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Batch removal of hazardous safranin-O in wastewater using pineapple peels as an agricultural waste based adsorbent

Mohammed, M. A.*, Ibrahim, A., Shitu, A.

Department of Chemical and Environmental Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400, UPM Serdang, Salengor, Darul-Ehsan Malaysia

Email address:
alisulezee@gmail.com (Mohammed, M. A)

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Abstract: Towards attaining a sustainable engineered chemical processes, pineapple peels waste (Ananas comoscus), a low cost agricultural waste material was investigated to serve as an adsorbent for removal of safranin-O in wastewater treatment. The process was carried out in a batch with different initial concentrations of the adsorbate. The amount of adsorbent dose used was varied, as well as pH and contact time. Initially, the uptake of dye was very fast, but gradually slowed down indicating penetration into the interior of the adsorbent particles. it was observed that acidic pH was more favorable for the adsorption. Maximum adsorption capacity was reached after 90min, during which the adsorbate and adsorbent were in contact at 29oC. The results obtained fitted Freundlich and Langmuir models; the Freundlich model better described the equilibrium dye uptake than the Langmuir. The study suggests that Pineapple peel wastes can be a potential alternate adsorbent for safranin-O removal from wastewater.

Keywords: Agricultural Waste, Waste Water, Adsorption, Safranin-O, Pineapple Peel

1. Introduction

Over the past century, there has been a dramatic increasing concern about environmental protection. Today, Dyes are one of the most important groups of chemicals widely used in paper, textiles, pharmaceutical, rubber, plastics, leather, cosmetics, and food industries, in order to colour products. However, discharge of wastewater without proper treatment from these industries as a byproduct in to water bodies has been reported to impair the normal function of aquatic life and change in food web [1]. Dyes present in waste water may cause serious environmental pollution problems by reducing light penetration and reduce photosynthesis [2]. Moreover, waste water containing dyes are one of the sources that diminish the esthetic value of rivers and causes eutrophication. The Effluents are highly visible even at very low concentrations and undesirable. It might be also toxic and carcinogenic [3_4].

Safranin-O, a cationic dye discharged mostly in textile and pharmaceutical industries (veterinary medicine). Exposure to these effluents may be irritating to respiratory systems, skin, and digestive tract infections when ingested [5]. It is therefore, necessary to decolorize waste water to the lowest permissible concentration in order to the safeguard the water bodies as stated by environmental regulations. The different methods of conventional waste water treatment includes oxidation or ozonation, membrane separation, precipitation, coagulation/flocculation, ion-exchange, and reverse osmosis [6]. The complex nature of structure of the dyes and their synthetic origin, made it not to degrade easily; making it difficult for conventional methods to remove dye from waste water efficiently.

Commercial activated carbons are one of the most widely used agents of decontaminating waste water and its effectiveness in yielding good results after treatment has been confirmed [7], but from the economic point of view the cost is high, as such researchers have to device an alternative low-cost adsorbent that can compete favorably with Commercial activated. In the hunt of developing effective, clean and economic method, natural materials, agricultural waste and industrial by-products were utilized to substitute the more expensive commercially available activated carbons [8].

Table-1 listed some of the reported biosorbents used for removal of safranin-O from aqueous solutions. A compilation of extensive list of biosorbent used in for the treatment of industrial waste water has been reported recently by [9]

Table 1. Adsorption capacities of safranin dyes onto different adsorbents.

Adsorbent(s)	Adsorbate(s)	Adsorption Capacity	Reference
Alkali-treated Rice husk	safranin	9.77mg/g	[10]
Alkali–treated Mango seed	safranin	31mg/g	[11]
Micellar enhance ultra violet	safranin	99%	[12]
Activated Rice husk	safranin	82%	[13]
Corncob activated carbon	safranin	1428.57mg/g	[14]
NaOH-treated rice husk	safranin	37.97 mg/g	[15]
Pretreated Rice Husk	safranin	45.582mg/g	[16]
Pineapple peels	safranin	21.7mg/g	This work

In dealing with organic and inorganic micro pollutants from aqueous effluents, biosorption technology is rapidly gaining prominence as an efficient wastewater treatment technique. However, application of biosorbents at commercial level is still a challenge. This technique can handle fairly large flow rates, producing a high-quality effluent where no or less harmful sludge is produced [17]. Furthermore, biosorption process has the capacity to minimize both organic and inorganic pollutants concentrations and thus has a wider application in controlling pollution.

In this work, effort has been made in using pineapple peel, as a non-conventional low-cost adsorbent in removing safranin-O from aqueous solution. Pineapple peels waste, have been found as a high potential biosorbent in the treatment of dye. A total carbohydrates including hemicelluloses, celluloses, reducing and non-reducing sugar content of fresh, dry and ensiled pineapple wastes was found to be 55% [18]. Waste produced from this fruits also contributes to the large amount of agricultural waste generated. Thus, proper handling and utilization is essential.

2. Materials and Method

2.1. Safranin-O Solution

Safranin-O (cationic red dye; chemical formula, C20H19CIN4; MW, 350.84g mol-1; IUPAC name as 3, 7-Diamino-2, 8-dimethyl-5-phenylphenazinium chloride) was used as adsorbate. This product was obtained from Sigma–Aldrich (M) Sdn Bhd, Malaysia. A stock solution 1000ml was prepared by dissolving a weighed amount (1.0g) of safranin-O in one liter distilled water. Different concentrations were prepared by diluting the stock solution with suitable volume of distilled water and the natural pH of the stock solution was around 5.6. The reagent used of analytical grade.

2.2. Biosorbent Sample

Pineapple peel (Ananas comoscus) used in the batch experiment, were collected from nearby mini market at Sri Serdang area near UPM small gate. It was then cut in to smaller pieces and washed with distilled water to remove the surface-adhered particles. The sample was further rinsed repeatedly with distilled water dried in an oven at 60oc for 48 h. Then ground using mortar and pestle. The grounded samples were then sieved to (1.18mm) particle size range and stored in a sealed plastic bag with silica gel to minimize dampness. Powdered material was further preserved in the desiccator and used in the adsorption studies.

2.3. Adsorption Equilibrium

Batch Adsorption equilibrium experiments was carried out by adding a fixed amount of adsorbent (0.1g) into a number of 250mL-stoppered glass (Erlenmeyer's flasks) containing a definite volume (50mL in each case) of different initial concentrations (20–100mg/L) of dye solution without changing pH and temperature 30oC. The flasks were placed in orbital shaker and agitation was provided at 175rpm.

The experiment was performed in duplicate and the average results were used. The amount of dye adsorbed at time t, qt (mg/g), was obtained by calculating the difference between the initial and the final safranin concentration as shown in equation 1

$$q_e = (c_0 - c_e)\frac{V}{w} \tag{1}$$

Where qe (mg g-1) is the amount of dyes adsorbed, and C0 (mg/L) is the initial dye concentration, while Ce (mgL-1) is the concentration of dye in solution at equilibrium, V (L) is the volume, and w (g) is the weight of pineapple peel. The percentage removal of the dye was computed using the following equation

$$\text{Percentage of removal (\%)} = \frac{C_i - C_e}{C_i} \times 100 \tag{2}$$

Where Ci and Ce are the initial and equilibrium concentration of dye (mg/L) in solution.

3. Results and Discussion

3.1. Characterization of Pineapple Peel

Analysis of IR Spectra before and after adsorption by pineapple peel (Fig.1) show a number of absorption peaks, suggesting complex properties of the biosorbents. It could be seen that there is a much weaker characteristic stretching vibration absorption band at 1730.78 cm−1 is assigned to carbonyl group C=O, this may be due to acetyl, uronic ester groups of hemicelluloses or the ester linkage of carboxylic groups [19]. The dominant peak at 3708.54 cm−1 is attributed to O–H stretching vibrations in hydroxyl groups. A shift in hydroxide group from 3936 cm-1 to 3708.54 cm-

1, alcohol group from 1825 cm-1 to 1682.80 cm-1 while a shift in carbonate group from 1472 cm-1 to 1304 cm-1 was observed Shifting in peaks was observed after adsorption and this shows that all the functional groups are completely involved in biosorption process.

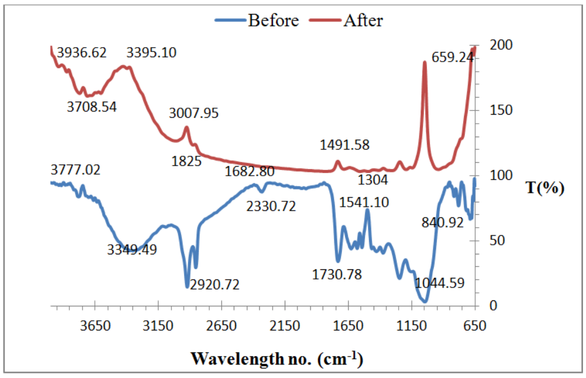

Figure 1. FTIR spectra of biosorbents.

3.2. Scanning Electron Microscopy (SEM) Analysis

The SEM image of pineapple peels before adsorption and after 90min. from Fig. 2(a). An irregular surface with a low and non-porous surface area can be seen clearly, indicating that there is a good possibility for safranin dye to be adsorbed into the surface. Biosorption of safranin leads to multiple attachment on the rough surface and occupation of pores Fig (2b). Pores in solids medium like adsorbents, may have properties such as shape, location, connectivity, and surface chemistry. Perhaps one of the properties of a pore easy to visualize, is its size i.e., its extent in one spatial dimension. This may probably be one of the reasons why size is often the first or main property used to characterize a pore.

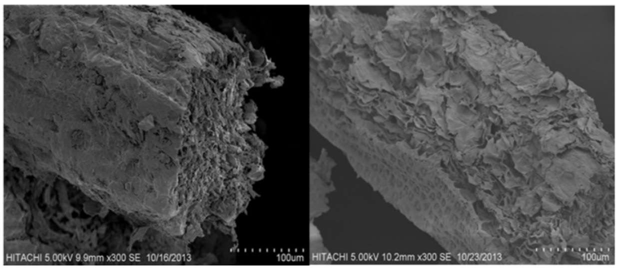

Figure 2. SEM images for pineapple peel adsorbent (a) before adsorption and (b) after 90min adsorption process.

3.3. Equilibrium Studies

3.3.1. Effect of Initial Concentration and Contact Time

The effect of initial concentration of safranin was studied between the range 20-100mg/L at a fixed adsorbent dosage (0.10g) and shaker speed (175rpm) at 29◦C. the effect of this is shown in Fig. 2. From the plot, it can be observed that dye uptake was rapid for the first 3min and thereafter it proceeded at a slower rate till the equilibrium state for each concentration reached. The result indicates that an increase in initial safranin-O concentration leads to increase in the adsorption of safranin on pineapple peel. At equilibrium, safranin adsorption increased from 3.8 to 21.7mg/g, with increase in the initial safranin concentration from 20 to 100mg/L. The increment in sorption capacity of the biosorbent may be due to the increase of dye concentration which resulted in higher concentration gradient of the dye, thus leading to higher sorption capacity. Similar trend was reported in the adsorption of methylene blue [20-21]. However, for the purpose of accuracy, the data were measured at 90min to be sure that full equilibrium was attained.

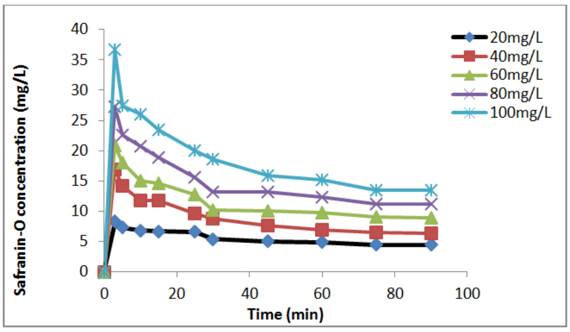

Figure 3. Effect of Initial Concentration and Contact Time on amount of safranin-O adsorbed (Adsorbent dose: 1g, agitation speed: 150rpm).

3.3.2. Effect of pH on Adsorption

In this work, the effect of initial pH on equilibrium uptake of pineapple peel was studied at 50mg/L initial safranin concentration and 29^{0}C as shown in Fig. 1. Variation in the pH affects the efficiency of dye adsorption. However, the adsorption of safranin increased with an increase pH. Optimum pH for the adsorption of dye was found to be in the range 6–8. Considering the electrostatic force of attraction the existence of a negatively charged surface of the adsorbent may be due to pectin and safranin, a cationic dye. At acidic pH, decrease in the number of positively charged sites and an increase in number of negatively charged sites may be responsible for the removal of the cationic dye. Similar trends were observed in the use of yellow passion fruit peel as adsorbent for the Removal of methylene blue dye [22]. It can be deduced that the lower biosorption at alkaline pH may be due excess H+ ions competing with the dye cations for adsorption sites.

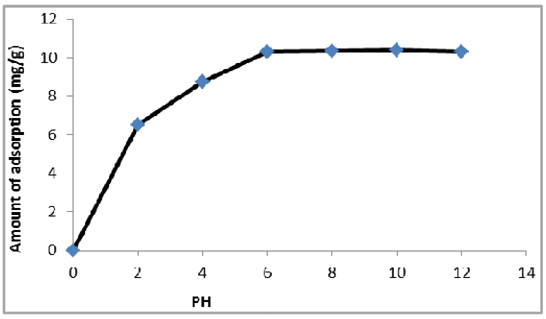

Figure 4. The Effect of pH on uptake of safranin-O (Initial concentration: 60 ppm, adsorbent dose: 1g, contact time: 90minutes, agitation speed: 150rpm).

3.3.3. Effect of Adsorbent Dose on Dye Adsorption

The effect of adsorbent dose on the removal of safranin-O was studied at initial concentration of Co =50mg/L and a temperature of 29◦C, while the amount of adsorbent added were varied from 0.5g to 2.5g as shown in Fig 5. The percentage removal of safranin increases rapidly with increased in adsorbent dose from 32.4 to 43.3% respectively. The increase in % color removal was due to the increase of the available sorption surface. A similar results was reported in sorption of methylene blue on peanut hull [23].

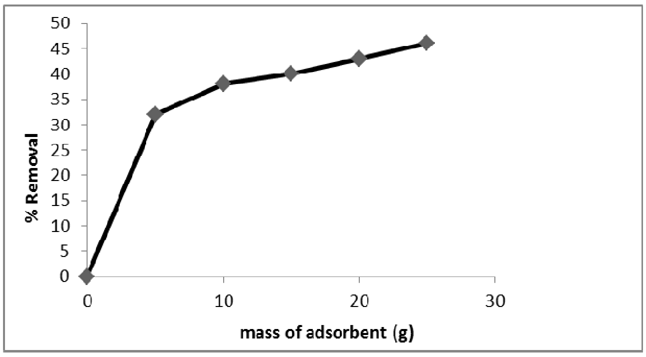

Figure 5. *The Effect of Adsorbent Dose to the Uptake of Safranin-O (Initial concentration: 50ppm, Adsorbate: 100mL, agitation speed: 150rpm, contact time: 90 minutes)* .

3.4. Isotherm Analysis

Adsorption isotherms analysis for the safranin-O removal were studied using initial concentration of safranin between 20 mg/L and 100 mg/L at an adsorbent dosage 1g. The adsorption isotherm is an arithmetic model that illustrates the distribution of the adsorbate species among adsorbent and correspond to the relationship between the mass of the solute adsorbed per unit mass of adsorbent q_e and the solute concentration at equilibrium Ce. The design of adsorption systems for equilibrium adsorption isotherm is of enormous and has been can be described by a number of models available in the literature. In this work, The results of the equilibrium isotherms were analyzed using the Langmuir [24] and Freundlich [25] isotherms.

3.4.1. Langmuir Isotherm

This model assumes maximum adsorption corresponds to a saturated monolayer of solute molecule on the homogenous adsorbent surface. It comprised of a finite number of identical sites with homogeneous adsorption energy [26-27]. A basic assumption states that sorption takes place at specific homogeneous sites within the adsorbent. Once a dye molecule occupies a site, no further transmigration can take place at same site. Saturated monolayer isotherm can be represented as

$$q_e = \frac{Q_m K_{Ce}}{1+KC_e} \tag{3}$$

Where q_e is the dye uptake capacity (mg/g) and Ce is the concentration of dye in the solution (mg/L) when equilibrium is reached, Q_m is the maximum biosorption capacity (mg/g), and K is a constant that signifies the affinity between the biosorbent and the dye. The plot of C_e/q_e against C_e was employed to generate the intercept value of $1/bq_m$ and the slope of $1/q_m$ as shown in (Fig. 6)

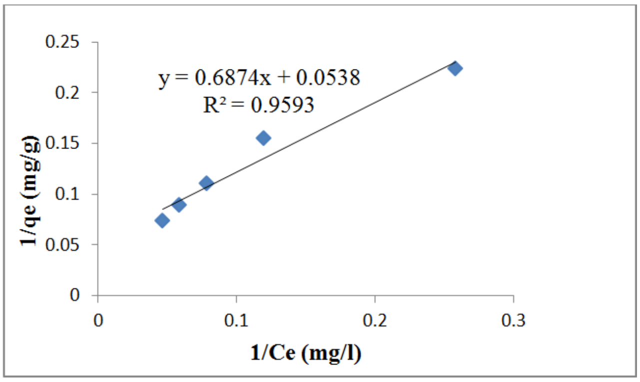

Figure 6. *Langmuir Isotherm for Adsorption of Safranin-O on Pineapple peel Adsorbent.*

3.4.2. Freundlich Isotherm

Freundlich isotherm is an empirical equation describing adsorption onto a heterogeneous surface or surfaces supporting sites of varied affinities it. Due to variation of interactions between the adsorbed molecules sites with stronger affinity are occupied first, and in this way multilayer setting of adsorbate molecules takes place. The validity of the Freundlich isotherm model was proved by using following relation:

$$\log q_e = \log K_f + \frac{1}{n}\log C_e \tag{4}$$

Here, Ce denotes the equilibrium concentration of the adsorbate, and qe, the amount adsorbed (mg/g), while K_F and n are the Freundlich constants related to the adsorption capacity and adsorption intensity of the adsorbate–adsorbent system, respectively. The Freundlich parameters for the adsorption of the dye onto adsorbents are shown in Table 1. The fit of the data for the dye onto these adsorbents suggests that Freundlich model (r^2 = 0.985) gave closer fittings than the Langmuir model.

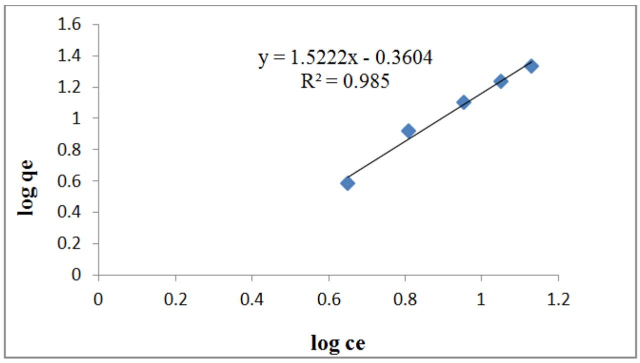

Figure 7. *Freundlich Isotherm for Adsorption of Safranin-O on Pineapple peel Adsorbent.*

3.5. Comparison of Langmuir, Freundlich and Experimental Data

A comparison between Langmuir, Freundlich and Experimental Data for Safranin-O adsorption was made. The equilibrium concentration of adsorbate, qe after adsorption was calculated. Fig. 7 shows the plots of comparison while table 1 below, shows the calculated data.

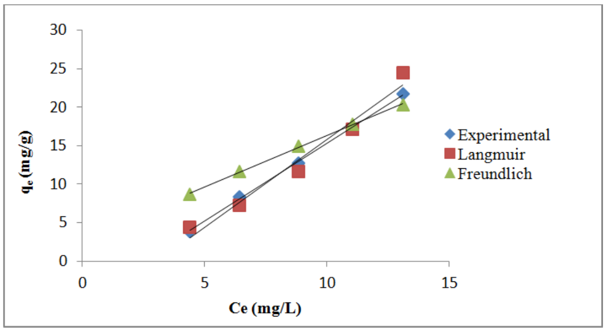

Figure 8. *Comparison of Experimental q_e and Isotherms q_e*

Table 2. *Experimental qe and calculated qe for Isotherms*

Ce (mg/L)	qe (mg/g)	q_l (mg/g)	q_f (mg/g)
4.402	3.899	12.622	6.656
6.415	8.396	12.670	11.808
8.833	12.792	12.699	19.215
11.053	17.327	12.715	27.031
13.104	21.724	12.724	35.026

From the table above it can be seen that, the value of qe Freundlich was closer to experimental qe compare to Langmuir. Moreover Freundlich R^2 from the graph is closer to 1, and for this reason, Freundlich correlation is a good representation of safranin-O adsorption. This case was also followed in the removal of basic yellow dye from aqueous solution using green alga Caulerpa scalpelliformis [28].

3.6. Intraparticle Diffusion

Intraparticle diffusion is the most common technique used for identifying the mechanism involved in biosorption process. In order to investigate the mechanism of the safranin-O adsorption onto pineapple peel adsorbent, intraparticle diffusion based mechanism was studied. It is an empirical functional relationship common to most adsorption processes, where uptake varies almost proportional with $t^{1/2}$ rather than with the contact time t. The possibility of intra-particle diffusion was explored by using the intra-particle diffusion model [29].

$$q_t = k_{id}t^{1/2} + I \qquad (5)$$

Where k_{id} is the intra-particle diffusion rate constant (mg/ (g $min^{1/2}$)) and I (mg/g) is a constant which gives information regarding the thickness of boundary layer. From fig. 8, it can be observed the plots are not linear over the whole time range, this implies that more than one process is controlling the process. This deviation from the origin is proportional to the boundary layer thickness. Therefore giving an insight on the tendency of the safranin to be adsorb to the pineapple peel or remain in solution [30].

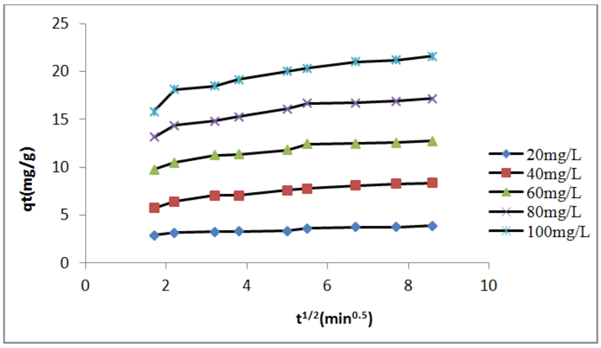

Figure 9. *Intraparticle diffusion plot for adsorption of safranin-O on pineapple peel waste for different initial safranin concentrations.*

4. Conclusions

The use of agricultural waste material (pineapple peels) as a low-cost adsorbents for removal of safranin-O in wastewater has been investigated and was found to be a potential adsorbent. The maximum adsorption capacity was found to be 21.7 mg/g without any pre-treatment of the adsorbent; It was also found that the adsorption performance of dye was significantly affected by pH, and particle size (surface area of the pineapple peels). The study suggests that Pineapple peel wastes can be a potential alternate adsorbent for safranin-O removal from wastewater.

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