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Removal of Orange 7 Dye from Wastewater Used by Natural Adsorbent of *Moringa Oleifera* Seeds

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Abstract Removal of Orange 7 from wastewater using natural adsorbent of Moringa *Oleifera* seeds were investigated. The effects of some operational parameters such as pH, the amount of biosorbent, initial dye concentration and temperature were examined. The present study revealed optimum conditions for the removal process which included dose of biosorbent (0.4 g), initial dye concentration (20 mg/L), initial pH (6) and temperature (25°C). The Langmuir and Freundich isotherms were applied for describing the biosorption equilibrium. The process was represented by the Freundich isotherm with a correlation coefficient of 0.94. The first-order, second-order and intra-particle diffusion kinetic models were implemented for demonstrating the biosorption mechanism and, as a result, intra-particle diffusion kinetics fitted best to the experimental data.

Keywords Wastewater, Orange 7, Moringa Oleifera, Biosorption

1. Introduction

Among waters, dye wastewater from dyestuff and textile industries is one the most difficult ones to be treated[1]. By absorbing sunlight, dyes can prevent photosynthesis in aqueous ecosystems. Sulfur dyes are harmful to aqueous organisms, since they can rapidly reduce the oxygen content of water[2]. Many studies have been done on physico-chemical methods in order to remove color from textile effluent. These methods include coagulation, oxidization, ultra filtration, electro-chemical, adsorption and combined electro-chemical and adsorption techniques[1-16].

Generally speaking, synthetic dyes' complex aromatic structures make them stable and difficult to biodegrade[17]. Adsorption has some specific benefits, which include treating high-flow wastewaters with good final quality and without producing any harmful substances[2].

Acid Orange 7 [*p*-(2-hydroxy-1-naphthylazo) benzene sulfonic acid, as a popular water-soluble dye, is used for dyeing a variety of materials such as nylon, aluminum, detergents, cosmetics, wool and silk. Similar to other azo dyes, it is usually disposed in industrial wastewater, which can be considered a serious health threat to humans. It is highly toxic; thus its ingestion can irritate eye, skin, mucous membrane and upper respiratory tract; severe headaches, nausea, water-borne diseases such as dermatitis and loss of bone marrow leading to anemia are also among its harmful consequences. Since it is carcinogenic in nature and leads to tumors, it can be fatal to consume it. Current research has found that the electron-with drawing character of the azo group is the main cause of its chronic toxicity, which develops an electron deficiency and is reduced to carcinogenic amino compounds[12].

Since Acid Orange 7 is toxic and carcinogenic by nature, its removal has been attempted by biodegradation and photosensitization on TiO2 particles. Unfortunately, metabolic intermediates produced after its biodegradation are carcinogenic and photosensitization produces 1,2 - naphthoquinone and phthalic acid during degradation, which are also harmful. Additionally, Biofilm systems have been applied for aerobic nitrification, anoxic denitrification and anaerobic digestionin order to decolorize Acid Orange7[12].

Therefore, it can be concluded that despite its high toxicity, thus far, few attempts have been made to remove Acid Orange 7 from aqueous solutions. Even common chemical/physical methods such as coagulation, flocculation, ozonation, reverse osmosis, electrolysis, ultra-chemical filtration and chemical treatments have not been attempted, which can be attributed to either high solubility of the dye in water or possibility of generating toxic intermediates/products during the process. Furthermore, adsorption, which is influenced by the chemical properties and structure of the dye and also do not produce any toxic products, is deliberately used for the eradication of Acid Orange 7 from waste wate[12].

One of the natural coagulants is the water -soluble extract of the dry seeds of Moringa oleifera, which is a tropical plant from the family of Moringaceae. So far, up to fourteen different species have been identified for it. Moringa oleifera is the most widespread species and grows quickly at low altitudes of the whole tropical belt including arid zones[18].

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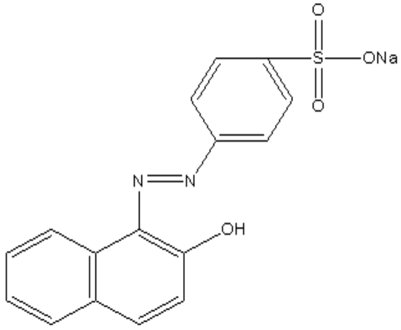
It is drought-tolerant, has nutritional, medicinal and water cleaning attributes and its leaves, flowers, fruits and roots are locally used as food articles. Because of its medical and therapeutic properties, it is applied as a cure for different ailments and diseases and physiological disorders in eastern allopathic medicine.

The present investigation aims at examining the sorption potential of SMOS in order to remove organics from aquous media[19].

Table 1-1. Chemical stucture and general properties of Acid Orange 7.

Characteristic Information
Structure (CI 15510) ~85% purity

Formula
$C_{16}H_{11}N_2NaO_4S$
Molecular weight
350.33
Molecular volume ($A^{\circ 3}$ molecule $^{-1}$)
231.95
Molecular surface ($A^{\circ 2}$)
279.02
Molecular
1.24×0.68×0.22
dimensions (nm)
1.27×0.7×0.33
1.36 ×0.73×0.23
λ_{max} (nm)
485
Natural pH in deionised water
6.1

M. oleifera prefers hot, semi-arid regions (annual rainfall 250–1500mm); thus it grows widely throughout the tropics. Furthermore, it is suitable for lowland cultivation at altitudes less than 600m. This tree is tolerant of light frosts and slightly alkaline soils up to pH 9. Its seeds which posess effective coagulation properties are quite efficient in reducing turbidity, micro-organisms of wastewaters and also in sludge conditioning. The active agents of coagulation are dimeric cationic proteins with the molecular weight of approximately 13 (kDa) with an isoelectric point between 10 and 11[20].

Coagulation's active component in *Moringa oleifera* is supposed to remove the suspended solids in wastewater by the sweep coagulation mechanism[20].

Moringa oleifera, as a tropical plant who can survive heat, desiccating dryness and destitute soils, is abundantly available throughout the year. Its various parts are used for innumerable pharmacological properties viz. antimicrobial activity, analgesic activity and antihypertensive activity. Coagulating behavior of its seed powder has been studied for different aspects of water treatment like turbidity, alkalinity, total dissolved solids and hardness[21].

2. Materials and Methods

2.1. Instruments

1-METTLER balance with accuracy 0.0001 g
2-EDT Instruments pH meter
3-Sigma 301 centrifuge
4-GFL 3005 Shaker
5- Heidolph unimax 1010 Shaker Incubator
6- unico 4802 UV-Vis spectrophotometer

2.2. Preparing Acid Orange 7 and Biosorption Capacity

The Acid Orange 7 used in this study was obtained from Merck and Stock AO7 solution (500 mg/L) was prepared by dissolving 0.05 g of AO7 in 10 mL of double-distilled water and diluting quantitatively to 100 mL. AO7 solutions of different concentrations (2.5–50mg/L) were prepared by the adequate dilution of the stock solution to 250 mL. The applied glassware was rinsed several times with double-distilled water before being used. The absorbance of standard AO7solutions was measured and plotted against the concentration.

In brief, batch experiments were carried out as functions of biomass dose (0.05–1.0g), dye concentration (2.5–50 mg/L), pH (3.0–9.0), temperature (10–30^{0}C) and contact time (1–60 min). Known solutions of AO7 were poured into separate 100ml Erlenmeyer flasks. After pH adjustment, a known quantity of biosorbent was added and, at last, dye bearing suspensions were kept on the shaker (250 RPM) for 60 minutes at the temperature. The samples were taken from 0 to 60 minutes and the biomass was separated from the solutions by centrifuge and was subjected to dye estimation by UV/Vis spectrophotometry. Dye concentrations were recorded before and after biosorption. Removal efficiency was computed by the following equation[19]:

$$\%R = \frac{C_0 - C_t}{C_0} \times 100 \tag{1}$$

where C_0 (mg/L) and C_t (mg/L) are initial and residual concentrations of dye, respectively.

2.3. Analytical Methods

Maximum absorbance wavelength of AO7 was determined experimentally. The concentration of AO7 in the solutions before and after the equilibrium was determined by UV/Vis spectrophotometry at a λ_{max} of 485 nm, using a unico 4802 UV/Vis spectrophotometer. pH of the solutions was measured by a EDT Instruments pH meter.NaOH and HCl were obtained from Merck, Germany. Dilute solutions of NaOH and HCl were prepared and used for adjusting the pH.All the chemicals used in this study were of analytical reagent grade.

3. Results and Discussion

To acquire a comprehensive understanding of biosorption of AO7 onto SMOS, a meticulous investigation of effective parameters was carried out. Prior knowledge on the optimal conditions provides a better design and modeling which are, therefore, of vital importance for exploiting the potentiality of the SMOS biosorbent[19]. The biosorption of AO7 onto SMOS is influenced by the sorbent dose, concentration of sorbate, initial pH, reaction temperature and contact time.

3.1. Effect of Biosorbent Dose

A number of investigations were carried out by varying the amount of SMOS from 0.05 to 1.0 g at the fixed initial dye concentration of 20 mg/L, pH of 7 and room temperature of 25±1 °C. These studies showed an increase in biosorption with the increase in the dose of biosorbent (Fig.3-1). Optimum biosorbent dose was found to be 0.4 g.

3.2. Effect of Initial Dye Concentration

Fig.3-2, show the results of experiments conducted in the range of dye concentration (2.5–50mg/L) at the constant biosorbent dose (0.2 g), pH (7) and temperature (25^0C). The obtained results reveal that percent removal of dye depends on the initial dye concentration. The obtained optimum dye concentration was 20 mg/L. These observations can be explained by the fact that sufficient biosorption sites are available for accommodating an increasing number of dye molecules. In other words, the number of molecules available for sorbing onto the sorbent surface is relatively lower in comparison with the ones available for biosorption sites[19,24]. Higher dye concentrations are capable of affecting the chemical equilibrium between dye molecules in the liquid phase and the ones adsorbed onto the biosorbent surface resulting in a further biosorption process. Moreover, this sorption behavior can be attributed to higher chance of effective interaction between adsorbate molecules and biosorbent surface as the concentration of dye increases. These findings demonstrate the applicability and efficacy of SMOS for the removal of AO7 dye from the aqueous media.

3.3. Effect of Initial pH

Effect of initial pH on the biosorption process was monitored in the pH range of 3 and 9 at the biosorbent dose of 0.20 g, initial dye concentration of 20 mg/L and temperature of 25±1 ^{0}C (Fig.3-3). The most appreciable biosorption was found in pH 6. Variation in the removal of AO7 with respect to pH can be explained by considering surface properties of biosorbent material and ionization state of AO7[24]. Protaineous amino acids are mainly considered as the active functional compounds present in shelled *Moringa Oleifera* seeds. These amino acids have a variety of structurally pH-dependent properties and the ability to generate negatively charged atmosphere.

3.4. Effect of Temperature

Effect of temperature over the ranges 10, 25and 30^0C on AO7 biosorption was examined under the conditions of 0.20 g biosorbent dose, 20 mg/L dye concentration and the initial pH of 7. The biosorption was fast at the beginning of the experiment. The sensitivity of biosorption process toward temperature is presented in Fig.3-4. This figure exhibits a small fluctuation in the percent sorption of dye as temperature increases from 10^0C to 25^0C and decreases from 25^0C to 30^0C.

3.5. Effect of Contact Time

The variation in the sorption of AO7 as a function of contact time is presented in Fig. 3-5. The results obtained under constant conditions of 0.20 g biosorbent dose, 20 mg/L initial dye concentration, pH of 7 and room temperature of 25±1 ^{0}C reveal that the percent removal of AO7 rises as time proceeds. The sorption is quite rapid initially, then slows down gradually and, finally, levels out and reaches a maximum dye removal. This trend may be attributed to the elevation of contact between the sorbate and sorbent during the experiment. Interestingly, near 60% of the ultimate biosorption occurred within first 5 minutes of contact. Bearing in mind the time restraints in the practice, it is recommended that SMOS be capable of being used as an effective pretreatment technology in the dye wastewater treatment.

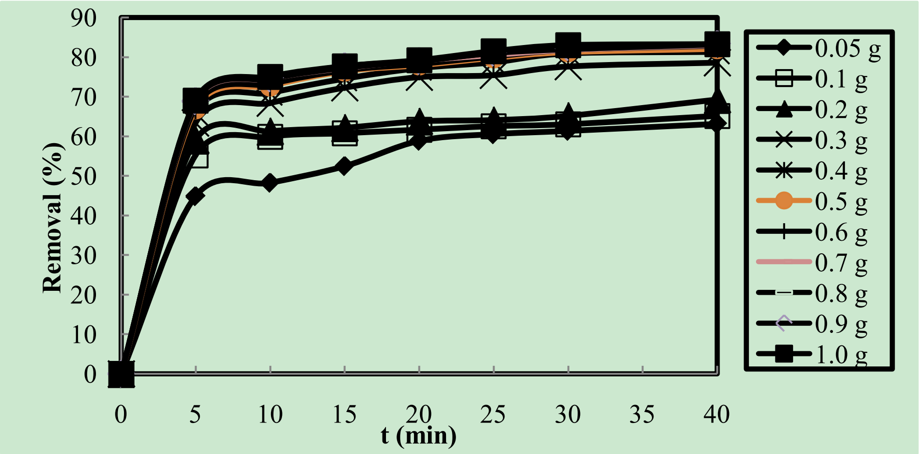


Figure 3-1. Effect of biosorbent dose on the removal of AO7 using SMOS at C_0=20 mg/L, pH=7 and T=25 ^{0}C.

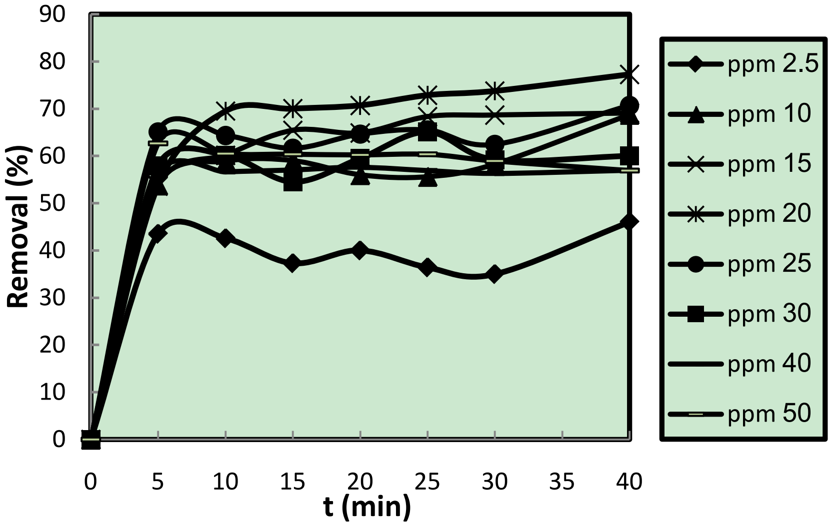


Figure 3-2. Effect of initial dye concentration on the biosorption behavior of AO7 on SMOS at m=0.2g, pH=7 and T=25 ^{0}C.

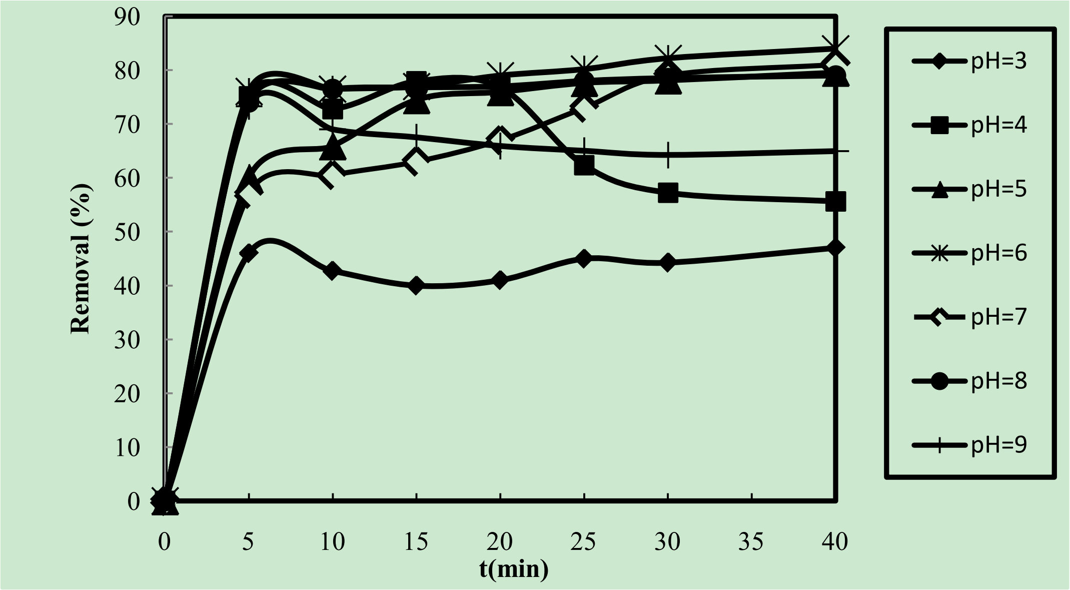


Figure 3-3. Effect of varying initial pH on the removal of AO7 by SMOS at m=0.20 g, C_0=20 mg/L and T=25 ^{0}C.

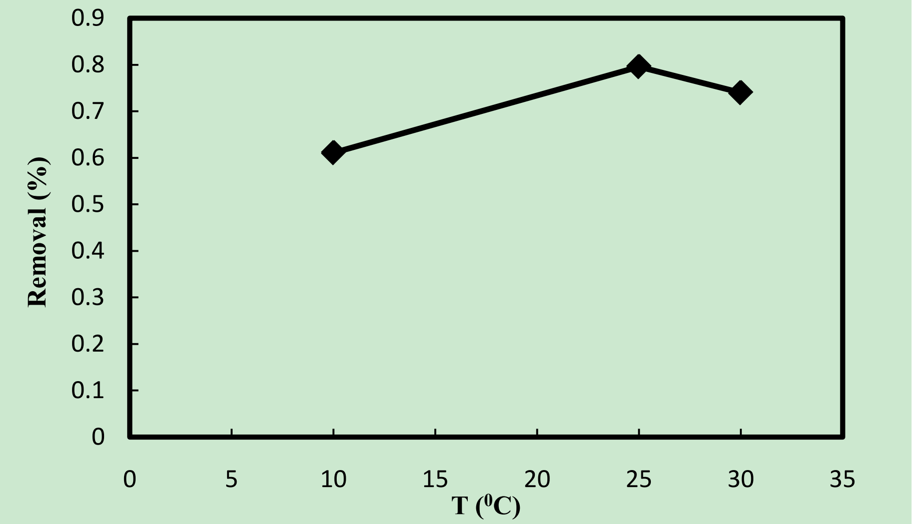


Figure 3-4. Effect of different temperatures on the biosorption of AO7 by SMOS at m=0.20 g, C_0=20 mg/L and pH=7.

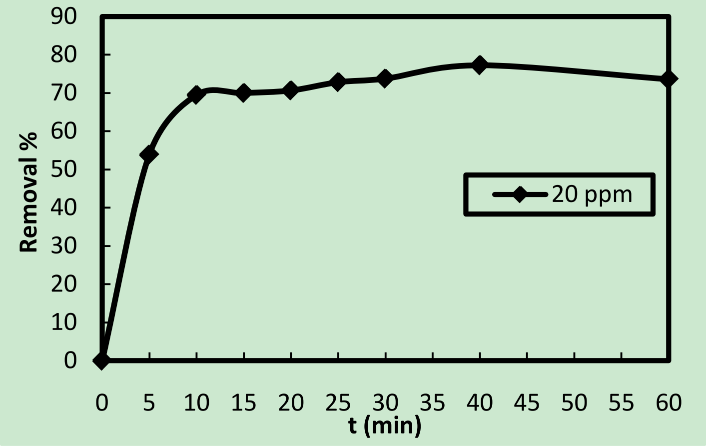


Figure 3-5. Effect of contact time on the removal of AB9 using SMOS at m=0.40 g, C_0=20 mg/L, pH=7 and T= 25 ^{0}C.

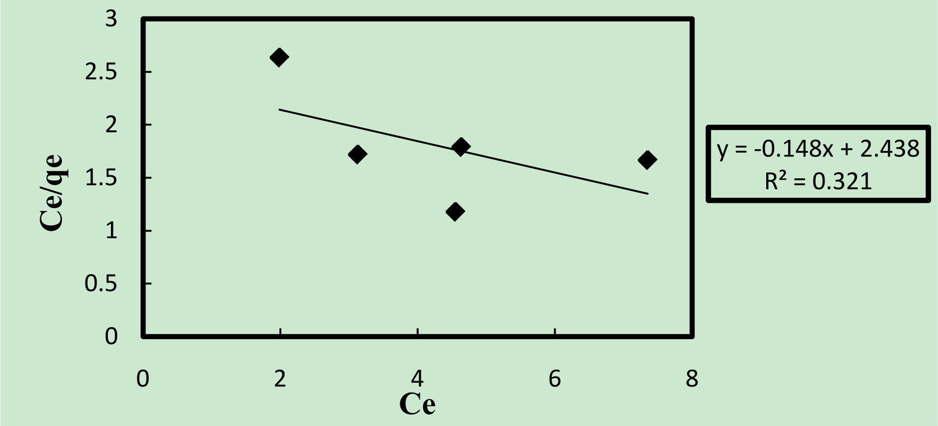


Figure 3-6. Langmuir isotherm chart.

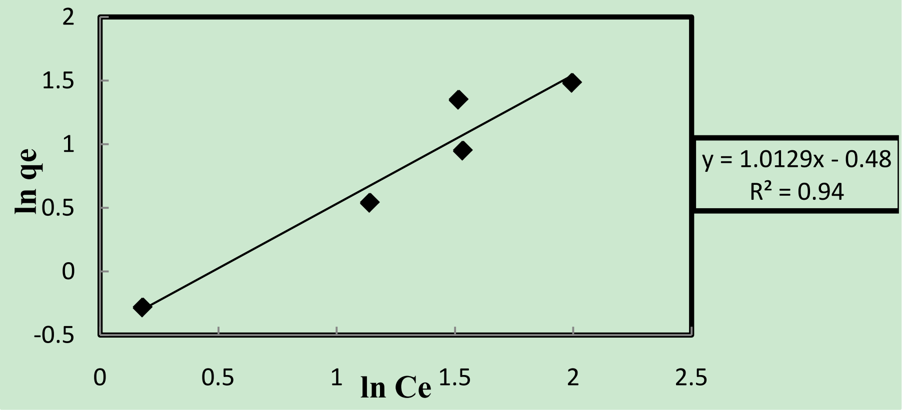


Figure 3-7. The Freundlich isotherm's chart.

3.6. Adsorption Isotherm

In order to understand the adsorption process, the Langmuir and Freundlich isotherms, Figs. 3-6 and 3-7, respectively, were used to represent the equilibrium relationship for different initial Acid Orange 7 concentrations' experiments. The Langmuir equation assumes that adsorption is limited to monolayer; its linearized form can be represented as:

$$\frac{C_e}{q_e} = \frac{1}{q_{\max}} C_e + \frac{1}{b \cdot q_{\max}} \quad (2)$$

where qe is the metal ion sorbed (mg/g), Ce equilibrium concentration of dye solution and q_{max} and b Langmuir constants.

The Freundlich equation is an empirical relationship which describes the adsorption of the solutes from a liquid to solid surface. Linearized form of the Freundlich equation is as follows:

$$\ln q_e = \frac{1}{n} \ln C_e + \ln K_f \quad (3)$$

where qe is the metal ion sorbed (mg/g), Ce equilibrium concentration of metal ion solution (mg/L) and K_f and 1/n constants.

As indicated in Table 3-2, the coefficients of determination (R^2) of the Freundlich model are greater than 0.9 and close to one, which indicates that the Freundlich model adequately describes the experimental data of these dye biosorption experiments[21].

Table3-1. Results of Langmuir isotherm.

C_0	C_e	q_e	C_e/q_e
ppm 5	1.985 ppm	0.75375	2.633
ppm 10	3.126 ppm	1.7185	1.7185
15 ppm	4.6335 ppm	2.59	1.7889
20 ppm	4.55 ppm	3.8625	1.1781
ppm 25	7.35 ppm	4.4125	1.6657

Table 3-2. Constants of Langmuir isotherm.

q_{max}	b	R^2
-6.75	-0.06	0.3217

Table 3-3. Results of the Freundlich isotherm.

C_0	C_e	q_e	ln q_e	ln C_e
5 ppm	1.198 ppm	0.75375	-0.282	0.18
10 ppm	3.126 ppm	1.545	0.435	1.34
15 ppm	4.633 ppm	2.59	0.951	1.533
20 ppm	4.55 ppm	3.8625	1.3513	1.5151
25 ppm	7.35 ppm	4.4125	1.484	1.9947

Table 3-4. Constants of Freundlich isotherm.

K_f	n	R^2
0.61878	0.987	0.94

3.7. Kinetic Studies

In order to evaluate the kinetics of AO7 biosorption onto SMOS, three models were applied, i.e. first-order model, second-order model and intra-particle diffusion model. The linear form of the first-order model can be formulated as:

$$\ln C_t - \ln C_0 = -k_1 t \tag{4}$$

The second-order linear equation can be written as:

$$\frac{1}{C_t} - \frac{1}{C_0} = k_2 t \tag{5}$$

Film resistance plays an important role in the overall transport of the solute. In order to estimate the intra-particle diffusion rate, Morris–Weber equation was also subjected to sorption kinetic data in the following way[25]:

$$q_t = k_i t^{1/2} \tag{6}$$

where q_t (mg/g) is the amount of dye adsorbed per unit mass of biosorbent at time t, C_0 (mg/L) represents initial concentration of adsorbate in the solution, C_t (mg/L) is the concentration of adsorbate in the solution at time t, time is represented by t (min) and k_1 ($mg.L^{-1}.min^{-1}$), k_2($mg^{-1}.L.min^{-1}$) and k_i ($mg.g^{-1}.min^{-1/2}$) stand for rate constants of the first-order, second-order and intra-particle diffusion models, respectively. q_t is obtained from the following equation:

$$q_t = \frac{(C_0 - C_t)V}{m} \tag{7}$$

where V (L) represents the volume of solution and m (g) is the biosorbent mass. The applicability of each model was studied by plotting ($\ln C_t - \ln C_0$) against t, ($1/C_t - 1/C_0$) against t and q_t against $t^{1/2}$ for the first-order, second order, and intra-particle diffusion models, respectively.

If q_t is plotted against $t^{1/2}$, the sorption data yields a straight line. This observation indicates that kinetics of AO7 dye sorption computed from the slope of linear plots onto SMOS surface follows the linearity in the range of 5–40 min, as shown in Fig. 3-8[25].

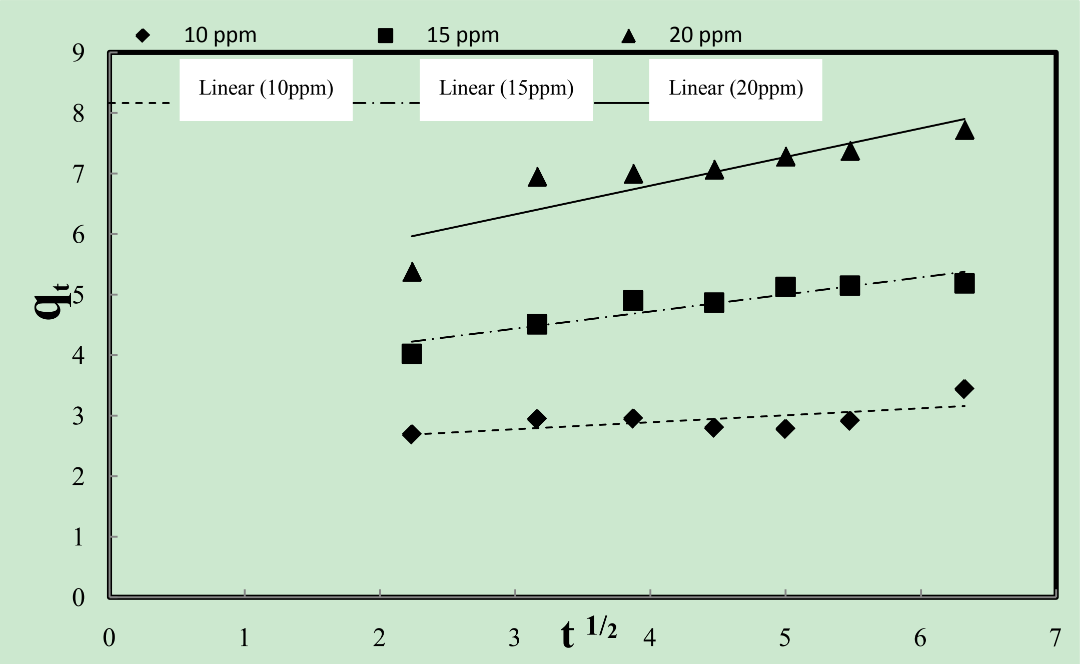


Figure 3-8. The intra-particle diffusion kinetic plots for the biosorption of AO7 dye by SMOS at different initial dye concentrations, 0.2 g biosorbent dose, pH 7 and 25^{0}C.

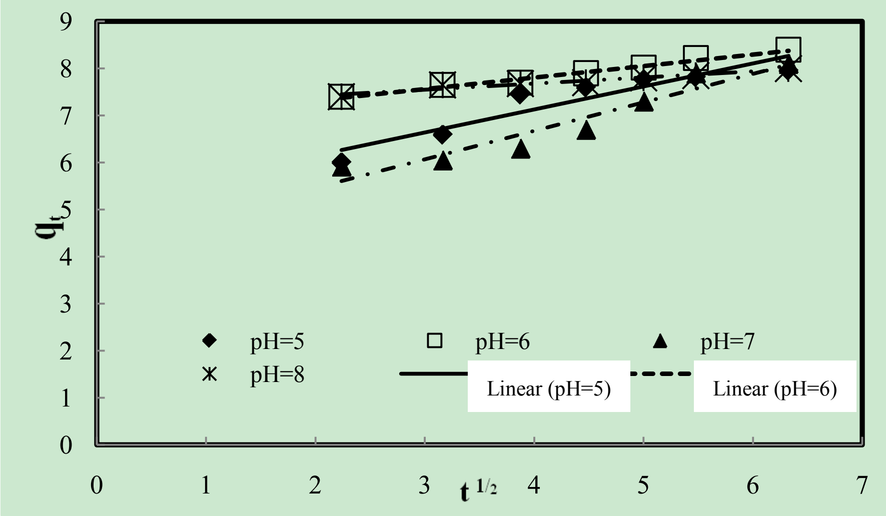


Figure 3-9. The intra-particle diffusion kinetic plots for the biosorption of AO7 dye by SMOS at different initial pH, 20 mg/L initial dye concentration, 0.2 g biosorbent dose and 25 ^{0}C.

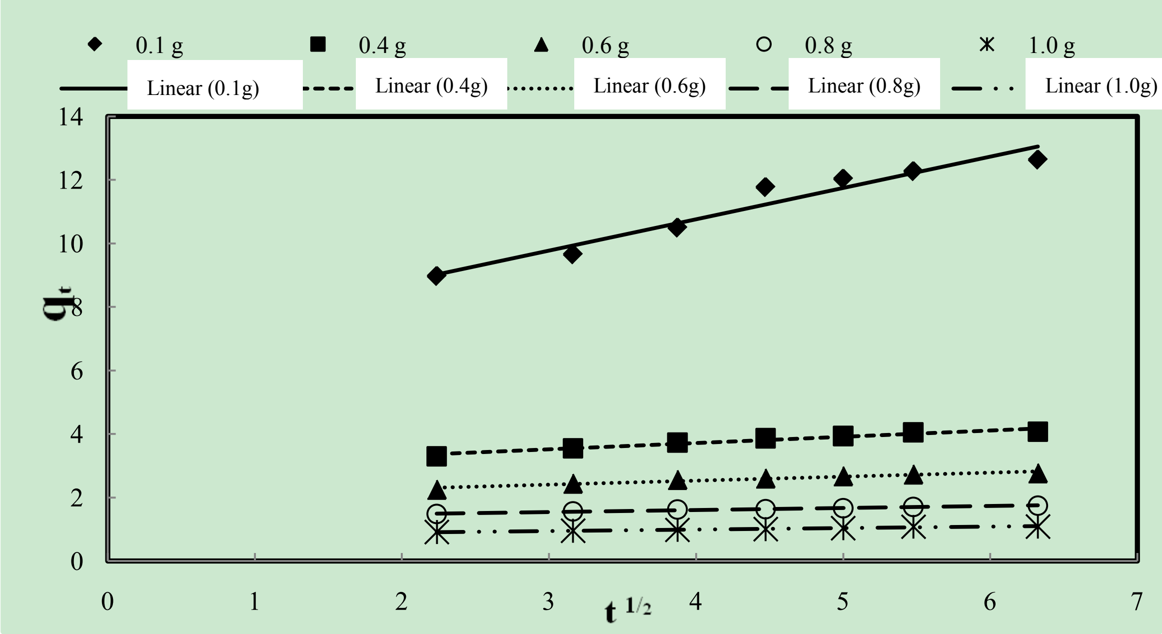


Figure 3-10. The intra-particle diffusion kinetic plots for biosorption of AO7 dye by SMOS at different biosorbent doses, 20 mg/L initial dye concentration, pH 7 and 25 ^{0}C.

Biosorption kinetics was monitored very carefully in various biosorbent doses, initial pollutant concentrations and initial pH. Correlation coefficients and rate constants obtained from the slopes of the corresponding linear plots are tabulated in Tables 3-5,3-6 and 3-7. The results suggest that the sorption phenomenon follows kinetics intra-particle diffusion at relatively low temperature levels. Correlation coefficients were found closer to unity in the model's intra-particle diffusion in comparison with the first-order and second-order models in the whole initial dye concentration range. Therefore, for different initial dye concentrations, the kinetics could be explained by the intra-particle diffusion kinetic model. Intra-particle diffusion kinetics also fitted best to the experimental data at both high biosorbent doses and little acidic pH.

Table 3-5. Kinetics of biosorption of AO7 dye by shelled *Moringa Oleifera* seeds. Rate constant and correlation coefficients for three kinetic models at different biosorbent doses.

	First order		Second order		Intra-particle diffusion	
m(g)	K_1	R^2	K_2	R^2	K_i	R^2
0.05	0.0049	0.7348	0,0006	0.7032	0.7247	0.8358
0.1	0.0126	0.8052	0.0014	0.9274	0.9849	0.9454
0.2	0.0059	0.8395	0.0008	0.8674	0.2083	0.9019
0.3	0.014	0.7452	0.0029	0.9315	0.2519	0.9441
0.4	0.0176	0.9113	0.0036	0.945	0.1968	0.9517
0.5	0.0172	0.90	0.0037	0.9448	0.1494	0.9352
0.6	0.0181	0.93	0.004	0.9634	0.1257	0.9521
0.7	0.0172	0.9161	0.0038	0.9582	0.0979	0.9408
0.8	0.0082	0.9488	0.0012	0.9637	0.0634	0.9748
0.9	0.0074	0.9461	0.001	0.9669	0.0553	0.9485
1.0	0.0052	0.9456	0.0005	0.9521	0.0456	0.9702

Table 3-6. Kinetics of biosorption of AO7 dye by shelled *Moringa Oleifera* seeds (Rate constant and correlation coefficients for three kinetic models at different initial dye concentrations).

	First order		Second order		Intra-particle diffusion	
C_0 (ppm)	K_1	R^2	K_2	R^2	K_i	R^2
2.5	0.0002	0.0009	0.0002	0.0025	0.003	0.0095
5	0.0016	0.0175	0.0005	0.0061	0.022	0.047
10	0.0077	0.5095	0.0021	0.5181	0.1154	0.4393
15	0.011	0.7946	0.002	0.825	0.282	0.8698
20	0.016	0.7731	0.0026	0.85	0.472	0.7759
25	0.0041	0.3195	0.0005	0.3358	0.1159	0.197
30	0.0022	0.1034	0.0002	0.1084	0.1197	0.1251
40	0.0003	0.1063	0.0002	0.079	0.029	0.1568
50	0.0033	0.8732	0.0002	0.5367	0.286	0.8404

Table 3-7. Kinetics of biosorption of AO7 dye by shelled *Moringa Oleifera* seeds (Rate constant and correlation coefficients for three kinetic models at different initial pH).

	First order		Second order		Intra-particle diffusion	
pH	K_1	R^2	K_2	R^2	K_i	R^2
3	0.0015	0.1539	0.0001	0.1366	0.049	0.0701
4	0.0226	0.7539	0.0032	0.6459	0.06005	0.7061
5	0.0187	0.8218	0.0033	0.8723	0.4895	0.8739
6	0.0066	0.684	0.0028	0.9561	0.2454	0.9808
7	0.0233	0.9048	0.0046	0.9355	0.6062	0.9191
8	0.0055	0.8724	0.0009	0.8897	0.1304	0.9446
9	0.004	0.6583	0.0011	0.6733	0.02094	0.8448

4. Conclusions

Successful application of shelled *Moringa Oleifera* Lamarck seeds as a biosorbent at the laboratory scale introduces an effective, feasible and environmentally-friendly method for removing C.I. Acid Orange 7 from the aqueous systems. The biosorption process was found to be dependent on biosorbent dose, initial dye concentration, initial pH, temperature and contact time. Under the studied conditions, biosorption equilibrium data followed Freundlich isotherm more favorably than Langmuir isotherm. The values of $1/n$ and K_f from Freundlich and Langmuir isotherms confirmed the effectiveness of SMOS biosorbent for removing AO7 dye at low concentrations. Batch biosorption capacity tests have shown that intra-particle diffusion kinetics can be applied for describing the biosorption mechanism in certain conditions, whereas the first-order and second-order models with little differences did not fit the experimental data. The removal of Acid Orange 7 was observed to be rapid at the primary stages of biosorption process. The main conclusion is that the shelled *Moringa Oleifera* seeds demonstrate an adequate potential for being exploited as an economically viable and indigenous pretreatment approach in modern technology of wastewater treatment. Future investigations should be conducted with a view to selectively separating the present dye contaminants, regenerating the exhausted biomass, recovering the sorbed dye and designing to continuous dye treatment systems. These studies can further ameliorate the economic aspects of dye wastewater treatment.

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