Equilibrium, Kinetics and Thermodynamic of Dye Adsorption by Low – Cost Adsorbents

S. Sawasdee and P. Watcharabundit

Abstract—**This work is to study the adsorption of a commercial dye (magenta color) for silk yarn by low-cost adsorbents such as green perlite and shrimp shell (*Macrobrachium rosenbergii*). The adsorption experiments were carried out in batch process without adjusting pH. The various parameters, such as contact time, initial dye concentration and adsorbent doses were evaluated at the natural pH. The equilibrium data at various concentrations were analyzed using Langmuir, Freundlich and Temkin isotherms. The results showed that the maximum adsorption capacities of Langmuir isotherm valued 14.805 mg/g for green perlite and 5.621 mg/g for shrimp shell at 30 ℃. The kinetic data was better fitted with pseudo–second order for the adsorbents. Thermodynamic properties such as free energy (ΔG), enthalpy (ΔH) and entropy (ΔS) indicated spontaneous and endothermic in nature of adsorption for all adsorbents. The study revealed that green perlite and shrimp shell can be used as an efficient adsorbents for the removal of dye solution.**

Index Terms—**Adsorption isotherm, kinetic, thermodynamic, low–cost adsorbents.**

I. INTRODUCTION

Synthetic dyes are used for dying wool, cotton, nylon, silk and modified acrylics. It is estimated that 10–15% of the dyes are lost in the dye effluent during such dyeing processes [1]. The presence of dyes can cause damage to living beings in water. In addition, the stability of most dye molecules under condition of light, heat and chemicals leads to the fact that the dying effluents are difficult to degrade [2]. Many treatment system have been used for the removal of synthetic dyes from aqueous solutions including ion exchange, membrane filtration, precipitation, photocatalytic degradation, oxidation, electrochemical and adsorption. Among several dye removal techniques, adsorption is an efficient one to remove different kinds of dyes from water and wastewater [3] and it has wide applicability in wastewater treatment [4]. This process is simple to design, easy to operate, cost effective, eco–friendly and insensitive toxic substances. Activated carbon is the most efficient and popular adsorbent and has been used with the great success [5] because of high surface area, high adsorption capacity, but it is relatively high price and difficult to regenerate. Therefore, much attention has recently been paid in seeking cheap, locally available and effective adsorbent [6].

Natural adsorbents such as agricultural waste, waste food or low–cost inorganic material have been most popular for wastewater treatment due to availability and low– cost adsorbent. Recently, these have been alternative natural adsorbents for treatment of dye wastewater such as peanut hull [7], bentonite from China [8], [9], diatomite from Jordan [1] and shrimp shell [3] etc.

The purpose of this work was to study the adsorption of a synthetic dye for silk yarn on low-cost adsorbents namely, green perlite and shrimp shell. The equilibrium study was investigated to observe the effects of various adsorption parameters such as contact time, adsorbent dose and initial dye concentration on the process. Adsorption data were analyzed using Langmuir, Freundli*c*h and Temkin adsorption isotherm models. Moreover, the kinetic and thermodynamic studies were investigated.

II. EXPERIMENTAL

A. Adsorbents Preparation

Green perlite was collected from Sa Bot, Lopburi, Thailand. It was removed all dirt by double distilled water and dried in an hot air oven at 100 ℃ for 24 h.

Shrimp shell was obtained from fresh food market in Lopburi City, Thailand. It was removed all dirt by double distilled water and dried in an hot air oven at 80 ℃ for 24 h.

Particle size all adsorbents were ground and sieved to particle size 50–100 mesh and stored in a dessicator until use.
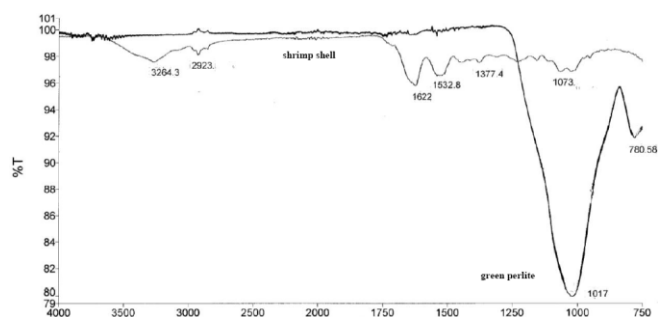

Fig. 1. FTIR of green perlite and shrimp shell.

B. Adsorbate Preparation

Adsorbate is a commercial synthetic dye for silk yarn. Its color is magenta and its brand is "Cats run around the World" (Thailand). It was obtained from a local shop in Lopburi, Thailand. The dye was used as such without further purification. It was dried at 80 ℃ for 2 h before use. The stock solution was prepared by dissolving a constant mass of dye in double distilled water to achieve the concentration of 100 mg/L and subsequently diluted to the required concentrations. The pH of its solution at 20mg/L is 7.85. Absorbance value of dye solution was measured by a double beam UV–Vis

spectrophotometer (Analytik Jena, Specord 210 plus) and dye concentration was determined by comparing with its calibration curve at the wavelength 548 nm.

C. Characterization of Adsorbents

The surface functional groups of adsorbents were analysed by FTIR (Perkin Elmer model two) and the spectra were recorded from 4000–750 cm^{-1}.

D. Batch Experiment Adsorption

Various adsorption parameters, such as contact time, adsorbent dose and initial dye concentration were evaluated. The pH of the solution was not adjusted. Adsorption experiments were carried out in 250 ml Erlenmeyer flasks by known mass of adsorbent, 100 ml of dye solution under an isothermal shaker (30±1°C) and agitation speed 250 rpm. Then, the suspended matter in each sample was filtered and supernatant was measured for dye concentration by the double beam UV−Vis spectrophotometer at 548 nm. The amount of adsorption capacity (*q*) and percentage of adsorption were calculated as follows:

$$q = \frac{(C_O - C_t)V}{W} \qquad (1)$$

$$\% \text{ adsorption} = \frac{(C_O - C_t)}{C_O} \times 100 \qquad (2)$$

When C_O and C_t (mg/L) are dye concentrations at the initial and any time (*t*, min). *V*(*L*) is the volume of the solution, *q* (mg/g) is the amount adsorbed at any time (*t*, min) and *W*(g) is the mass of adsorbent.

III. RESULTS AND DISCUSSIONS

A. Characterization of Adsorbents

FTIR spectra of green perlite and shrimp shell have shown in Fig. 1 The peak from green perlite around 1017 cm^{-1} due to siloxane (Si−O−Si) and 794 cm^{-1} correspond to SiO−H [10]. The peak from shrimp shell around 3264.3 cm^{-1} due to the stretching of OH group overlapping stretching band of N−H [11]. The peak of 1622 cm^{-1} correspond to the bending in plane of N−H. The band at 1073 cm^{-1} is assigned to C−N bending [12].

B. The Effect of Contact Time

Fig. 2 shows the effect of contact time of dye adsorption on green perlite (0.6 g) and shrimp shell (1.0 g) at the initial concentration 20 mg/L. The plot of adsorption capacity (q) versus the contact time for initial dye concentration 20 mg/L is shown in Fig. 2. The removal of dye was rapid at the initial stage of contact time and gradually decreased with time until equilibrium. The rapid adsorption observed during the first 30 min was probably due to the abundant availability of active sites on the adsorbents surface, and with the gradual occupancy of these sites, the adsorption became less efficient. It appeared that the contact time needed to reach equilibrium conditions were 180 min and 30 min for green perlite and shrimp shell, respectively. The contact time of adsorption in

this experiment is accordance with the results obtained in dye adsorption experiments onto different types of adsorbents as for example, Ca−bentonite [13], shell of *Penaeus indicus* shrimp [3]. The adsorption capacity increased with an increase in the contact time and was constant at the equilibrium time. The equilibrium capacities of adsorption were 3.077 mg/g and 1.840 mg/g onto green perlite and shrimp shell.
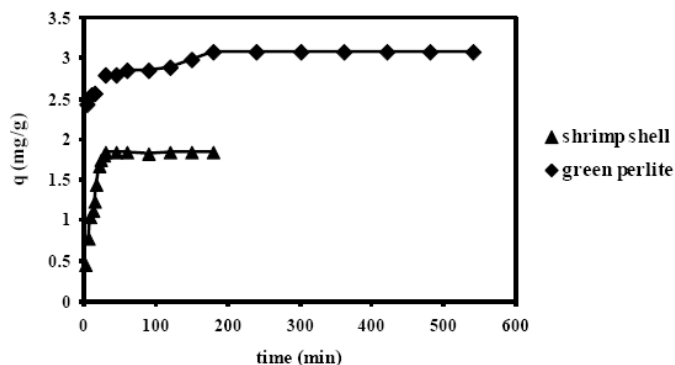

Fig. 2. effect of contact time on dye adsorption.

C. Effect of Adsorbent Doses

The equilibrium adsorption of dye on green perlite and shrimp shell was investigated at 30°C by varying the adsorbent amount from 0.20 g to 1.00 g of green perlite and from 0.60 g to 2.00 g of shrimp shell. Fig. 3 shows the plot of percentage of adsorption against the adsorbent doses at the initial dye concentration 20 mg/L. The results were shown that the percentage of dye adsorption was 75.10−95.21% for green perlite and 38.82−75.07% for shrimp shell. The percentage of adsorption increased with an increase in adsorbent dose (g) due to increase binding site at higher amount of adsorbent [14]. However, it was observed that the rate of dye removal percentage slightly decreased as the adsorbent dose increased. It was due to the presence of higher amount of adsorbent in dye solution resulting in reduced distances between the adsorbent particles, thus making many binding sites unoccupied (15).
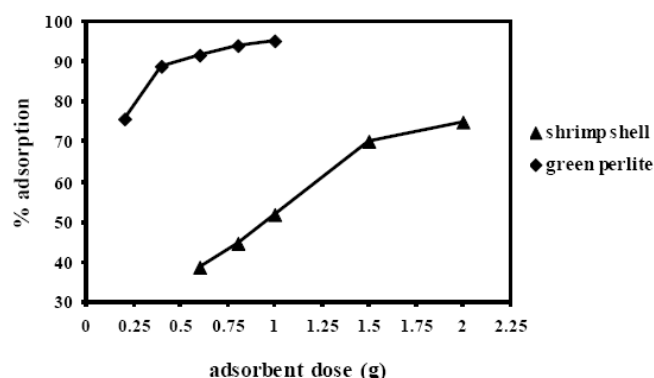

Fig. 3. effect of adsorbent dose on dye adsorption

D. Adsorption Isotherm

Equilibrium isotherm equations are used to describe experimental adsorption data. The equation parameters of these equilibrium models often provide some insight into both the sorption mechanism and the surface properties and

affinity of the adsorbent. Therefore, the best–fit isotherm is required for the adsorption system.

The Langmuir model is the most widely used isotherm equation. It assumes that equilibrium is attained when a monolayer of the adsorbate molecules saturates the adsorbent. The Langmuir isotherm in nonlinear and linear form are represented as follows:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \qquad (3)$$

$$\frac{C_e}{q_e} = \frac{1}{q_m} C_e + \frac{1}{K_L q_m} \qquad (4)$$

When C_e (mg/l) is the equilibrium concentration, q_e (mg/g) is the amount adsorbed at equilibrium, K_L is the Langmuir constant and q_{max} (mg/g) is the maximum adsorption capacity. The values of q_{max} and K_L are calculated from the slope and intercept of the straight line plotting of C_e / q_e versus C_e.

The essential characteristics of a Langmuir isotherm can be expressed in terms of a dimensionless separation factor or equilibrium parameter, R_L which is defined by

$$R_L = \frac{1}{1 + K_L C_o} \qquad (5)$$

When C_O = initial dye concentration(mg/L) and K_L = Langmuir constant (L/mg). The value of R_L indicates the shape of the isotherm to be either unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$) or irreversible ($R_L = 0$).

The Freundlich isotherm describes equilibrium on heterogeneous surfaces and hence does not assume monolayer capacity. The Freundlich isotherm in nonlinear and linear form are represented as follows:

$$q_e = K_F C_e^{1/n} \qquad (6)$$

$$\log q_e = \log K_F + 1/n \log C_e \qquad (7)$$

When K_F is the adsorption capacity and $1/n$ is the adsorption intensity. $1/n$ and K_F are calculated from slope and intercept of the Freundlich plot. It has been shown that $1/n$ value between 0 and 1 represents a good adsorption. A value for $1/n$ below one indicates a normal Langmuir isotherm while $1/n$ above one is indicative cooperative adsorption [14].

The Temkin isotherm assumes that the heat of adsorption of all the molecules in a layer decreases linearly with surface coverage of the adsorbent due to sorbate–adsorbate interactions. This adsorption is characterized by a uniform distribution of binding energies. The nonlinear and linear form of the Temkin isotherm can be expressed as follows:

$$q_e = B \ln (A_T . C_e) \qquad (8)$$

When $B = \dfrac{RT}{b_T}$

$$q_e = \frac{RT}{b_T} \ln A_T + \frac{RT}{b_T} \ln C_e \qquad (9)$$

When B is the Temkin constant related to heat of sorption. A_T (L/mg) is the Temkin isotherm constant, b_T (J/mole) is a constant related to the heat of adsorption, R is the universal gas constant (8.314 J/mol) and T is the absolute temperature (K).
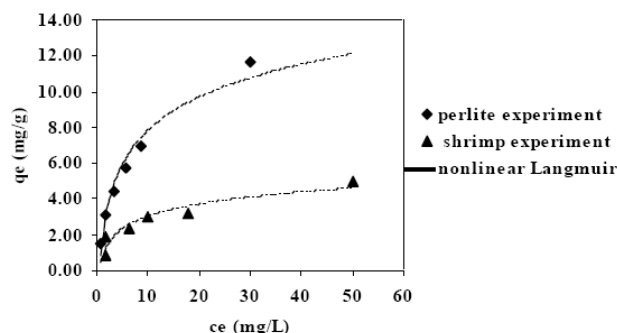

Fig. 4. Isotherm of adsorption.

The isotherm constants b_T and A_T are calculated from the slope and intercept of the q_e versus $\ln C_e$ plot.

Fig. 4 shows plot of experimental data between q_e versus C_e using nonlinear Langmuir equation with Excel solver. The results showed the adsorption capacity increased with increase in initial dye concentration. The initial dye concentration provides an important driving force to overcome all mass transfer resistances of the surface adsorbent between the aqueous and solid phases. Also, as the adsorption process precedes the driving force decrease with time, indicating that there is a saturation limit for the adsorbents [15]. Similar observations were reported by adsorption of cadmium on sugarcane bagasse [16], adsorption of chromium (VI) by ethylenediamine–modified cross-linked magnetic chitosan resin [17], adsorption of anionic dye on natural untreated clay [18].

Fig. 5 and Table I show values of three isotherms investigated at initial dye concentrations of 10–100 mg/L.

The experimental data were described by Langmuir, Freundlich and Temkin isotherms. Due to the linear correlation coefficient, the experiment data of dye adsorption onto green perlite and shrimp shell were better fitted by Langmuir than Freundlich and Temkin isotherms as in Table I. The results indicated the homogeneous nature by the monolayer coverage on surface between sorbed molecule and the maximum adsorption capacity valued 14.085 mg/g for green perlite and 5.621 mg/g for shrimp shell. Similar observation was reported by adsorption of anionic dyes onto natural untreated clay [18]. The effect of Langmuir isotherm shape could be used to predict whether adsorption system favorable or unfavorable. The separation factor (R_L) of adsorption onto green perlite and shrimp shell valued

0.071−0.277 and 0.071−0.278, respectively. The value of $0 < R_L < 1$ indicated that the adsorption of dye on adsorbent was favorable. For this work, the adsorption of dye onto green perlite and shrimp shell was favorable. From Freundlich isotherm, the valued for $1/n$ below one indicated a normal Langmuir isotherm and reflected the favorable adsorption [19]. And the value of RT/b_T (less than 20 kJ/mol) which is an indication of heat of adsorption obtained from Temkin isotherm indicated the adsorption was physi−sorption [20]. Similar observation was reported by the adsorption of malachite green onto bentonite [21].
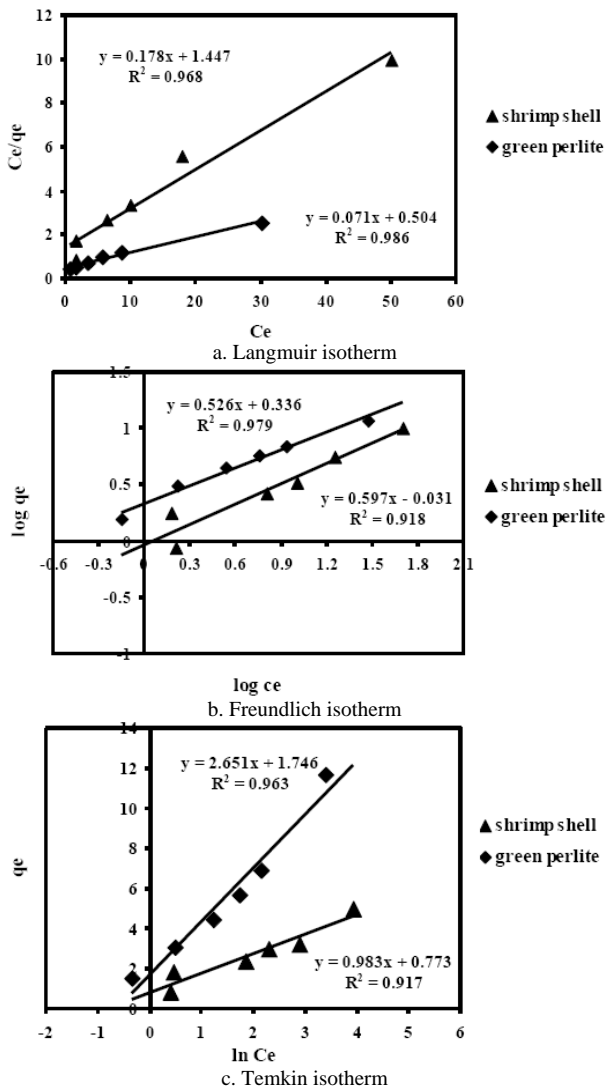

a. Langmuir isotherm

b. Freundlich isotherm

c. Temkin isotherm

Fig. 5. Isotherm models of adsorption.

TABLE I: ADSORPTION ISOTHERMS

Isotherms model	Values	
	green perlite	shrimp shell
Langmuir isotherm		
K_L (L/mg)	0.141	0.123
q_{max} (mg/g)	14.085	5.618
R_L	0.124–0.415	0.140–0.449
R^2	0.986	0.968
Freundlich isotherm		
K_F (L/mg)	2.169	2.107
$1/n$	0.526	0.597
R^2	0.979	0.918
Temkin isotherm		
B: RT/b_T	2.651	0.983
A (L/mg)	1.932	2.195
R^2	0.963	0.917

E. Adsorption Kinetic

In order to investigate the mechanism of adsorption, kinetic models of adsorption have been used to test experimental data. The pseudo−first order kinetic in a linear form is written as follows:

$$\log (q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \quad (10)$$

When k_1 (min^{-1}) is the rate constant of pseudo−first order adsorption, q_e (mg/g) is the amount adsorbed at equilibrium and q_t (mg/g) is the amount adsorbed at any time (min). The pseudo−second order kinetic in a linear form is written as follows:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (11)$$

When k_2 (g.mg^{-1}.min^{-1}) is the rate constant of pseudo−second order adsorption. Fig. 6 shows the linear pseudo−first order kinetic and pseudo−second order kinetic of adsorption onto the adsorbents at initial dye concentration 20 mg/L.
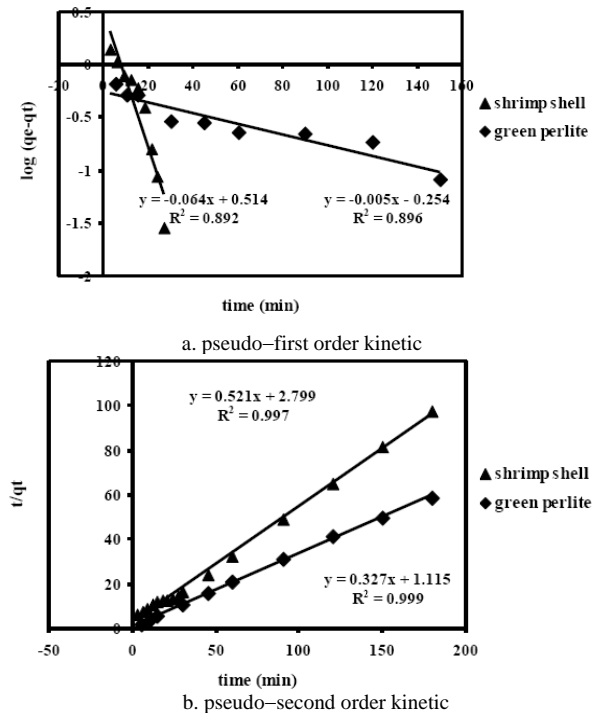

a. pseudo−first order kinetic

b. pseudo−second order kinetic

Fig. 6. kinetic model of adsorption.

TABLE II: KINETIC MODELS OF DYE

Kinetic model	Values	
	green perlite	shrimp shell
q_e (mg/g) exp.	3.077	1.840
first−order		
q_e (mg/g)	1.092	3.265
k_1 (min^{-1})	0.263	1.184
R^2	0.896	0.892
second−order		
q_e (mg/g)	3.106	1.919
k_2 (g.mg^{-1}.min^{-1})	0.078	0.098
R^2	0.999	0.997

The kinetic adsorption of dye on two adsorbents for initial dye concentration at 20 mg/L reached the equilibrium at 180 min for green perlite and 30 min for shrimp shell. As seen in Table II, the correlation coefficients of kinetic adsorption of dye onto green perlite and shrimp shell were 0.896 and 0.892 for pseudo−first order and 0.999 and 0.997 for pseudo−second order. Based on the correlation coefficient, the kinetic data were fitted better with pseudo−second order. The equilibrium adsorption capacities calculated by pseudo−second order (q_e, cal) valued 3.106 and 1.919 mg/g for green perlite and shrimp shell, respectively. As seen from Table II, the q_e calculated by pseudo−second order equation was closer to the experimental q_e. The pseudo−second order model is based on the assumption that the reaction is chemi−sorption involving the exchange or sharing of ions between adsorbent and adsorbate [22]. Similar observations were reported by adsorption of anionic dyes onto organo−bentonite [9] and adsorption of dye on shrimp shell [3].

F. Thermodynamic Adsorption

The thermodynamic parameter such as the Gibbs free energy change(ΔG), enthalpy change (ΔH) and entropy change(ΔS) were studied to understand better the effect of temperature on the adsorption. The experiments were performed at equilibrium time using 20 mg/L dye solutions at 25, 30 and 35 °C. The Gibbs free energy of adsorption (ΔG) can be expressed as follows:

$$\Delta G = -RT \ln K_C \qquad (12)$$

When K_C ($= [C_0 - C_e]/C_e$) is the equilibrium constant, R is the gas constant (8.314 J/mol) and T is the absolute temperature (K). The enthalpy (ΔH) and entropy (ΔS) changes can be estimated by van't Hoff equation as follows:

$$\ln K_C = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \qquad (13)$$

$$\log K_C = \frac{\Delta S}{2.303R} - \frac{\Delta H}{2.303RT} \qquad (14)$$

The graph of log K_C versus $1/T$ is shown in Fig. 7 and thermodynamic parameters of adsorption onto green perlite and shrimp shell are shown in Table III.

As shown in Fig. 7, the free energy of dye adsorption on green perlite and shrimp shell for initial dye concentration 20 mg/L at the temperature 25, 30 and 35°C were −5.464, −6.103, −6.776 and −5.721, −6.145, −6.576 kJ/mol, respectively. Whereas the temperature was raised from 25 to 35°C , the adsorption capacity (not shown) increased from 3.002 mg/g to 3.113 mg/g for green perlite and from 1.819 mg/g to 1.858 mg/g for shrimp shell. The negative value of ΔG indicated that the dye adsorption was spontaneous in nature. Generally, the Gibbs free energy changes for physical and chemical adsorption are in the range of 0.0 to 20 kJ/mol and 80 to 400 kJ/mol, respectively. Therefore, the adsorption of the dye onto green perlite and shrimp shell can be considered to physi−sorption in nature.

The enthalpy changes (ΔH) of dye adsorption on green perlite and shrimp shell were 18.286 and 10.722 kJ/mol, respectively. Positive value of ΔH indicated the endothermic nature of the process. The entropy change (ΔS) of dye adsorption on green perlite and shrimp shell were 0.080 and 0.056 kJ/mol, respectively. Positive value of ΔS indicated the increasing randomness at the solid/liquid interface during the adsorption process. Similar observation was reported by the adsorption of congo red from aqueous solutions onto Ca−bentonite [13].
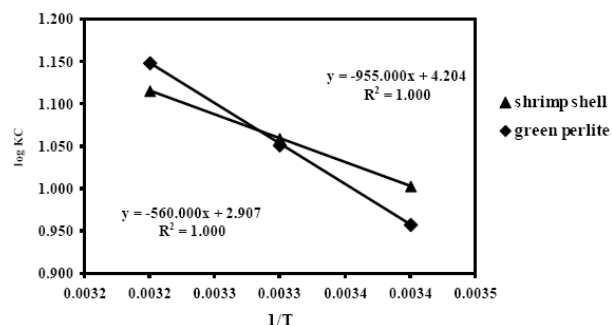

Fig. 7. Thermodynamic of dye adsorption onto green perlite and shrimp shell.

TABLE III: THERMODYNAMIC PARAMETERS

Adsorbents	Temperature (K)	-ΔG (kJ/mol)	ΔH (kJ/mol)	ΔS (kJ/mol)
green perlite	298	5.464	18.286	0.080
	303	6.103		
	308	6.776		
shrimp shell	298	5.721	10.722	0.056
	303	6.145		
	308	6.576		

IV. CONCLUSION

In this paper, investigation of the equilibrium adsorption of a magenta dye for silk yarn onto low−cost adsorbents, green perlite and shrimp shell, was carried out at 30 °C in batch process. The initial rate of adsorption of dye onto green perlite and shrimp shell was high at the first 30 min, then gradually approached the plateau. The adsorption reached equilibrium time at 180 min for green perlite and 30 min for shrimp shell. The Langmuir, Freundlich and Temkin model were used to fit the equilibrium data and estimated model parameters but the overall data was slightly better fitted by the Langmuir isotherm. The maximum adsorption capacities value 14.805 mg/g for green perlite and 5.621 mg/g for shrimp shell. The separation factor (R_L) indicated that the adsorption was favorable. The kinetic studies showed that the adsorption of dye onto green perlite and shrimp shell followed pseudo−second order model. Thermodynamic analysis confirmed that the adsorption was a physi−sorption process. This study proved that green perlite and shrimp shell can be used as an efficient adsorbents for the removal of dye in water. Moreover, it can be concluded the use of green perlite and shrimp shell as adsorbents could contribute not only to solve the environmental problems but also to decrease the overall cost of wastewater treatment.

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