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RESEARCH ARTICLE

Removal of malachite green from water by *Firmiana simplex* wood fiber

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Keywords: adsorption, kinetics, malachite green, thermodynamics, wood fiber.

Abbreviations: MG: malachite green

This study shows that wood fiber of Phoenix tree (*Firmiana simplex*) is an effective adsorbent for malachite green (MG). MG sorption behavior onto the wood adsorbent was investigated in this study. Basic condition was favorable for MG adsorption to the adsorbent. The pseudo second order equation well described MG adsorption onto the wood adsorbent. The Freundlich Isotherm could describe the sorption data. The positive value of ΔH^0 showed that adsorption of malachite green onto the wood adsorbent was endothermic. The negative values of ΔG at various temperatures indicate the spontaneous nature of the adsorption process.

Malachite green (MG), a triphenylmethane dye, is used as an antifungal, anti-bacterial, and anti-parasitical therapeutic agent in aquacultures and animal husbandry. It is also widely used as a direct dye for silk, wool, jute and leather. Malachite green has detrimental effects on liver, gill, kidney, intestine and gonads of aquatic organisms (Srivastava et al. 2004). When it was inhaled or ingested by human, it may cause irritation to the gastrointestinal tract and even cancer (Garg et al. 2004). Contact of malachite green with skin causes irritation with redness and pain. Intermediate products after degradation of MG are also reported to be carcinogenic (Srivastava et al. 2004). Therefore, the use of malachite green in aquaculture was banned in many countries. However, MG in fishes, animal milk and other foodstuff is still detected due to illegal use of MG (Srivastava et al. 1995), which alarm the health hazards against human being.

Adsorption is the most commonly used method for color removal. Adsorption onto activated carbon is widely practiced for removal of dissolved dyes from wastewater. However, adsorbent-grade activated carbon is cost-prohibitive. So it is necessary to seek some alternative low-cost adsorbents that do not need pretreatment to replace activated carbon (Popuri et al. 2007; Vieira et al. 2007). Recently, a number of low-cost adsorbents for dye removal from mineral wastes (Yener et al. 2006), agricultural wastes, microbial biomass (Aksu, 2005) and higher plant biomass (Ho et al. 2005) were reported in the literature. Among them, biomaterials from higher plants seem to be one type of popular low-cost adsorbents because they usually have higher biomass compared with microbes and are easily available. For example, tree fern (Ho et al. 2005), orange peel (Arami et al. 2005), date pits (Banat et al. 2003), palm kernel fiber, sawdust (Garg et al. 2004), peanut hull (Gong et al. 2005), neem leaf (Bhattacharyy et al. 2003) and de-oiled soya (Mittal et al. 2005) were tested for treatment of dye-bearing wastewaters with different success.

Phoenix tree (*Firmiana simplex*) is a species of deciduous tree, which grows very fast and is widely spread in China. Its wood had no economic value. The aim of this study was to clarify adsorption behavior of phoenix tree wood fiber for removal of MG from water. The equilibrium, kinetics and thermodynamics of adsorption of MG from water to wood fiber were investigated.

MATERIALS AND METHODS

Preparation of adsorbent and dye solution

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Wood fiber of phoenix tree (*Firmiana simplex*) was powdered and sieved through a 100-mesh sieve. The powder was soaked in distilled water overnight and rinsed with several times till the wash water contains no color, monitored by a UV-vis spectrometer. The woody powder was air dried and stored in a desiccator for use. pH of the adsorbent was determined by Gindl and Tschegg's method (2002). Other physicochemical properties were characterized by adopting the standard procedures.

Analytical reagent MG, CI=42000, chemical formula= C50H52N4O8, λ_{max} = 617 nm, purity over 99% was obtained from Shanghai Chemicals Co., China. Since the dye has high purity, we did not take the effect of impurity into account in our study. 1 g L^{-1} stock solution was prepared with deionized water. All working solutions used in tests were prepared by appropriately diluting the stock solution to a pre-determined concentration. All other chemicals used in this study were analytical reagent.

Sorption studies

The adsorption of malachite green onto the adsorbent was investigated in batch experiments.

Effect of initial solution pH

The effect of pH on dye malachite green sorption was evaluated by adding 0.25 g of adsorbent into flasks containing 100 ml of 100 mg L^{-1} malachite green solutions at different initial pH (3-11). pH of the solutions was adjusted using 0.1M HCl/NaOH. The adsorbent was added after MG solution pH was fixed. Flasks containing the adsorbent and MG solution were shaken at 300 rpm and 25°C for 180 min. Initial and equilibrium pH of solutions and residual malachite green concentrations were measured.

Effect of adsorbent dosage

Effect of adsorbent dosage was studied by adding different adsorbent doses (0.05-0.25 g) into flasks containing 100 ml of 100 mg L^{-1} malachite green solutions. The pH of the solutions was preadjusted to 7 according to the result of the study on effect of pH. Flasks were shaken at 300 rpm and 25°C for 180 min. Initial and equilibrium pH of solutions and residual malachite green concentrations in solutions were measured.

Kinetic study

For kinetic sorption experiment, 1.25 g of dry adsorbents were added to flasks containing 500 ml of 100 mg L^{-1} malachite green-bearing solution with pH adjusted to 7. Flasks were shaken at 300 rpm at predetermined temperature. Aliquot amounts of solution were taken, periodically. Residual malachite green concentrations in solutions were measured.

Isothermal and thermodynamic study

For isotherm analysis, adsorption experiments were conducted by varying the initial malachite green concentration from 10 mg/l to 500 mg L^{-1}. 0.25 g of dry adsorbent were added to flasks containing 100 ml of malachite green-bearing solution with pH preadjusted to 7. Flasks were shaken at 300 rpm and predetermined temperature for 180 min. Residual malachite green concentrations in solutions were measured.

Analytical methods

The samples were centrifuged and malachite green concentrations in supernatant were determined by measuring the absorbance using a spectrophotmeter (UV-2000, Unico, Shanghai, China). pH was measured using a pH meter.

RESULTS AND DISCUSSION

Physicochemical properties of the adsorbent

The physico-chemical characteristics of the adsorbent were: apparent density 1.36 g/ml; surface area 226.5 m^2/g; Cation exchange capacity (CEC) 0.87 meq/g dry matter; pH 5.1; and EC 0.11 mS/cm.

Effect of initial pH

Figure 1 shows that adsorption percentage of MG increased with increasing pH. The maximum of adsorption

Table 1. Pseudo-first and pseudo-second-order constants and values of R^2 for MG adsorption to wood adsorbent.

$q_{e,exp}$ (mg g^{-1})	First order			Second order				
	$q_{1,cal}$ (mg g^{-1})	k_1 (min^{-1})	R^2	$q_{2,cal}$ (mg g^{-1})	k_2 (g mg^{-1} min^{-1})	u (mg g^{-1} min^{-1})	$t_{1/2}$ (min)	r
36.0	11.98	0.022	0.85	36.38	0.0064	8.52	4.29	0.999

Table 2. Isothermal parameters of ethyl violet adsorption onto the wood adsorbent.

Temperature (K)	Freundlich parameters		
	K_F(mg g^{-1})	*1/n*	*r*
278	0.288	0.919	0.975
288	0.457	0.878	0.978
298	0.706	0.746	0.975
308	0.813	0.714	0.977

percentage of malachite green was observed at pH 11. At lower pH, the number of positively charged adsorbent surface sites increased at the expense of the number of negatively charged surface sites. The carboxylic groups of MG (p*K*a = 10.3) were protonated and had high positive charge density at a lower pH (Garg et al. 2003; Crini et al. 2007). Consequently, electrostatic repulsion between the positively charged surface and the positively charged dye molecule increased with increasing solution pH and resulted in the decreasing of adsorption capacity of MG to the adsorbent with increasing of pH. In addition, the competition of H^+ with the cationic dye molecules due to the presence of excess H^+ also decreased the adsorption (Porkodi and Kumar, 2007). On the contrary, the surface of the adsorbent was negatively charged at higher pH, which favored for adsorption of the positively charged dye cations through electrostatic force of attraction. The adsorption of MG to wood fiber consequently increased with increasing of pH values.

A similar trend was observed for the adsorption of MG to cyclodextrin-based adsorbent (Crini et al. 2007), anaerobic granular sludge (Cheng et al. 2008), de-oiled soya (Mittal et al. 2005), hen feathers (Mittal, 2006), rattan sawdust (Hameed and El-Khaiary, 2008a; Hameed and El-Khaiary, 2008b) and rice straw.

Effect of adsorbent dosage. It is evident from Figure 2 that the removal percentage of malachite green increased on the increasing of the adsorbent dosage. This can be attributed to the increase in surface area with a high dosage of the adsorbent. On the contrary, the sorption capacity decreased from 142.4 mg/g^{-1} to 35.6 mg/g^{-1} as adsorbent dosage increased from 0.05 g to 0.25 g. The decrease in sorption capacity may be attributed to the splitting effect of flux (concentration gradient) between sorbate and adsorbent, with increasing adsorbent concentration causing a decrease in amount of malachite green adsorbed onto unit weight of adsorbent.

Sorption kinetics

Study of sorption kinetics can provide important information on sorption rate and the factors affecting the sorption rate, which is extremely important in designing batch sorption systems. Time courses of malachite green adsorption onto woody fiber were given in Figure 3a. In the present study, pseudo-first order and pseudo-second order models were employed to analyze the kinetics of malachite green adsorption onto the adsorbent.

First-order model

The pseudo-first order equation of Lagergren is generally expressed as follows (Ho and McKay, 1999):

$$dq_t/dt = k_1(q_1-q_t) \qquad (1)$$

where q_1 and q_t are the amount of malachite green adsorbed per unit weight of adsorbent at equilibrium and at time t, respectively (mg/g) and k_1 the rate constant of pseudo-first order sorption (l/min). given the boundary conditions for t=0, q_t=0, the equation(1) can be integrated to give (Ho and McKay, 1999)

$$log(q_1-q_t) = logq_1 - (k_1/2.303)t \qquad (2)$$

If the sorption process can be described by pseudo-first order equation, there should be good linear relationship between $log(q_1-q)$ and t.

In the present study, the plot of $log(q_1-q_t)$ versus time t and the relationship was not linear over the entire time range (Figure 3b), indicating that more than one mechanism involved in adsorption. The $q_{1,\ cal}$ obtained from first-order equation was much different from the expected values (q_{exp}) (Table 1).

Table 3. thermodynamic parameters of ethyl violet sorption onto the wood adsorbent

ΔH^0 (kJ mol^{-1})	ΔS^0 (J mol^{-1})	ΔG(KJ mol^{-1})			
		278K	288K	298K	308K
61.4	234.3	-3.65	-5.99	-8.33	-10.67

Second-order model

If the rate of sorption is a second order mechanism, the pseudo-second order chemisorption kinetic rate equation is expressed as (Ho and McKay, 1999)

$$dq_t/dt = k_2(q_2-q_t)^2 \qquad (3)$$

Where q_2 is the amount of malachite green adsorbed at equilibrium (mg g^{-1}), k_2 is pseudo-second order rate constant (g mg^{-1} min^{-1}), and q_t is the amount of malachite green adsorbed per unit weight of adsorbent at time t. After integrating and applying boundary conditions for t=0, q_t=0, equation (3) becomes

$$t/q_t = 1/(k_2q_2^2)+ t/q_2 \qquad (4)$$

The rate constant k_2 can be obtained from the intercept of the linearized pseudo-second order rate equation. If the pseudo-second order equation can fit the sorption data, there should be good linearity between t/q_t and t. when $t\rightarrow 0$, the initial sorption rate u can be defined as

$$u=k_2q_2^2 \qquad (5)$$

Half-adsorption time ($t_{1/2}$) is the time required for the adsorption to take up half as much malachite green as its equilibrium value. This time is an indicator for the adsorption rate. It was calculated from the following equation:

$$t_{1/2}= 1/k_2q_2 \qquad (6)$$

Figure 3c showed that the pseudo-second order equation was satisfactorily applicable to all the sorption data (r=0.999). The pseudo second-order sorption constants were summarized in Table 1. The $q_{2,\ cal}$ calculated from second-order equation was close to the expected values.

MG adsorption kinetics to cyclodextrin-based adsorbent (Crini et al. 2007), *Pithophora* spp. (Kumar et al. 2005) and carbon (Zhang et al. 2008) were also reported to follow the pesudo second-order equation.

Sorption isotherms

The Freundlich isotherm is a nonlinear model and is shown to be consistent with exponential distribution of active centres, characteristic of heterogeneous surfaces. It is usually expressed as follows:

$$q_e=k_FC_e^{1/n} \qquad (7)$$

where q_e is the amount of malachite green adsorbed, mg/g^{-1}(dry mass); C_e is the equilibrium malachite green concentration in solution, mg L^{-1}; k_F and n are rate constants, being indicative of the extent of adsorption and the degree of nonlinearity between solution and concentration, respectively. A high value of n is indicative of good adsorption over the entire range of concentrations studied, while small n is indicative of good adsorptin at high concentrations but much less at lower concentrations. A higher value of k_F indicates a higher capacity for adsorption than a lower value.

The Freundlich equation describing malachite green adsorption by the wood adsorbent was illustrated in Figure 4, and the Freundlich constants calculated from the linear equations were summarized in Table 2. The Freundlich equations could describe the sorption. However, Kumar and Sivanesan (2007) demonstrated that sorption process of malachite onto rubber wood could not well represented by the linear Freundlich equation but could be well described by the nonlinear Freundlich equation.

The values of the exponent 1/n were in the range of 0-1, indicating favorable adsorption at all temperatures tested. The value of K_F increased on increasing of temperature, indicating that higher temperature favored malachite green sorption onto the wood adsorbent. The MG sorption capacity to wood fiber increased on increasing of the temperature. At an initial malachite green concentration of 100 mg/l, the sorption capacity steadily increased from 32.6 mg/g^{-1} at 278 K to 36.6 mg/g^{-1} at 308 K, indicating that the process is endothermic in nature and higher temperature was favorable for malachite green adsorption onto adsorbent.

Thermodynamic studies

Analysis of thermodynamics of equilibrium sorption data can give more important information on sorption process. In the present study, thermodynamic parameters were calculated by using the equation (8)

$$lnK_d= \Delta S^0/R - \Delta H^0/RT \qquad (8)$$

where K_d is the distribution coefficient (ml/g^{-1}), ΔH^0 , ΔS^0, and T are the enthalpy, entropy, and temperature in kelvin, respectively, and R is the gas constant. ΔH^0 and ΔS^0 were

Table 4. Comparison of adsorption capacities of various adsorbents for MG

Adsorbent	q_m or experimental sorption capacity (mg g^{-1})	Temperature (°C)	Reference
Bamboo-based activated carbon	263.6	30	Hameed and El-Khaiary, 2008a
Activated carbon	466-565	32-60	Kumar, 2006
Groundnut shell based activated carbon	222.2	30	Malik et al. 2007
Commercial activated carbon	8.27	30	Mall et al. 2005
Laboratory grade activated carbon	42.18	30	Mall et al. 2005
Arundo donax root carbon	8.69	30	Zhang et al. 2008
Bentonite	178.6	25 ± 2	Bulut et al. 2008
rattan sawdust	62.7	25	Hameed and El-khaiary, 2008b
Rubber wood sawdust	25.8-36.3	-	Kumar and Sivanesan, 2007
Functionalized sawdust	85.5-196.1	20-40	Gong et al. 2009
Dead tree leaves	77.5-89.4	25-45	Hamdaouia et al. 2008
Hen feather	10.3-10.7	30-50	Mittal, 2006
Lemon peel	3.2-51.7	32	Kumar, 2007
Jute fiber carbon	136.58	30	Porkodi and Kumar, 2007
Firmiana simplex wood fiber	> 142.4	25	This study

*Unless stated; LOQ = Limit of quantification, lactic acid = 0.03%; mean value and standard deviation of three determinations are presented.

obtained from the slope and intercept of the plot of $\ln K_d$ against $1/T$ (Figure 5). Gibbs free energy ΔG was calculated using the equation (9)

$$\Delta G = \Delta H^0 - T\Delta S^0 \qquad (9)$$

The values of the thermodynamic parameters for the sorption of malachite green onto adsorbent are given in Table 3. The positive value of ΔH^0 showed that adsorption of malachite green onto the wood adsorbent was endothermic. The negative values of ΔG at various temperatures indicate the spontaneous nature of the adsorption process. ΔG decreases with increased temperature indicated that the adsorption was more favorable at higher temperature. The positive value of ΔS^0 indicated that the adsorption process was irreversible and random at the solid/liquid interface during the sorption of malachite green onto the wood adsorbent. In addition, the positive value of ΔS^0 suggested some structural change of malachite green and the wood adsorbent (Gupta, 1998) and favored complexion and sorption stability (Donat et al. 2005). Similar results were also observed on carbon prepared from *Arundo donax* root (Zhang et al. 2008) and de-oiled soya (Mittal et al. 2005), bentonite (Bulut et al. 2008) and hen feathers (Mittal, 2006).

Comparison with other adsorbents for MG

Many adsorbents for MG removal, including activated carbon, various biosorbents, minerals, were reported in the literature (*e.g.*, Mall et al. 2005; Kumar, 2006; Malik et al. 2007; Bulut et al. 2008; Zhang et al. 2008). Part of the data of MG sorption capacity (values of q_m derived from the Langmuir equation) of various adsorbents, especially the low-cost adsorbents, was summarized in Table 4. In the present study, the experimental value was used due to

failure of the Langmuir equation to describe the isothermal sorption data. It was found that *Firmiana Simplex* wood fiber is an excellent adsorbent for MG. Activated carbon, sometimes, was more effective in sorption of MG than this woody adsorbent. However, activated carbon is cost-prohibitive since a great deal of energy would be consumed during production of activated carbon. On the contrary, *Firmiana simplex* wood almost costs nothing since it has little economic value and can be directly used as adsorbent without further procedures. In this sense, *Firmiana simplex* wood fiber is an excellent adsorbent for MG.

CONCLUDING REMARKS

The wood adsorbent was an excellent adsorbent for malachite green. The sorption kinetics followed the pseudo second order equation, indicating that several processes were involved in malachite green sorption onto the wood adsorbent. Basic condition was favorable for MG adsorption to the adsorbent. The Freundlich Isotherm satisfactorily described the sorption data. The MG adsorption was a spontaneous endothermic process.

Since *Firmiana simplex* grows widely in the world and rapidly with great biomass, has little economic values, and has excellent adsorption capacity for MG, *Firmiana simplex* wood fiber should be a promising and cost-effective adsorbent for MG removal in industry. Further studies on quantitative characterization of this adsorbent and involved mechanisms, and feasibility of using this adsorbent for other triphenylmethane dyes and for its possible industrial application are needed.

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APPENDIX

FIGURES

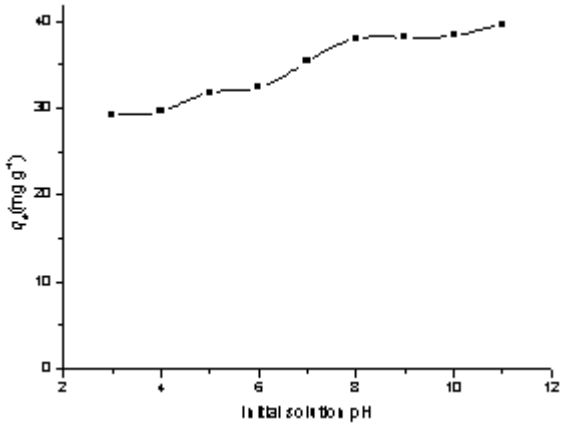

Figure 1. Effect of initial pH on MG removal by the wood fiber.

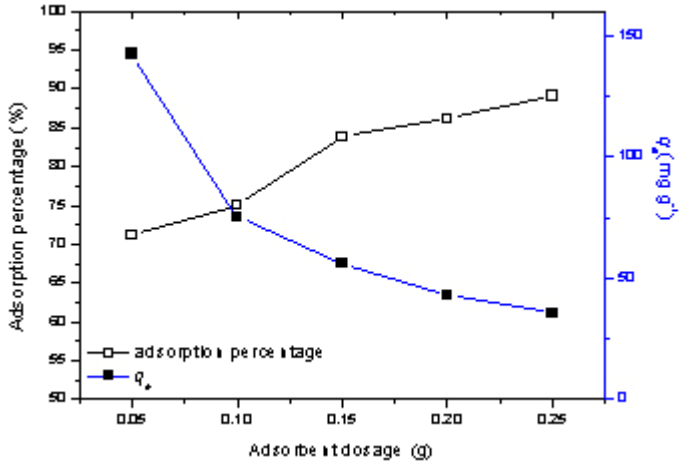

Figure 2. Effect of adsorbent dosage on MG adsorption percentage and sorption capacity.

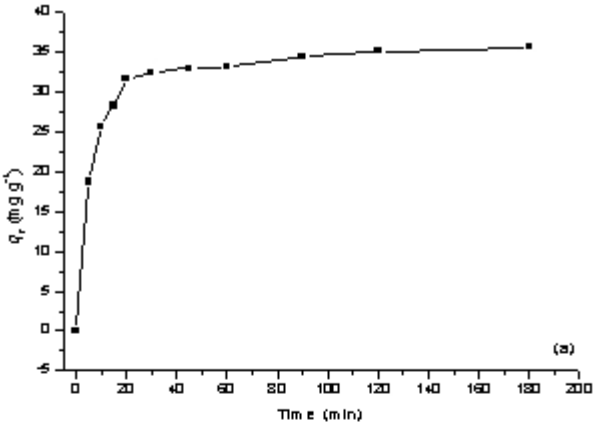

Figure 3 (a)

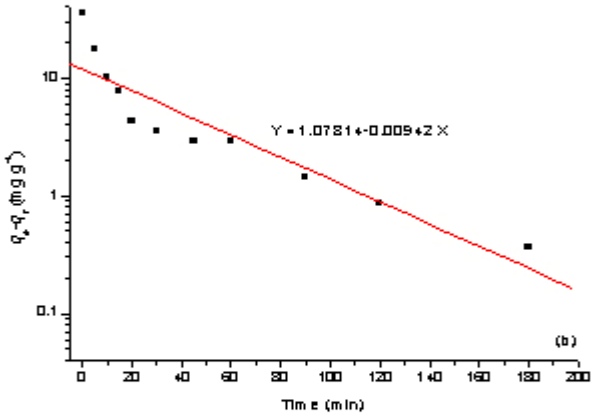

Figure 3 (b)

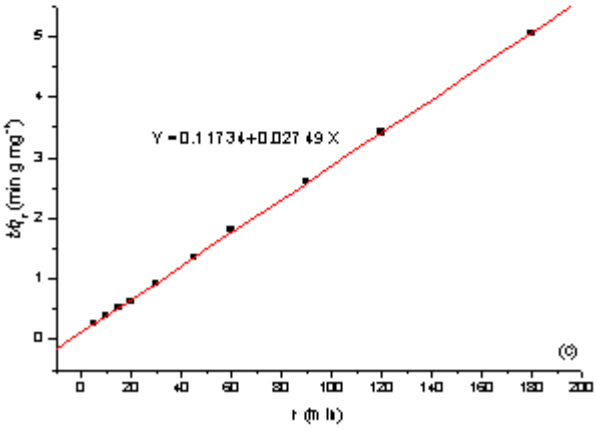

Figure 3 (c)

Figure 3. Time courses of MG adsorption onto the wood fiber.

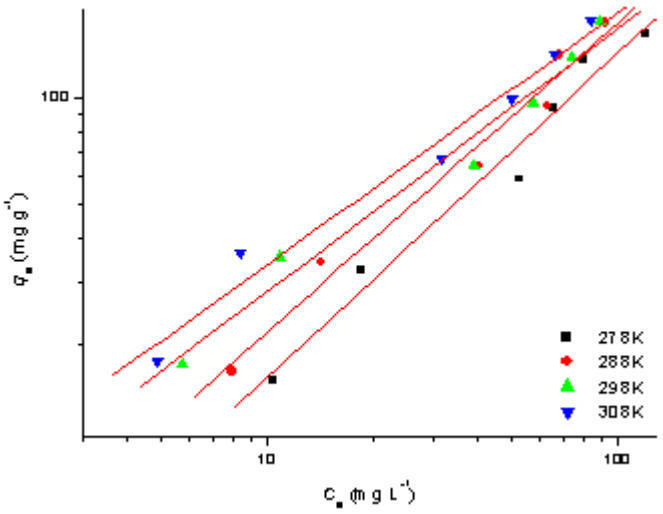

Figure 4. Freundlich Isotherm for the adsorption of MG to the wood fiber.

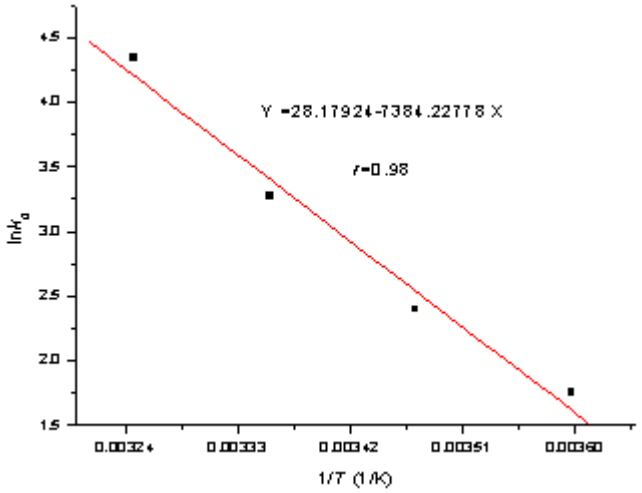

Figure 5. The plot of *lnk* against *1/T* (1/K).