 25-30 ºC and pH=7. It was shown that increasing the 23initial concentration of dye and its contact time resulted in decreasing the dye taking quantity by the absorbent. Also, type of dye has an effective role in the process. The highest dye taking capacity was reported in the concentration of 15 mg/L that was 64.52%, 37.53%, and 32.98% for acidic red 14, blue 25, and yellow 17 dyes respectively. Adsorption isotherm models of Langmuir, Freundlich, Dubinin- Radushkovich, and Temkin were analyzed in different concentrations. Adsorption kinetic data were considered by kinetic models of pseudo-first-order and pseudo-second-order.

Keywords: *Biosorption, Acidic dyes, Kinetic, Isotherm, Azolla filliculoides, Wastewater*

INTRODUCTION

Environmental pollution and its monitoring is one of the major anxieties of communities in the 21th century. The entry of unfiltered effluent of industries into natural ecosystem, has resulted in serious problems to the environment (Padmesh *et al.* 2005). Among industrial effluents, colored effluents of color and textile industries are the most problematic ones due to the aromatic and artificial complex structure of dyes (Fewson 1988 and Viraraghavan 2001). Dyes used in textile industry have different structures such as acidic, reactive, basic, disperse, azo, antrakinon, and metal complex (Banat *et al.* 1996).

Dye removal methods from of industrial effluents include biological infiltration, accumulation, adsorption, oxidation, filtration, and so on (Mckay Mckay *et al.* 1999 and Vanderviere *et al.* 1998). Recently, many technologies have focused on low-costs and bio-friendliness for infiltration of polluted effluents and among them biological method have been selected (Venkatamohan *et al. 2002*). Biosorption refers to dye removal from water solutions by living organisms. Researchers have reported many biosorbents for dye biosorption from water solutions that include bacteria (Aksu 2004), fungus (Hu 1996 and Gallagher *et al.* 1997), and algae (Viraraghavan 2001). These biosorbents have shown high efficiency in dye removal of water environments.

Among the biosorbents, *Azolla filliculoides* is an aqueous fern and its stems are like floating branching rhizomes with

small leafs that are placed alternately on each other. The stems are suspended in water (Padmesh *et al.* 2005; Smith and Alleman 1994). Recently, *Azolla* has received a lot of attention. It has been reported that *Azolla* has been used widely to remove heavy metal ions and dyes from solutions and it has shown a high potential in biosorption (Aksu & Tezer 2005 and Volesky 1999).

In this research, biosorption of three dyes, acidic red 14, blue 25, and yellow 17 by the Azolla fern (*A. filliculoides*) was studied. Adsorption kinetics and isotherm relations were studied to evaluate the contaminators adsorption capacity level.

MATERIALS AND METHODS

Experimental Part

A. filliculoides a living fern that is the dominant species in Iran was collected from the Anzali pond in northern Iran (Fig. 1). It was preserved in water with normal pH for 30 days for compatibility with laboratory conditions, and it was used for biosorption discontinuous tests. Since we were dealing with a living creature, attempts were made as much as possible to maintain the natural conditions of pH and temperature. Experiments were conducted only in the presence of *A. filliculoides* and other aquatic plants such as *Lemna minor* were disregarded.

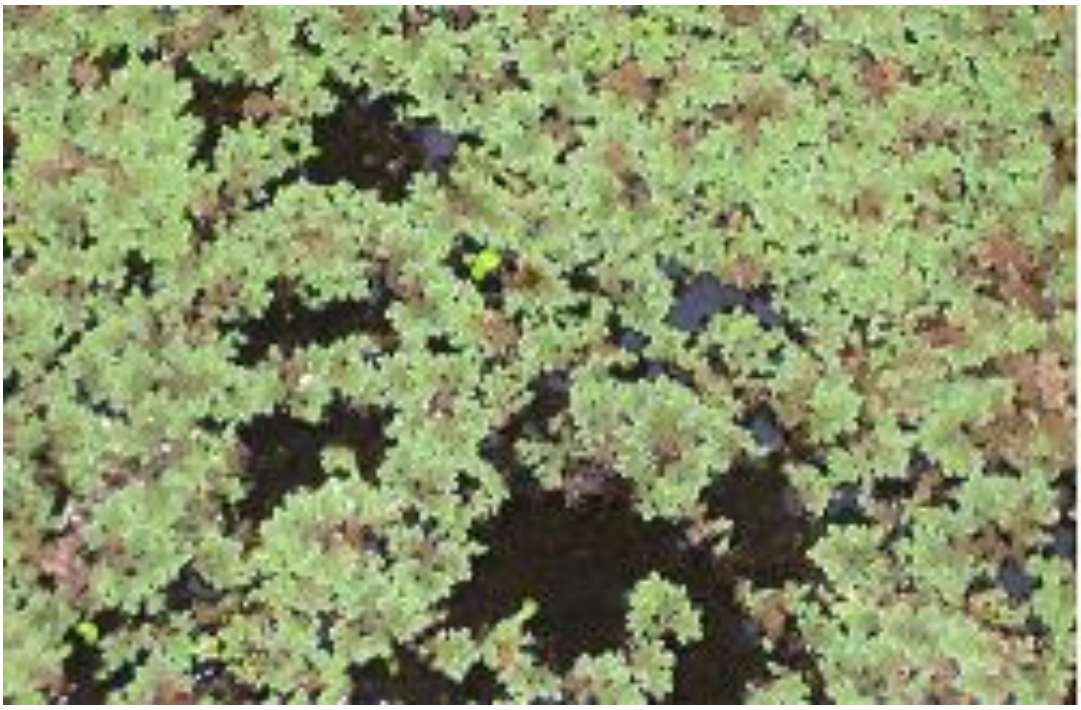

Fig. 1. *Azolla filliculoides* in Laboratory conditions

Acidic red 14 (AR14), yellow 17 (AY17), and blue 25 (AB25) used in this study were prepared at the Iran Kimia Kav Company. Their chemical structure and specifications have been shown in (Fig. 2 and Table 1) respectively. The light source used was two 40 W electric bulbs and long fluorescent lamps.

The light intensity was measured by Testo 545 photometer. In order to measure concentration changes during process, the values of which are shown in Table 1, a Genova MK3 UV/Visible spectrophotometer was used.

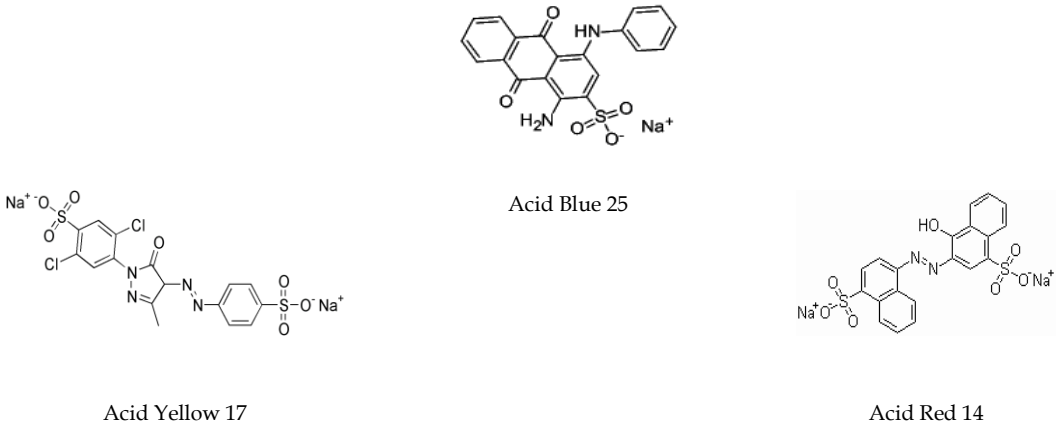

Fig. 2. Chemical structure of three used acidic dyes in research (Ghodbane and Hamdaoui 2010; Gao *et al.* 2010 & Idel-aouada *et al.* 2010).

Table 1. Characteristic of three used acidic dyes in research

Color index name	Molecular formula	Chemical class	λmax(nm)	Mw
Acid Blue 25	$C_2OH_{13}N_2Na0_5S$	Antrakinon	602	416.38
Acid yellow 17	$C_2OH_{13}N_2Na0_5S$	Mono azo	402	551.29
Acid red 14	$C_2OH_{12}N_2Na_20_5S$	Mono azo	515	501

METHODS

Biosorption discontinuous tests were implemented in 1L beakers containing 4g of *Azolla* living fern in 300ml simulated aqueous solution with three acidic dyes in 5 initial concentrations of 15, 30, 45, 60, and 75 mg/l in temperature range of 25-30°C, pH=7, and the solution were preserved there for 14 days. Beakers were preserved in an aquarium that was completely covered with aluminum foils to prevent light influence and subjected to lamp lights to simulate sun light. Dye concentration in aqueous solutions was determined by spectrophotometer.

Adsorbed dye quantity was calculated from the difference between added dye and adsorbed dye content by the following formula:

$q_e = (C_0 - C_e) \times V / M$ (1)

The dye removal percentage (%) was calculated by using the following equation:

Dye removal in percentage =

$(C_0 - C_e) / C_0 \times 100$ (2)

where,

q_e= taken dye quantity (mg/g)

C_0= dye initial concentration (mg/L) in solution

C_e= dye balanced concentration (mg/L) in solution

V= solution volume (L)

M= adsorbent mass (mg)

RESULTS AND DISCUSSION

Test Process Parameters Effects on Biosorption

To consider the initial concentration of the dye, the biosorption role of *Azolla* was studied in three dyes in different initial concentrations (15, 30, 45, 60, and 75 mg/l). It was found that by increasing the initial concentration of each dyes, the removal amount (percent) of the dyes decreased. Dynamics for biosorption was different in concentrations between dye on adsorbent and in solution (Aksu & Gönen 2003) and this difference more in AR14.

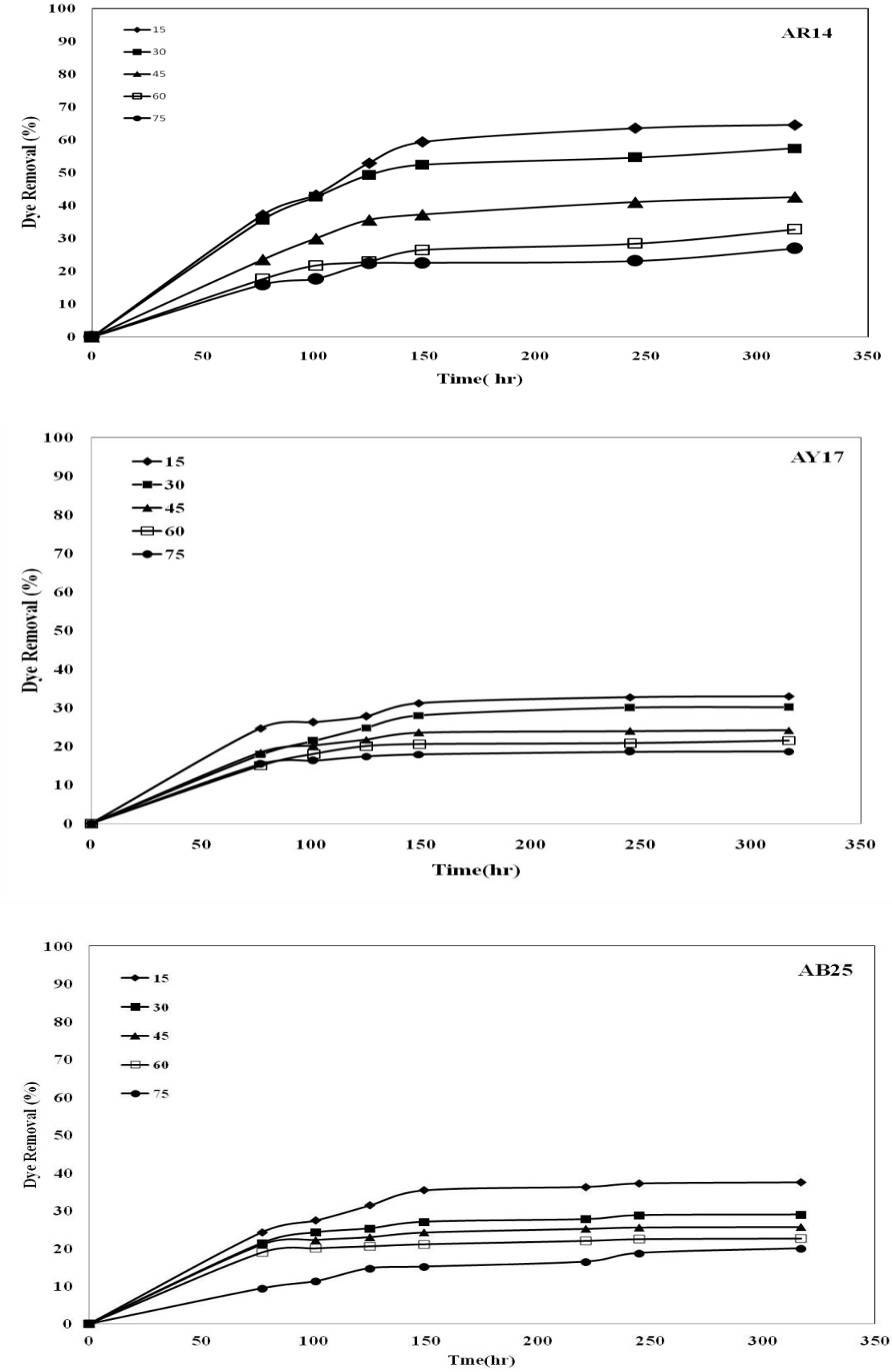

Fig. 3. Effect of initial concentration of AR14, AY17, and AB25 acidic dye on the biosorption potential of *A. filliculoides* for different concentrations (temperature range 25-30ºC and pH=7)

Fig. 3 shows the removal efficiency in three dyes. It was found that with concentration change from 15 mg/l to 75 mg/l, dye removal efficiency for AR14, AY17, and AB25 decreases from 64.51%, 32.98%, and 37.53% to 26.92%, 18.62%, and 19.93% respectively. It reveals that in higher concentrations, the number of adsorption available places becomes lesser and as a result dye removal depends on initial concentration.

Laboratory results showed that in the presence of *Azolla*, dye removal speed was two times higher than those in the other two cases.

Adsorbent contact time with dye is one of the important alternatives in biological infiltration of aqueous solutions. As shown in Fig. 3, adsorption potential and removal efficiency of the three dyes by adsorbent have a direct relation with contact time. Adsorption speed increases at the beginning and then becomes slower. By filling the available pores on the adsorbent surface, the adsorbent will not be able to absorb more dye and there was no significant change in adsorption efficiency after one week.

Molecular structure was noted in the study of the three dyes. As shown in Fig. 3, AR14 dye has a more open structure and more symmetrical molecules in comparison to the two other dyes. Also, it has more sulfated and hydroxyl polar operational groups that have an important role in bonding between dye and adsorbent. Also, monoazo polar bond in AR14 and AY17 is important against the antrakinon bond in AB25.

Adsorption Kinetics Study

There have been several reports (on the use of different kinetic models to adjust the biosorbtion of experimental dyes Tan *et al* 2011 and Padmesh *et al.* 2005 and Rakhshaee 2006). Kinetic models are used to study adsorption mechanism and control potential rate of the steps that help to select an ideal condition for discontinuous process. Pseudo-first-order and pseudo-second-order (Ho and McKay 1998) were used. Pseudo-first-order is determined according to solid capacity the linear form of which is generally as in relation (2):

$$\ln(q_e + q_t) = \ln q_e - k_1 t \quad (2)$$

where

q_e= adsorbed dye quantity in balance condition (mg/g)

q_t= adsorbed dye quantity in *t* time, and

k_1= first order kinetic speed balance constant (L/min) that is taken from

$ln(q_e + q_t)$ line diagram slope against *t* (Fig 4). Pseudo-second-order is determined according to solid phase adsorption the linear form of which is as relation (3):

$$t/q_t = 1/k_2 q_e^2 + (1/q_e)t \quad (3)$$

Where:

K_2 is second order kinetic equation balance speed constant (g/mg.min) that is got from t/q_t diagram drawn against t and calculation of width from origin.

Regarding results of this test (Table 2), generally for different concentrations of the three tested dyes, second-order kinetic model is a proper model for biosorption by *Azolla*. Fig. 5 shows the results of kinetic first-order and second-order respectively.

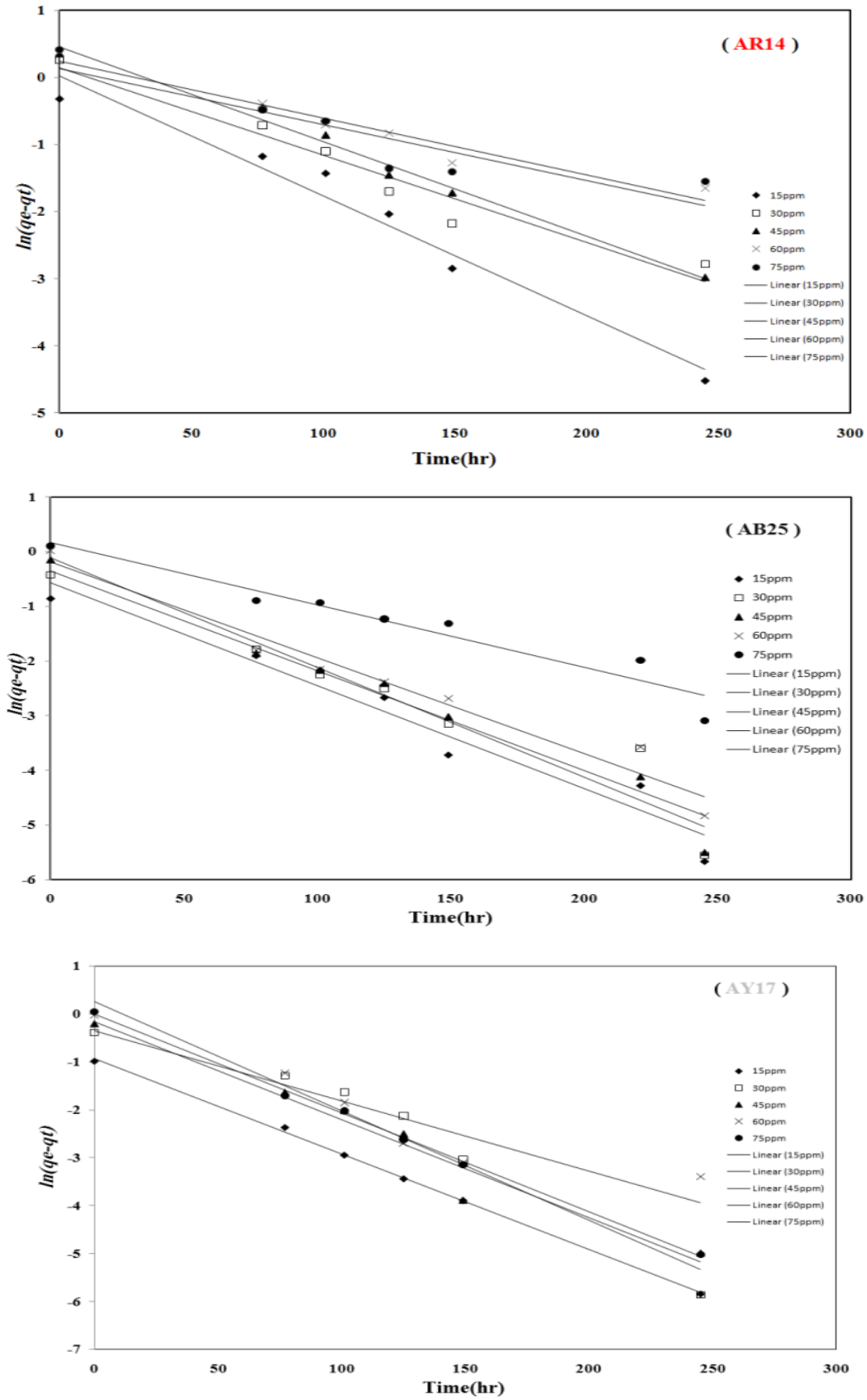

Fig. 4. Pseudo-first-order biosorption kinetics of AR14, AB25 and AY17 the *A. filliculoides*. Conditions: temperature range 25-30°C and pH=7.

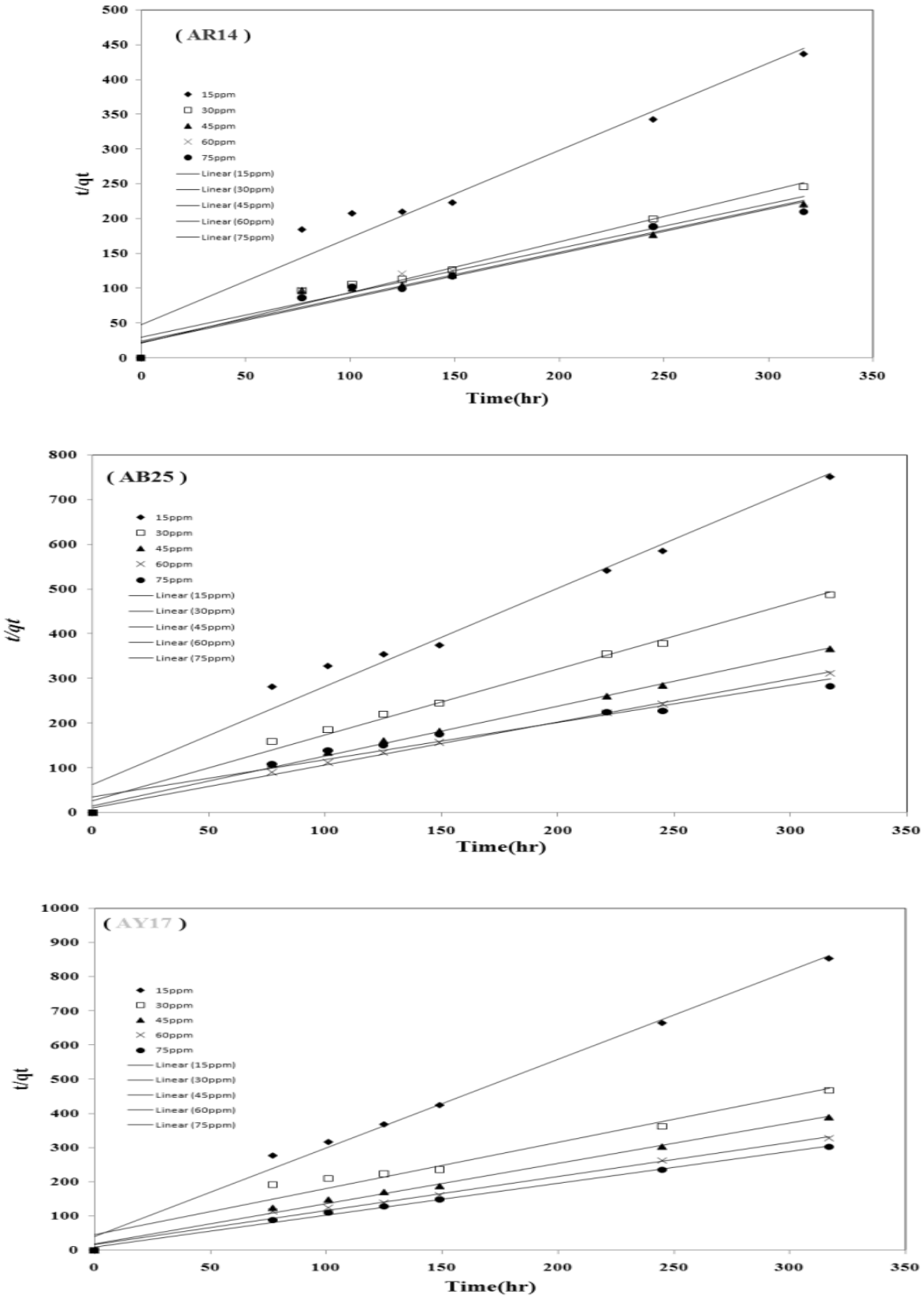

Fig. 5. Pseudo-secend-order biosorption kinetics of AR14, AB25 and AY17 the *A. filliculoides*. Conditions: temperature range 25-30°C and pH=7.

Table 2. Kinetic parameters for dye biosorption on *Azolla* in different concentrations

Dyes	Initial conc. (mg/L)	Pseudo-first-order model			Pseudo-second-order model			
		$q_{e,cal}$(mg/g)	k_1(min^{-1})	R^2	k_2 (min^{-1})	$q_{e,cal}$(mg/g)	R^2	u (mg/(g. min))
AR14	**15**	1.01	0.017	0.96	0.01	0.93	0.97	0.01
	30	1.15	0.013	0.94	0.02	1.36	0.97	0.04
	45	1.74	0.014	0.98	0.01	1.96	0.98	0.02
	60	1.20	0.01	0.94	0.02	1.43	0.96	0.04
	75	1.15	0.008	0.82	0.02	1.56	0.95	0.04
AB25	**15**	0.56	0.018	0.94	2.20	0.45	0.97	0.45
	30	0.70	0.018	0.92	0.08	0.67	0.99	0.04
	45	0.90	0.02	0.97	0.08	0.90	0.99	0.06
	60	0.83	0.017	0.96	0.08	1.04	0.99	0.09
	75	1.18	0.011	0.92	0.02	1.20	0.95	0.02
AY17	**15**	0.40	0.019	0.99	0.17	0.38	0.99	0.02
	30	1.30	0.022	0.93	0.04	0.74	0.96	0.02
	45	0.84	0.02	0.94	0.07	0.90	0.99	0.05
	60	0.70	0.1	0.86	0.06	1.01	0.98	0.05
	75	0.99	0.02	0.99	0.08	1.08	0.99	0.09

The results of the study derived from the pseudo first and second order models are shown in the Table 2 for the biosorbtion of AR14, AB25 and AY17 on the *A. filliculoides* at various concentrations.

Adsorption Isotherm Study

Generally, adsorption balance models are used to show adsorption balance results and calculation of adsorption quantity in different conditions. Adsorption models show how adsorption molecules are distributed in liquid and solid phase in balance time.

The most important adsorption models include linear model adsorption, Langmuir isotherm, Freundlich model, Temkin model, Dubinin-Radushkovich (D-R) model, Brunauer-Emmet-Teller model, etc. The first three models are very important in chemical adsorption. Langmuir and Freundlich models are also important in physical adsorption. Adherence of each system is determined by the curve related to each balance and study of model correlation coefficient (R^2) with experimental results. Among adsorption models, Langmuir model is the first isotherm adsorption model and is based on some assumptions the most important of which are: adsorb materials (atom, molecule, or ion) connect to adsorbent material surface in specific points and a monolayer adsorption occurs. Moreover, there is no reaction between adsorbed materials. Its linear equation is explained as relation 4 (Langmuir 1918):

$$^{1}/_{q_e} = \left(^{1}/_{K_L q_m}\right)\left(^{1}/_{C_e}\right) + {}^{1}/_{q_m} \quad (4)$$

where,

q_e= adsorbed dye quantity in adsorbent mass unite in balance condition (mg/g),

q_m= maximum absorbed dye to create an adsorbed layer or monolayer (mg/g), and

C_e=dye balance concentration in solution (mg/l).

The basic character in Langmuir isotherm is balance constant without R_L that is explained by following equation:

$$R_L = {}^{1}/_{(1 + K_l C_0)} \quad (5)$$

where C_0=dye initial concentration (mg/l). R_L determines adsorption nature and This means that R_L>1 ⇨ non-optimum adsorption, R_L=1 ⇨ linear adsorption, R_L<1 ⇨ optimum adsorption, and R_L=0 ⇨ reverse adsorption.

According to test results (Table 3), biosorption is optimum from Langmuir point of view.

Freundlich model assumes that the surface of adsorbent material is not homogeneous and the quantity of adsorbed material from solution depends on the balance concentration of the adsorbed material in solution. This equation assumes that by increasing surface coverage, adsorption energy decreases logarithmically due to surface heterogeneity that is presented as the following linear relation (Freundlich 1907):

$$\lg q_e = {}^{1}/_{n} \lg C_e + \lg K_f \quad (6)$$

By drawing of q_e logarithm curve as a function of C_e logarithm, n and K_f quantities can be calculated. n and K_f are Freundlich parameters that explain adsorption intensity and absorption capacity respectively.

1/n with quantity between 0 to 1 indicates surface heterogeneity. By approaching n to zero, surface heterogeneity is increased. If 1/n quantity is less than 1, it indicates Freundlich isotherm adsorption, and if it is greater than 1, it indicates communal adsorption. In other words, 1/n quantity less than 1 indicates that color materials adsorption by adsorbent is better in lower concentrations of color in comparison with higher concentrations. With regard to biosorption of the three dyes by the biosorbent it may be concluded that adsorbent surface is heterogeneous and adsorption speed is higher in lower concentrations (Table 3) Figures 6 and 7 show Langmuir and Freundlich biosorption isotherms of three acidic dyes; AR14, AB25, and AY17 by 1g on living *Azolla*.

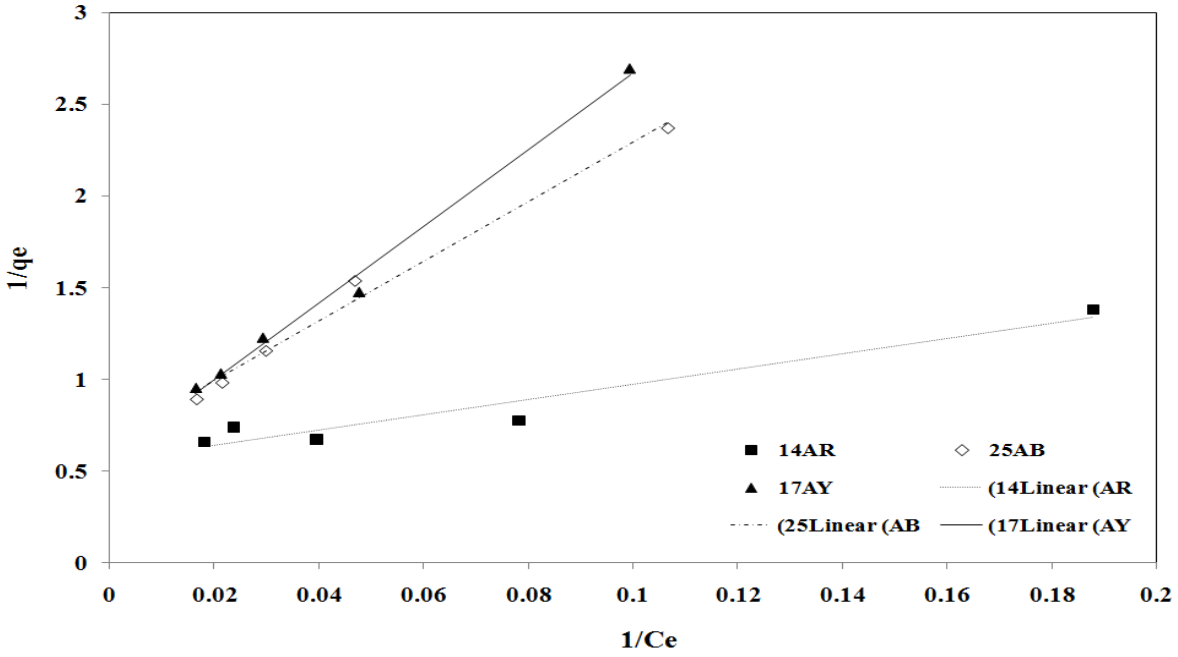

Fig. 6. Langmuir isotherm of AR14, AB25 and AY17 on *A. filliculoides*. Initial Conditions: temperature range 25-30°C and pH=7.

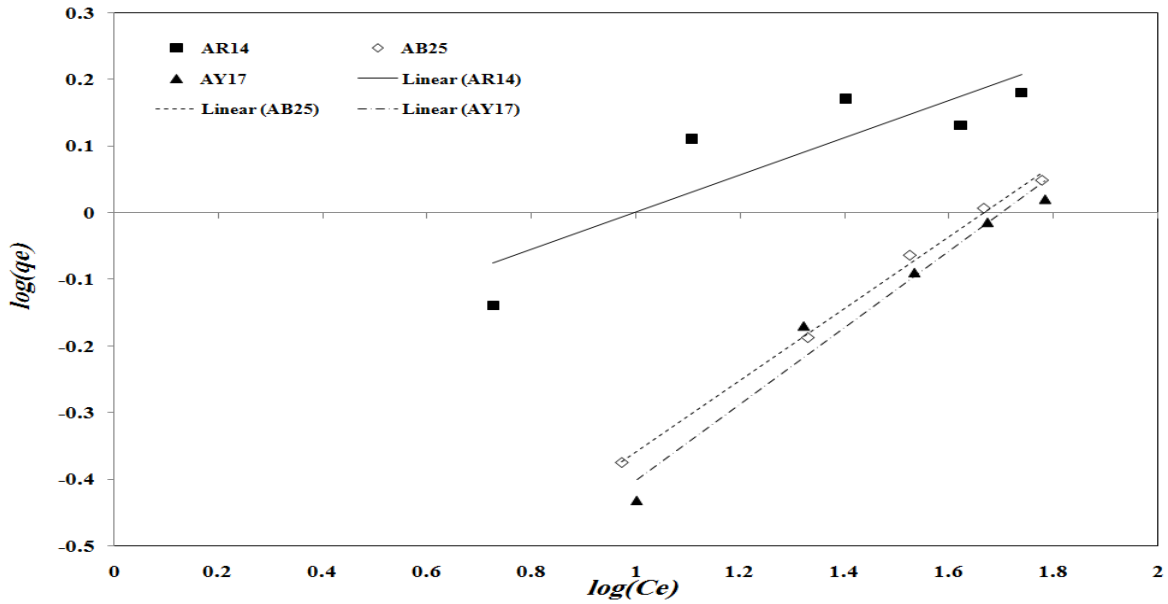

Fig. 7. Freundlich isotherm of AR14, AB25 and AY17 on *A. filliculoides*. Initial Conditions: temperature range 25-30°C and pH=7.

In the Temkin model, regarding the possible interaction between adsorbed-adsorbed and adsorbed-adsorbent types, adsorption heat of all molecules in the layer decreases linearly with coverage quantity. Temkin adsorption isotherm linear form is explained as:

$$q_e = Bl_nA + Bl_nC_e \quad (7)$$

where B=RT/b, R=gases general constant=8.314 kJ/mol and T is absolute temperature in Kelvin. Constant A (L/g) is balance bond corresponding with bond energy maximum and constant B is adsorption heat (Bayramoğlu *et al.* 2006, Carlos 2004).

In order to calculate average biosorption free energy that implies available mechanisms in biosorption (e.g. physical or chemical), the Dubinin-Radushkovich (D-R) isotherm is used the linear form of which is as follows (Dubinin 1960):

$$l_n q_e = l_n q_m - \beta \varepsilon^2 \quad (8)$$

Here, q_e and q_m are the quantity of adsorbed material in the adsorbent and biosorption capacity maximum respectively (mol/g), β is activity coefficient that depends on biosorption energy average and it is taken from l_n q_e linear diagram slope against ε^2:

$$\varepsilon = R_t l_n(1 + 1/C_e) \quad (9)$$

$$E = \frac{1}{\sqrt{2\beta}} \quad (10)$$

E is average biosorption energy (kJ/mol) that shows available mechanism type. If E is in range of 8-16 kJ/mol the biosorption is chemical, but if it is less than 8 kJ/mol biosorption is physical. Figure 9 shows Dubinin-Radushkovich (D-R) isotherm of three acidic dyes on living Azolla.

According to the results, biosorption of AR14 is chemical and that of AY17 and AB25 is physical (Table 3).

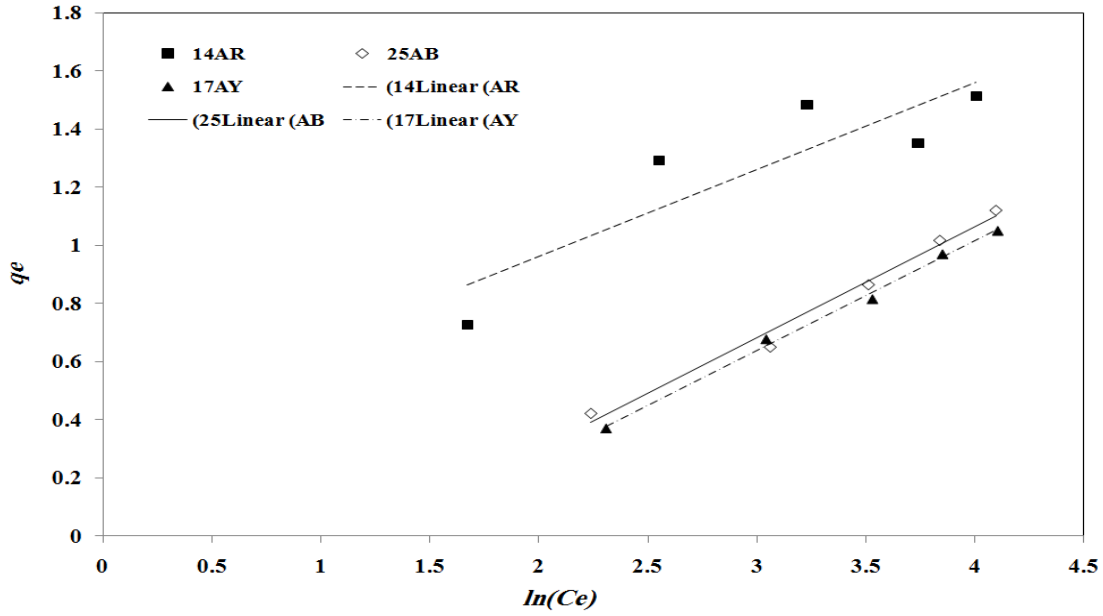

Fig. 8. Temkin isotherm of AR14, AB25 and AY17 on *A. filliculoides*. Initial Conditions: temperature range 25-30°C and pH=7.

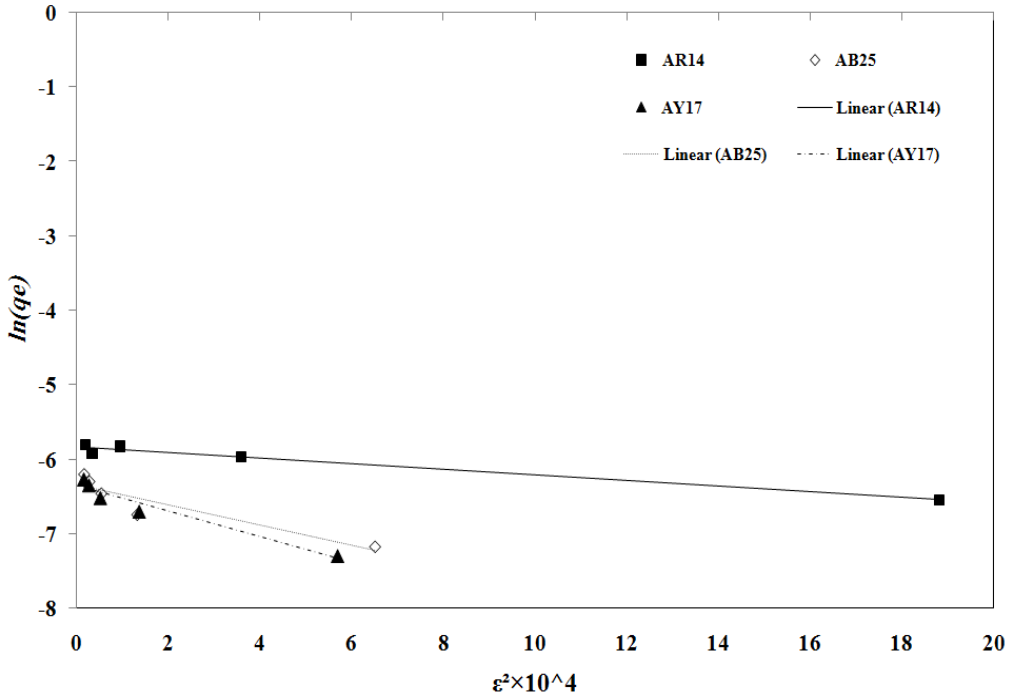

Fig. 9. Dubinin-Radushkovich isotherm of AR14, AB25 and AY17 on the *A. filliculoides*. Initial Conditions: temperature range 25-30°C and pH=7.

Table 3. Langmuir, Freundlich, Tamkin, and Dubinin-Radushkovich model parameters for adsorption of three acidic dyes by *Azolla*.

	Langmuir				Freundlich			Tamkin		Dubinin-Radushkovich		
Dyes	R^2	q_m(mg/g)	K_L(L/mg)	R_L	R^2	K_f(L/g)	n	R^2	b(J/mol)	R^2	E(kJ/mol)	q_m(mg/g)
AR14	0.994	1.79	2.31	0.00628	0.76	0.52	0.28	0.78	41985.67	0.97	13.51	0.003
AY17	0.999	1.73	12.09	0.00606	0.97	0.10	0.57	0.99	35987.71	0.94	2.90	0.002
AB25	0.986	1.50	10.82	0.00606	0.99	0.12	0.53	0.98	65778	0.85	3.68	0.002

CONCLUSION

It was shown that *A.filliculoides* a dominated species in the northern areas of Iran can be used as a cheap and available biosorbent in complementary stages of infiltration of aqueous solutions. Living *Azolla* removed AR14, AB25, and AY17 acidic dyes from aqueous solutions easily and cheaply. Dye removal depends on initial concentration, contact time, and dye type. The highest adsorption occurs in concentration of 15 mg/l for all dyes studied and the pattern of adsorption is as follows:
AR14=64.52%>AB25=37.53%>AY17=32.98 %. It was found that in Kinetic study of pseudo first order and pseudo second order reaction, in a given quantity of adsorbent, all three dyes followed pseudo second order reaction. In general, a review of four isotherms of Langmuir, Freundlich, Dubinin-Radushkovich, and Temkin in fixed reactor for three dyes, and based on the available conditions and experiment results for AR14 dye it can be concluded that biosorption of AR14 is optimum in the Dubinin-Radushkovich adsorption isotherm. This means that adsorption process is not a simple physical

phenomenon and it has a chemical structure. It may be assumed that in such conditions, the planet considers a contaminant as nourishment and changes it during a biological process that can result in the permanent removal of organic dye material, although, the possible damage of the plant should be considered. If such damages occur, the toxic contaminant will be removed from nature and there will not be any anxiety about biosorbent excretion after using. Temkin adsorption isotherm was suggested for AY17 and the best explanation to express AB25 adsorption is the Freundlich isotherm.

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جذب زیستی محلول آبی شبیه سازی شده رنگزاهای اسیدی با استفاده از آزولا گونه *Filliculoides*

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(تاریخ دریافت:۲۷ /۹۲/۱ - تاریخ پذیرش: ۹۲/۶/۱۴)

چکیده

جذب زیستی رنگزاهای اسیدی با استفاده از سرخس زنده آزولا گونه *Filiculodes*، در یک سیستم ناپیوسته در ۵غلظت اولیه مختلف۱۵، ۳۰، ۴۵، ۶۰ و ۷۵ mg/l در مدت زمان ۳۳۶ ساعت مورد بررسی قرار گرفت. پارامترهای رنگزا، غلظت اولیه رنگزا و زمان تماس در محدوده دمایی $^{\circ}C$ ۳۰–۲۵ و pH= ۷ مورد مطالعه قرار گرفت. نشان داده شد که با افزایش غلظت اولیه رنگزا و زمان تماس، میزان برداشت رنگزا توسط جاذب کاهش می یابد. بیشترین ظرفیت برداشت رنگزا در غلظت ۱۵mg/L ، ۶۴،۵۲٪ ، ۳۷،۵۳٪ و ۳۲،۹۸٪ به ترتیب برای رنگزاهای اسیدی قرمز۱۴ ، آبی۲۵ و زرد۱۷ گزارش شد. مدل های ایزوترم جذب لانگمویر، فروندلیش، دابینین-رادوشکویچ و تمپکین در غلظت های مختلف مورد آنالیز قرار گرفت. بررسی مدل های سنتیکی شبه مرتبه اول و شبه مرتبه دوم نشان داد که مدل سنتیکی شبه درجه دوم مدل مناسبی برای جذب زیستی رنگزای مذکور است.

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