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Kinetic studies of Safranin-O removal from Aqueous Solutions using Pineapple Peels

M. Yusuf[1], F.M. Elfghi[1,2]*, S. K. Mallak[3]

[1]*Malaysia Japan International Institute of Technology, Department of Environmental Engineering and Green Technology, Jalan Semarak, 54100 Kaula Lumpur, Malaysia*

[2]*Chemical Reaction Engineering Group, Faculty of Chemical and Natural Resources Engineering, University Technology Malaysia, 81310 UTM Skudai*

[3]*Department of Environmental Management, Faculty of Environmental studies, 43400 Universiti Putra Malaysia, Selangor, Malaysia*

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A B S T R A C T

This study aimed to investigate how safranin-O can be removed from aqueous solutions by adsorption on pineapple peels. The effect of solution pH, initial dye concentration, contact time and adsorbent dose were studied. The optimum adsorption capacity of 26.08 mg/g was achieved under the experimental condition of pH, temperature and contact time of 6, 293K and 80 min, respectively. Also further analysis revealed that 93.24% of safranin-O was significantly removed at 120 mg/L dye concentration in 80 minutes contact time. From the result of the isotherm studies, it was revealed that the equilibrium data is well fitted to Freundlich model while the adsorption kinetic data showed that the adsorption process was well described by the pseudo-second order kinetic model. Finally, it can be deduced that pineapple peels had a great potential in adsorbing and removal of safranin-o from aqueous solution.

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INTRODUCTION[1]

Industrialization play an important role in terms of providing human's need and improving their life style. However the negative consequences of industrial activities such as paper, printing, textile and pharmaceutical manufacturing due to existence of hazardous chemicals and dyes in their effluents cannot be ignored [1, 2].

Deterioration of both flora and fauna's normal function has resulted from releasing untreated industrial wastewater to the water body [3]. Safranin-O, a cationic dye is one of the hazardous contaminant that has been found in the effluents of pharmaceutical and textile manufacturing industries. It was reported that this contaminant is harmful for the human's health due to its negative impact on the skin and digestive and respiratory systems [4]. Industrial activities are increasing all over the world. But, the drawback of this growth, relates to activities involving with dyes production. The accurate data regarding the amount of dyes production all over the world was not released. Currently it is estimated that, more than 10,000 are commercially available with about 0.7 million tons production per year [5]. Exact data on the quantity of dyes discharged into environment are also not available. It is assumed that about 1–2% loss of dyes in production and about 10–15% loss in application are fair estimates [6].

Arguably, dyes have adverse effect on water bodies as they are visible pollutants. The presence of color in water bodies reduces light penetration which in turn upsets the biological metabolism process [7]. Disposal of dye wastewater without proper treatment destructs the aquatic communities present in the ecosystem [8].

The conventional method for removal of dyes from wastewater includes coagulation/flocculation, oxidation or ozonation, membrane separation precipitation, ion-exchange, reverse osmosis, complexation/sequestration and activated carbon [9, 10]. But the major drawback,

* Corresponding author: F.M. Elfghi
E-mail: felfghi@gmail.com; Tel: +60189682307

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especially for developing countries is the high cost for recycling/regeneration ability. Similarly, the application of the above mentioned methods in the treatment of wastewater may not be economically viable. Therefore, biosorption technique could be efficient in such cases [11].

Agricultural and fruit wastes

In many developing countries agricultural productions have a considerable contribution in human's survival [12]. But over the past decades land disposal remains as the major method for handling the wastes generate from agricultural activities. Generated wastes from agricultural activities, fruit and vegetable processing have negative impacts on the environment. For instance in food industries, huge quantity of byproducts generate from cannery processing includes peeling, slicing and sorting of fruit products. Therefore lack of appropriate method for managing these waste and illegal dumping; have been attributed to environmental pollution and issues such as cancer and acute respiratory diseases [13].

In some tropical countries such as Thailand, canned fruit formed one of the main export products. It was reported that about 523 ton of pine apple provided the material for canned production in 2007 [14]. Considerable amount of solid wastes generated through processing of pine apple canned products such as pineapple peels that comprising approximately 28% of fresh pineapple weight [14].

Thus, pineapple peels can be reused and obtained for several purposes such as pharmaceutical purposes and effluent treatment application [15, 16] .

Recently, there are many researches on cheap, available agricultural or plant-based material such as tea (*Camellia sinensis* L.) seed shells, pea shells (*Pisum sativum*), Casuarina Equisetifolia needle, alfa grass, neem leaf powder, corn husk and papaya stems have been used as a sorbent for the removal of various dye effluents [17-24] .

In this paper, the adsorption equilibrium and kinetics of safranin-O on pineapple peel were studied. Pineapple peel is a kind of agricultural waste that used as an adsorbent. It will considerably replace the use of conventional treatments in terms of cost effectiveness, economical and environmentally friendly. Major advantage in the use of biomass is the renewability and abundant supply. In fact, reliable and domestically produced especially those biomass resources that are by-products of agricultural activity.

MATERIALS AND METHODS

Adsorbate

In this study, the safranin-O, was selected as an adsorbate. The molecular formula of this cationic red dye is $C_{20}H_{19}ClN_4$ with IUPAC name of 3,7-Diamino-2,8-dimethyl-5-phenylphenazinium chloride. In order to prepare 1000 mL stock solution, 1.0g of safranin-O was dissolved into one liter of distilled water. The natural solution's pH of 5.6 was kept constant during the experiment.

All the reagents used in the present studies were of analytical grade. The chemical structure of the adsorbate in the present study is shown in Figure 1.

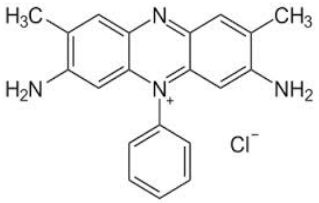

Figure 1. Molecular structure of safranin-O

Preparation of biosorbents

Pineapple peel (*Ananas comoscus*) used as sorbent in this study that was prepared from market near chokit area at Kuala lumpur, Malaysia. Firstly, sample was cut into small slices, and then washed thoroughly under running tapped water for several minutes and distilled water in order to remove dirt and sand particles. Secondly the cut samples were dried at 60 0C in an oven (Sigma Tech Scienctific Product, India) for 48h. After drying, samples were grounded and sieved (size: 1.18 mm) then stored with silica gel. The same adsorbent was used in all the experiments.

Batch studies

In this study, all experiments were carried out at room temperature. A 250 mL stopper cork conical flasks filled with 100 mL of adsorbate were carried out at different initial concentration 20-100 mg/L, the solution was in contact with adsorbent for about 80 min, and a pH (pH meter model Jenway 3305, England) ranging from 2 to 12 ; while adsorbent dose of 0.1 to 1.0 g was used in an orbital shaker at a constant speed of 170 rpm (Model Heidolph, incubator 1000, Germany). The final concentrations of dye were recorded by a double beam UV-Vis spectrophotometer (GENESYS-10-UV Model) at a wavelength of 516 nm. The amount of biosorption at equilibrium, q_e (mg/g), was computed by the following equation:

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

where, the initial dye concentration (mg/L), C_e is the equilibrium dye concentration in solution (mg/L), V is the volume of the solution (L), and M is the mass of the biosorbent used (g). The percentage removal of dye was calculated by the following relation:

$$\%Removal = \frac{(C_0 - C_f)}{C_0} \times 100 \quad (2)$$

In order to ensure accuracy of the data, the experiment was done in triplicate and the average values were recorded.

Determination of point zero charge

The pH_{ZPC} is the pH on zero point charge, which is the point at which the net charge of the adsorbent is zero. Balistrieri and Murray [25] used the solid addition method to determine the point zero charge of the adsorbent. An amount of 0.1g of pineapple peels was added to each conical flask containing 50 mL of KCl (0.01N) solution. The initial pH was adjusted in the ranges of 2-11 by NaOH (0.1N) and H_2SO_4 (0.1N) solution. The solution is placed in an orbital shaker and allowed to shake for the period of 48 h, all the conical flaks were withdrawn from the shaker and the final pH was recorded. The point of intersection of the curve pH_i-pH_f against pH_i is the point zero charge.

RESULT AND DISCUSSION

Effect of time on stream and initial concentration

The adsorption data for the removal of safranin-O versus contact time at different concentrations are presented in Figure 2. It can be observed from contact time curve that, the removal shows rapid adsorption of safranin in the first 3 min, thereafter, the adsorption rate decreases gradually and the adsorption reaches equilibrium in 80 min. The sorption capacity of the sorbent increased from 3.883 to 26.084 mg/g as the initial dye concentration increased from 20 to 120 mg/L. The increment in sorption capacity of the biosorbent may be due to the increase of dye quantity which resulted in higher concentration gradient of the dye, thus leading to higher sorption capacity.

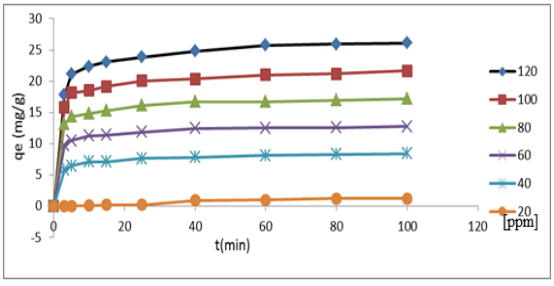

Figure 2. Effect of Time

Percentage uptake of safranin-O at different initial concentration

The percentage uptake of safranin-O increases with increase in initial dye concentration, and the sorption capacity of the adsorbent are directly proportional to the initial dye concentration. As illustrated in Figure 3, the plot of percentage uptake of safranin-O at different initial concentration against contact time, it can be seen that the trend of increments starts from 50.3 to 93.24% as the initial dye concentration increases from 20 to 120 mg/L.

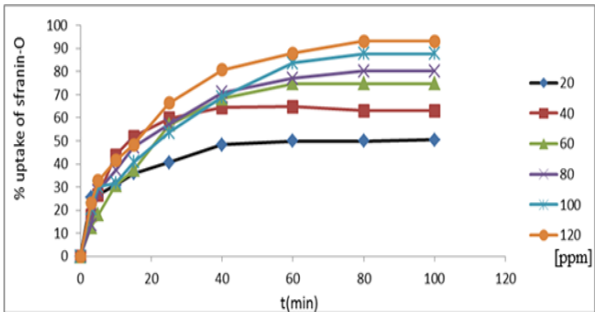

Figure 3. Percentage uptake of safranin-O

Effect of loaded adsorbent dose on safranin-O

The amount of pineapple peels used to remove safranin-O was varied from 0.1 to 1.0 g in order to investigate the relationship between adsorbent dosage and dye removal performance in which the results are shown in Figure 4. The effect of adsorbent dose on the amount of adsorbate removed increases with increase in adsorbent dose. The high quantity of adsorbent has resulted high uptake of safranin-O from the aqueous solution. The percentages of safranin-O adsorbed increased by increasing the mass of adsorbent from 20 to 86%. The increase in adsorption may be may be attributed to the present of more active functional group resulting in the availability of more adsorption site.

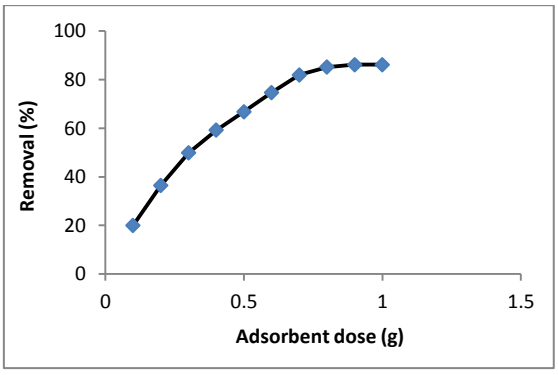

Figure 4. The Effect of Adsorbent dose

Influence of pH on safranin-O removal

Treatment of (safranin –O) wastewater is pH dependent. Therefore, the pH of the solution is known to affect its structural stability. Figure 5 has shown the dye removal trend from aqueous solution. The pH chosen for studies are 2 to 12. At pH above 6, the color of safranin-O irreversibly changes; that indicating an alteration in the structure of safranin-O molecules. Moreover, optimum removal due to pH was achieved at pH 6. The pH effect can be interpreted as the protonation and deprotonation of safranin-O occurring in the acidic and basic medium [26]. Furthermore, the adsorption behavior of such adsorbent at various pH may be due to surface charges and availability of active sites.

Point of zero charge

The pH_{ZPC} of adsorbent was measured to be 6 and the surface safranin-O dye was cationic in nature as shown in Figure 6. The pH_{ZPC} of the adsorbent revealed that the surface of the adsorbent is passively charged at pH less than 6 and negatively charge at pH values above 6. In acidic conditions, pH less than 6, a competition exists between the protons of protonated amine or sulphur groups [27]. Furthermore, it may contribute to a minor decline in the rate of adsorption. In addition when the pH greater than pH_{ZPC}, the presence of negatively charges in the pineapple peel surface, expedite electrostatic attraction between the negatively charged surface of the pineapple peel and the positive charged cationic safranin-O.

Adsorption Isotherm

Fundamentally, equilibrium adsorption isotherm is very significant in the design of adsorption systems. This study provides the adsorption capacity of the adsorbent. Moreover adsorption isotherm describes the equilibrium relationships between adsorbent and adsorbate, which is usually the ratio between the quantity adsorbed and that remaining in the solution at a fixed temperature. In this study, 20 to 120 mg/L concentration of adsorbate and 1g of adsorbent dose was used. The equilibrium data obtained experimentally were fitted to two most common models, Langmuir and Freundlich isotherm model.

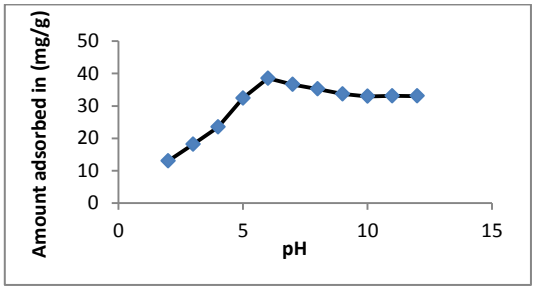

Figure 5. The effect of pH on safranin-O removal

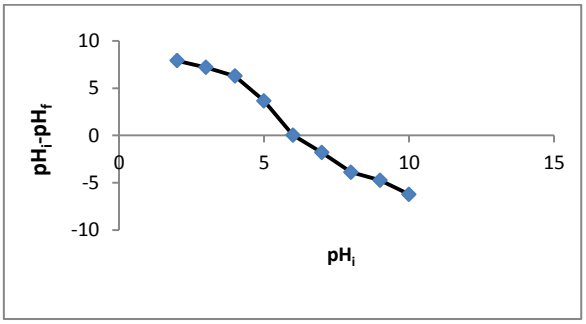

Figure 6. Point zero charge (pH_{ZPC}) of adsorbent using KCl (0.01 N)

Langmuir isotherm model

Once the molecule are concentrated on the adsorbent, there will be no transmigration of the solute (adsorbate) in the plane of the surface [28]. The adsorption energy will be uniform throughout the surface area otherwise the pollutant will have uniform adsorption or attractive forces. Based on the assumption that the maximum adsorption is relevant to a saturated monolayer of solute molecule on the adsorbent surface, the Langmuir model is explained by the following equation:

$$q_e = \frac{Q_{max} K_L c_e}{1 + a_L c_e} \tag{3}$$

where, q_e is the equilibrium concentration of adsorbate (mg/g), Qmax adsorption capacity, K_L is the constant related to efficiency of solute adsorption (L/mg), C_e Equilibrium concentration of adsorbate in solution (mg/L) and α_L constant related to energy of adsorption (L/mg). The above equation is termed as non- linear form of Langmuir, and can be linearized as stated below:

$$\frac{C_e}{q_e} = \frac{1}{K_L} + \frac{\alpha_L C_e}{K_L} \tag{4}$$

The essential characteristic of Langmuir isotherm can be expressed by separation or equilibrium parameter (R_L), a dimensionless constant, and can be define as follows:

$$R_L = \frac{1}{1 + bC_o} \tag{5}$$

The value of R_L indicates the nature of adsorption. If the separation factor is greater than one (R_L>1), it means the adsorption of safranin is unfavorable. While if the range is between 0 and 1 i.e. (0<R<1), this shows that, biosorption of safranin-O is favorable. But if the value of R_L equals to unity, the adsorption process is linear. And if equals to zero (R_L = 0) then, is an indication of an irreversible process. Table 1 is the summary of the aforementioned process [29].

TABLE 1. Values of R_L and corresponding isotherm type

Values of R_L	Type of isotherm
$R_L > 1$	Unfavorable
$R_L = 1$	Linear
$0 < R_L > 1$	Favorable
$R_L = 0$	Irreversible

From this plot, it can be seen that, R^2 = 0.83 which is relatively closed to unity; that shows a good absorption. This suggested that the Langmuir isotherm might be a suitable isotherm model. Thus it was concluded that the biosorption process of pineapple peels exhibit monolayer biosorption. The value of Langmuir constant, α_L and k_L were -0.042 and 0.81mg/L, respectively as shown in Table 2.

Freundlich isotherm

Freundlich [30] Proposed a model which is based on the assumption that, biosorption process takes place on

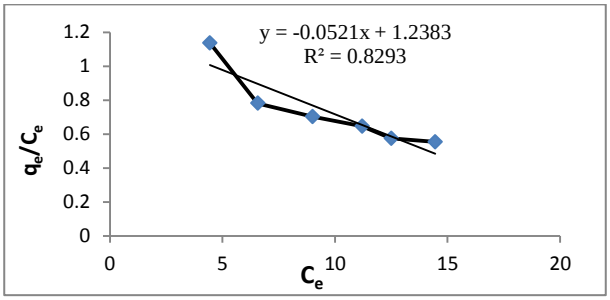

Figure 7. Langmuir isotherm for adsorption of safranin-O

heterogeneous surfaces and adsorption capacity is related to the concentration of adsorbate at equilibrium Mathematically, it is characterised by the heterogeneity factor 1/n.

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

where, K_f = constant that indicates sorption capacity of sorbate (mg/g). The unit is given as (L/g) (1/n), and n are constants indicating the intensity of adsorption. This constant can be one or any integer or decimal number. If n =1 (linear), n<1 (Chemisorption process), and n>1 is physic-sorption process. The linear expression of this model can take the logarithm of the equation on both sides, and is given by equation (7):

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \quad (7)$$

A plot of log q_e versus log C_e, gives a straight line with K_f and 1/n determined from the intercept and the slope, respectively. Data are summarized in Table 3 and also fitted data are shown in Figure 8.

TABLE 2. Langmuir isotherm parameter for safranin-O adsorption on pineapple peel adsorbent

Isotherm	K_L (mg/L)	α_L(mg/L)	R^2
Langmuir	0.808	-0.042	0.829

TABLE 3. Freundlich isotherm parameter for safranin-O adsorption on pineapple peel adsorbent

Isotherm	n	1/n	k_f	R^2
Freundlich	1.601	0.622	1.857	0.995

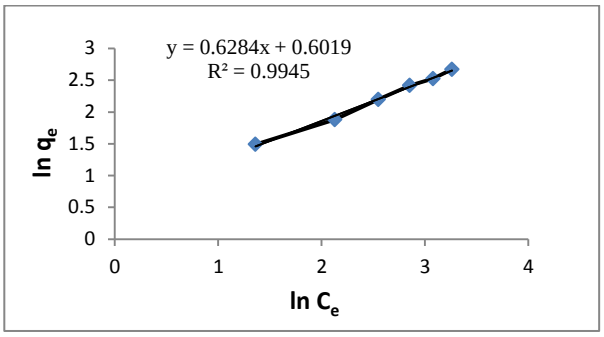

Figure 8. Freundlich isotherm for adsorption of safranin-O

Adsorption dynamic

The important of time dependent process on the adsorbent in the adsorption of individual compound in aqueous solution cannot be over emphasized. The adsorption of safranin-O on pineapple peels can be illustrated by the three successive steps. First the adsorbate migrates through the solution to the exterior surface of the adsorbent particles by molecular diffusion. The second step is the movement the solute from particle surface into the interior site by pore diffusion and finally the third step involved the adsorbate been adsorbed on to the active sites at the interior of the adsorbent particle. The final adsorption step is very rapid; the overall rate of adsorption will be controlled by film diffusion or internal diffusion [31].

Kinetics studies

The kinetic adsorption data provides the understanding of dynamics of adsorption process in terms of the order of rate constant [32]. The adsorption experiment was carried out at concentration of 20 to 120 mg/L, pH 6, adsorbent dose of 1g and contact time varied was from 3 to 80 min. Two kinetic models were used to find which model fits the experiment: Lagergren's Pseudo-First order and Second- order Kinetic models.

Pseudo-first order model

The pseudo-first-order rate model equation was first developed by Lagergren [33]. The relationship can be represented by equation (8) :

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \quad (8)$$

where, k_1 is the pseudo-first-order rate constant (1/min), q_e = amount of adsorbate at equilibrium (mg/g) and q_t = are biosorption capacities at equilibrium with respect to time t (mg/g). Integrating equation 8 for the boundary condition t = 0 to t = t and q_t = 0 and qt yields as below:

$$log(q_e - q_t) = log\, q_e - \frac{k_1}{2.303}t \quad (9)$$

The constant value K_L were obtained by plotting a straight line graph of log (qe –qt) against t. Correlation coefficients R^2 as it can be read off from Figure 9. Different safranin concentrations are summarized in Table 4 below. The correlation coefficient (R^2) of 0.808 is obtained at a concentration of 20 mg/L, while (R^2) of 0.965 is obtained at 120 mg/L. The calculated value of adsorption capacity (qcal) was obtained as 1.349 mg/g at 20 mg/L and 7.241 mg/g at 120 mg/L, respectively.

Pseudo-second-order kinetic mode

The pseudo-second-order equation based on equilibrium adsorption is expressed as equation (10).

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \quad (10)$$

where k_2 (g/mg-min) is the pseudo-second-order rate constant. Integrating Eq. (10) for the boundary condition t = 0 to t and qt = 0 to qt is given by equation (11).

$$\frac{1}{q_e - q_t} = \frac{1}{q_e} + k_2 t \quad (11)$$

The linear form of this model is represented as equations (12 and 13):

$$\frac{t}{q_t} = \frac{1}{h} + \frac{1}{q_e}t \quad (12)$$

where,

$$h = k_1 q_e{}^2 \quad (13)$$

The plot of t/q_t against t gives a straight line. Figure 10 shows the linear form of pseudo-second-order kinetic at different safranin concentration on pineapple peel. The correlation coefficient R^2 for the pseudo-second-order kinetic at different concentration was 0.998 (for 20 mg/L) to 0.999 (for 120 mg/L), respectively; while the calculated values of adsorption capacity (q_{cal}) tend to be higher than that of the pseudo-first-order kinetic, its value ranges from 2.349 (for 50 mg/L) to 17.331 (for 120 mg/L) at different concentrations. These facts suggest that the pseudo-second-order adsorption mechanism is predominant, and that the overall rate of the dye adsorption process appears to be controlled by the chemisorption process.

TABLE 4. Adsorption kinetic parameters of safranin-O on pineapple peel adsorbents

	First-order Model					Second-order Model				
C_o (mg/L)	R^2	q_e (cal) (mg/g)	q_e(exp) (mg/g)	K_L (min^{-1})	Intercept	R^2	q_e (cal) (mg/g)	q_e (exp) (mg/g)	K_2	Intercept
20	0.8075	1.3489	1.3489	2.2984	0.1300	0.9980	2.3491	3.8829	0.1558	0.4257
40	0.9148	3.5432	8.3891	2.3000	0.5494	0.9987	4.6795	8.3891	0.0665	0.2137
60	0.8280	3.9902	12.7518	2.3016	0.6010	0.9996	10.9051	12.7518	0.0671	0.0917
80	0.8288	5.1263	17.1992	2.3017	0.7098	0.9994	14.3884	17.1992	0.0486	0.0695
100	0.8407	7.1565	21.6253	2.3019	0.8547	0.9995	15.9489	21.6253	0.0314	0.0627
120	0.9652	7.2410	26.0836	2.3021	0.8598	0.9996	17.3310	26.0836	0.02547	0.0577

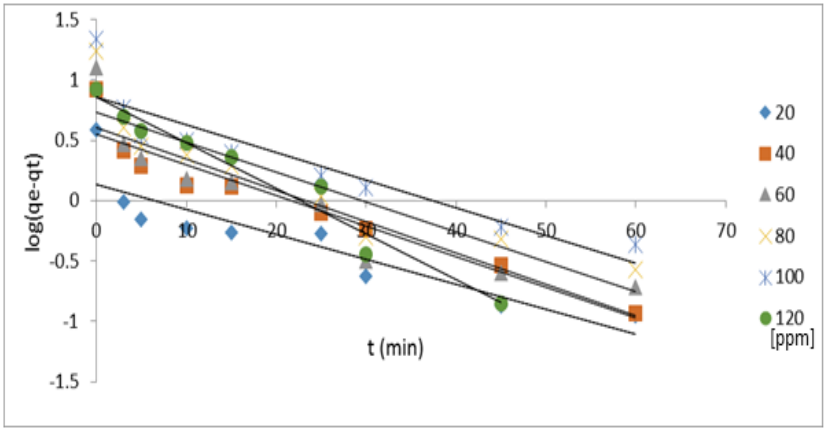

Figure 9. Pseudo–first order model for adsorption of safranin-O

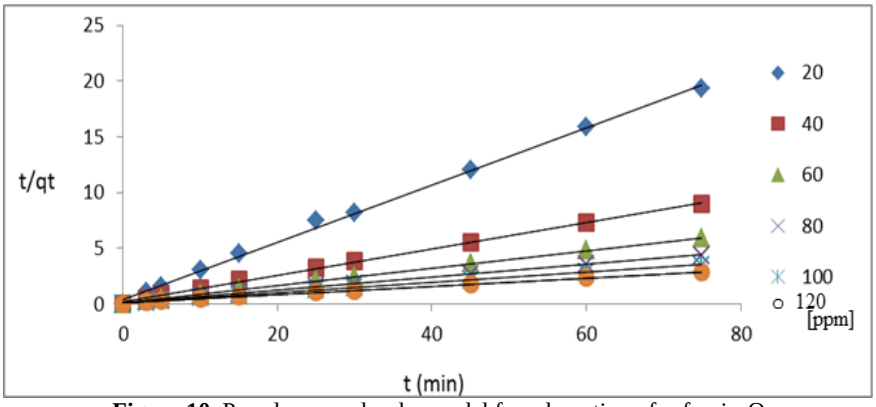

Figure 10. Pseudo-second order model for adsorption of safranin-O

CONCLUSION

The experimental result from this study suggests that pineapple peels is a potential adsorbents for removal of safranin-O from aqueous solutions. Conclusion can be drawn from the effect of parameters, such as initial dye concentration, adsorbent dosage, and pH of solutions. Furthermore, from the result obtained, the adsorbent is found to effectively remove safranin-O by 93.24% at initial dye concentration of 120 mg/L in 80 minutes contact time. The removal as a result of increases with increase in adsorbent dosage, while percentage uptake due to pH was achieved at a pH of 6, showing that the acidic condition is favorable for the sorption process. Adsorption isotherms behavior suggests that, the present adsorption exhibit Freundlich isotherm model characteristic, reflecting the presence of more than one kind of adsorbent–adsorbate surface interaction. Thus heterogeneous surfaces and adsorption capacity is related to the concentration of adsorbate at equilibrium. The adsorption kinetics data followed pseudo-second-order kinetic model with high correlation coefficient almost reaching a unit value (0.999). In a nutshell, this is an indication of chemisorption's or an effective electrostatic interaction playing a vital role in the study.

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Persian Abstract

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چکیده

هدف این پژوهش مطالعه چگونگی خارج کردن safranin-O از محلول آبی با جذب سطحی آن بر روی پوست آناناس می باشد. تاثیر pH محلول، غلظت اولیه رنگ، زمان تماس و مقدار جاذب مور مطالعه قرار گرفت. ظرفیت بهینه جذب به میزان ۲۶/۰۸ mg/g در دما و زمان بهینه ۶۲۹۳ کلوین و ۸۰ دقیقه بدست آمد. علاوه بر این مطالعات بعدی نشان داد که safranin-O ۹۳/۲۴% به میزان چشمگیری در غلظت رنگی ۱۲۰ mg/L در زمان تماس ۸۰ دقیقه اتفاق افتاد. نتایج مطالعات ایزوترم نشان داد که داده های تعادلی به خوبی بر مدل فرندلیچ قابل فیت کردن است درحالیکه داده های سینتیکی جذب نشان داد که فرآیند جذب به خوبی با مدل سنتیکی pseudo-second order (شبه درجه دو) به خوبی توصیف می شود. نهایتا این نتیجه حاصل شد که پوست آناناس پتانسیل بالایی برای جذب و خارج کردن safranin-O از محلول آبی دارد.