

## **“Removal of Hazardous Crystal Violet from Aqueous Media by Coffee-Husk Biomass: Equilibrium Behavior, Adsorption Kinetics, and Thermodynamic Assessment”**

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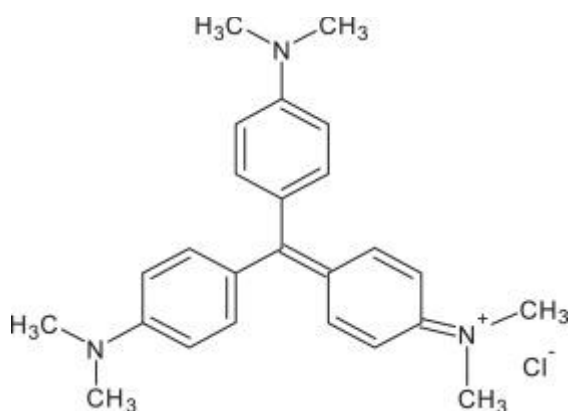
## Abstract:

Crystal violet (CV) is a cationic triphenylmethane Water-soluble dye which has the ability to persist in aquatic systems and has also been linked to negative health and environmental effects. The paper is a waste coffee husk (WCH) biomass that is examined as a low-cost and renewable biosorbent to remove CV in aqueous media through the batch adsorption experiment carried out at the University of Baghdad. Contact time, initial dye concentration, dose of adsorbent systematically investigated, particle size, initial pH, and temperature were also investigated. The uptake of CV was fast at low contacts and high decolorization was seen in the presence of acid and at ambient temperature. Higher doses of the WCH increased the percentage removal and decreased mass-normalized capacity whereas smaller particle sizes increased the removal since they had more surface area and decreased diffusion length. The higher the temperature the lower adsorption became, which was in favor of an exothermic reaction. Kinetic modelling indicated that the pseudo-second-order model accounted experimentally for the experimental results significantly better than the pseudo-first-order model. Both Langmuir and Freundlich isotherms were in equilibrium over the concentration range studied. The thermodynamic parameters recorded feasibility and spontaneity (negative  $\Delta G$  also known as negative  $\Delta G$ ), exothermicity (negative  $\Delta H$  also known as negative  $\Delta H$ ) and increased interfacial randomness (positive  $\Delta S$ ). The findings justify the use of WCH as a cheap and non-toxic biosorbent in the process of dye-bearing wastewater remediation.

**Keywords:** Crystal violet; Coffee husk; Biosorption; Kinetics; Isotherms; Thermodynamics; Wastewater treatment.

## 1 Introduction

Synthetic dyes find extensive applications in textiles, paper, plastics, leather, pharmaceuticals, etc., and a non-negligible part of them may find its way into the water during processing and pollute its quality, as well as restrict the penetration of light in even small amounts (Asad et al., 2007; Singh et al., 2015; Srikantan et al., 2018). Crystal violet (CV) is a cationic triphenylmethane dye (Figure 1) that has not only been used in the process industry and laboratory staining but has also been linked with irritation and a more considerable damage, which has prompted effective wastewater treatment plans (Sarma et al., 2016; Mittal et al., 2010). Traditional dye-removal techniques (e.g., biological treatment, oxidation, coagulation/flocculation, membrane filtration) can be characterized as expensive, semi-complete, or difficulties in handling the remnant (Gupta et al., 2010; Allen and Koumanova, 2005; Yagub et al., 2014). Activated carbon often is expensive and thus they tend to seek other inexpensive options such as clays, biochars, and farm waste (Adsorption) is often favored because it operates with a simple method and is economically effective (Ahmad, 2009; Mittal et al., 2010), but there has been an effort to find cheaper alternatives like clays, biochars, and agricultural wastes as adsorbent materials (Ahmad et al., 2012; Alshabanat et al., 2013; Gürses et al., 2006; Hameed et al., 2007). The reason why agricultural residues are appealing adsorbing materials is related to their availability and lignocellulosic properties (cellulose, hemicellulose, lignin) that may react with the dye molecules (Kushwaha et al., 2014). Coffee processing also produces large amounts of husk material that have no high-value application, thus such materials can be used in the environmental remediation (Bulut et al., 2007). Western blotting assay is used to evaluate WCH as a bio biosorbent in the removal of CV, to optimize the important parameters of the batch and to rationalize the process by using kinetic, equilibrium, and thermodynamic models (Lagergren, 1898; Ho & McKay, 1998).



**Figure 1: The molecular (chemical) structure of the crystal violet (CV) dye.**

## 2 Materials and methods

### 2.1 Preparation of the adsorbent material.

Husks of coffee waste were gathered and thoroughly washed using tap water to get any impurities stuck on them, and then dried under the sun over a few days. The dried material was milled in to powder and rinsed several times in distilled water and dried in the oven at 60 to constant mass. They stressed the sieving of the adsorbent to get particle-size fractions of 0.15-0.30, 0.30-0.60, 0.60-1.118, 1.118-2.36, and 2.36-4.75 mm to be used in further adsorption tests.

### 2.2 Preparation of the adsorbate solution.

Stock crystal violet (C<sub>25</sub>H<sub>30</sub>N<sub>3</sub>Cl) solution (c. 1.0 g/L) was weighed into a stock bottle using twice-distilled water, and then diluted to the desired levels to create batch experiments. The pH of the solution was adjusted by using 0.1 M. The new level of dye concentration was quantified by UV-Avis spectrophotometry at λ<sub>max</sub>=580nm.

### 2.3 Adsorption testing procedures.

The experiments of the batch adsorption were carried out in 250 mL Erlenmeyer flasks, in 50 mL of CV solution and a given amount of mass WCH. The flasks were tubed using a mechanical shaker at a speed that was around 140 rpm until equilibrium. There were repeated and average experiments. Aliquots were taken and measured at given times in order to analyze the concentration of dye that was left.

After calculating the adsorption capacity at equilibrium,  $q_e$  (mg/g), and the removal percentage (%) were determined through the utilization of Equations. (1) and (2):

$$q_e = \frac{(C_0 - C_e)V}{W}, \quad (1)$$

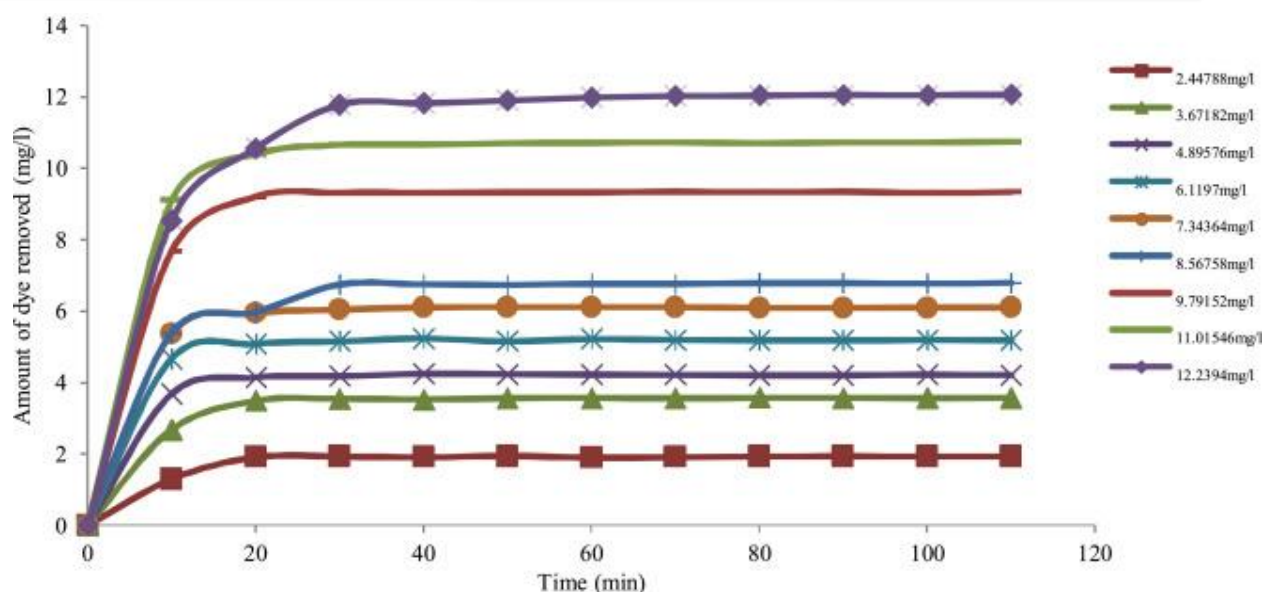
$$\%R = \frac{(C_0 - C_e)}{C_0} \times 100, \quad (2)$$

where  $C_0$  and  $C_e$  are initial and equilibrium CV concentrations (mg/L),  $V$  is solution volume (L), and  $W$  is WCH mass (g).

## 3 Results and discussion

### 3.1 Influence of exposure time and the starting dye concentration.

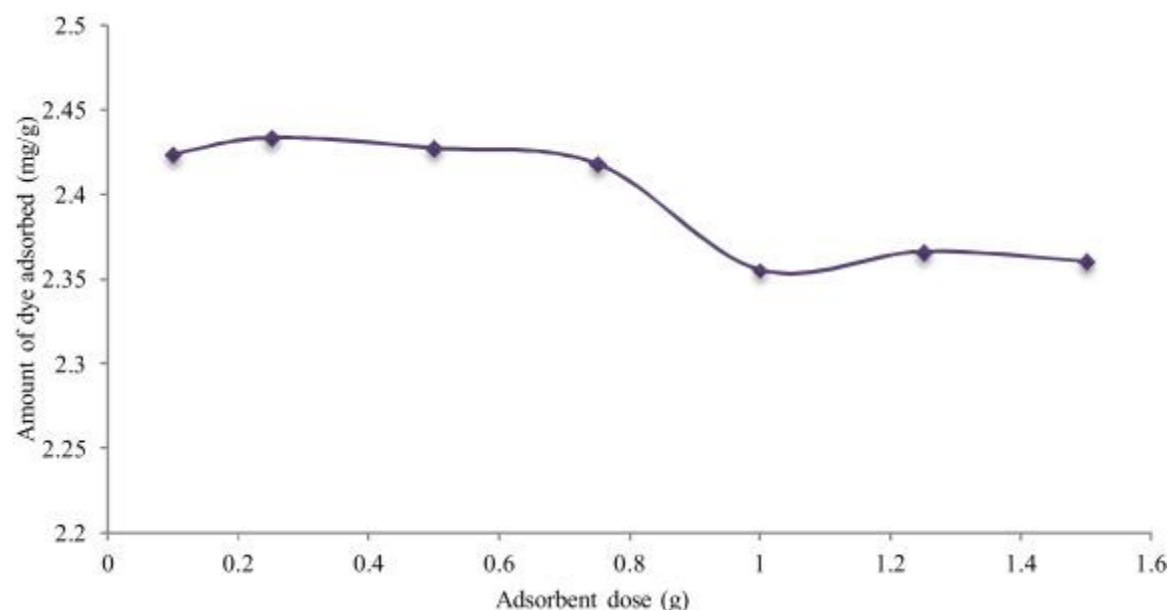
The concentration of CV was greatly affected by the time of contact where the initial rapid adsorption occurred and then the approach to equilibrium was gradual (Figure 2). Its rapid initial uptake may be explained by the presence of a large number of binding sites and the subsequent slow phase may be explained by the saturation of binding sites and diffusion barriers) Bulut & Aydin, 2006; Wang & Li, 2013). Additional concentration of initial CV increased the quantity of adsorbent since more quantity gradient was created to promote mass transfer to the biosorbent surface (Wang & Li, 2013). Similar rapid-then-plateau behaviour has been reported in other CV-based adsorption studies on biosorbents based on low-cost materials (Ahmad et al., 2017; Pavan et al., 2014; Saeed et al., 2010).



**Figure 2:** Influence of the starting CV concentration on its adsorption (WCH: 0.25 g; 50 mL; 25°C; pH 4).

### 3.2 Influence of adsorbent dosage.

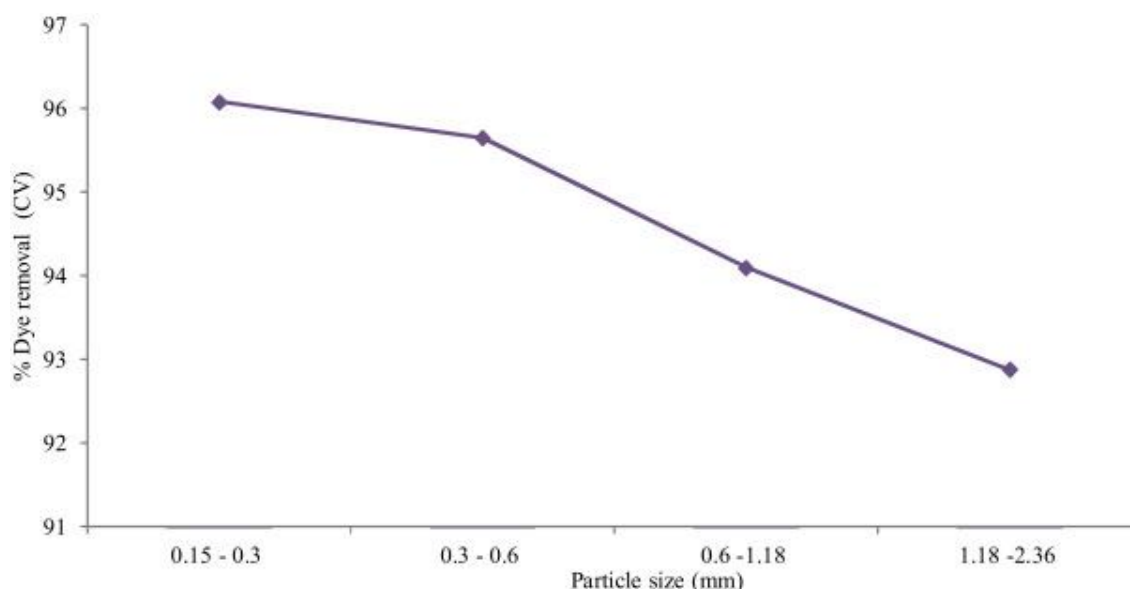
The dosage of adsorbent predetermines the adsorbent/sorbate ratio and influences the process and cost of operation (Crini, 2006; Wang & Li, 2013). With the increase in the dose of WCH, percent removal was as well as equilibrium was reached faster (Zhou et al., 2010) (Figure 3). Contrastingly, the adsorption capacity in terms of mg/g declined in the higher dosages which are normally linked to particle aggregation as well as overlap of adsorption sites that minimize effective use of the surface (Robinson et al., 2001).



**Figure 3: Influence of adsorbent dosage on CV removal (initial dye concentration: 12.2394 mg/L; 50 mL; 25°C; pH 4).**

### 3.3 Influence of particle size.

Figure 4 showed better CV removal with smaller particle size. The best fraction of WCH fraction had the best removal as it had a larger external surface area and shorter intraparticle diffusion length and, therefore, greater accessibility to functional adsorption sites [19, 27].



**Figure 4: Influence of particle size on CV removal (WCH: 0.25 g; 50 mL; initial dye concentration: 12.239 mg/L; 25°C; pH 4).**

### 3.4 Influence of the solution's initial pH.

PH of the solution controls the adsorbent surface charge and electrostatic interaction between the adsorbent and the cationic dye, thus affecting the uptake (Khattari & Singh, 2009). CV elimination was preferred in acidic environments, and the best results were achieved at pH of about 3 (Figure 5). The adsorption decreased with increased pH, which was in line with adsorption being unfavorable to electrostatic interactions and the competitive effects on the interface (Nasuha et al., 2010; Tahir & Rauf, 2006)

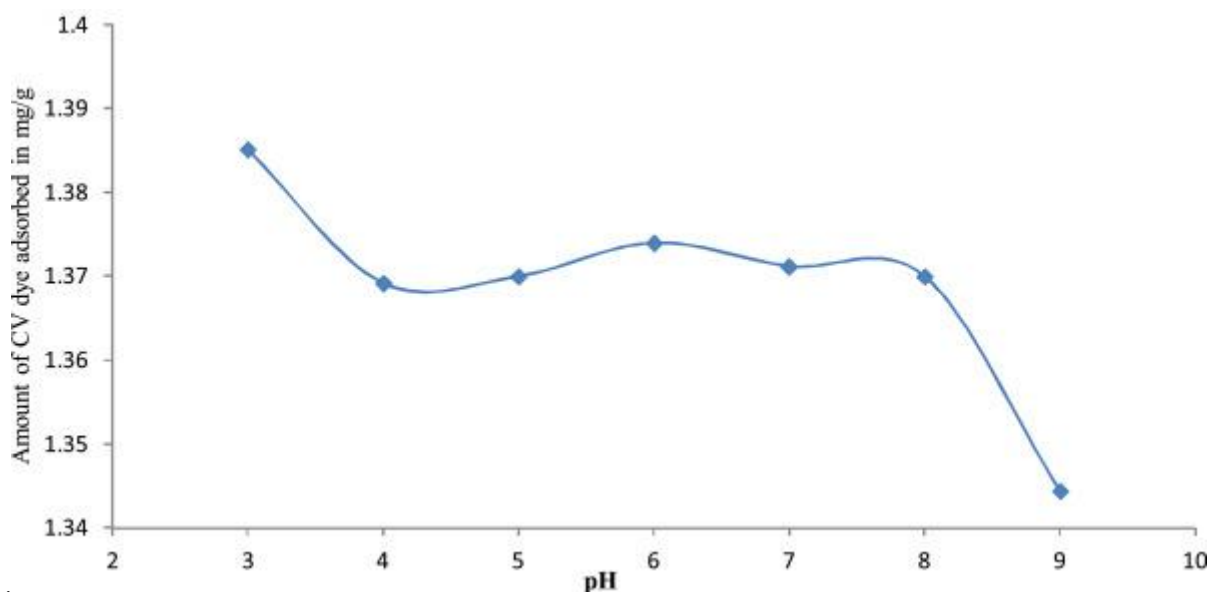


Figure 5: Effect of pH on CV removal (WCH: 0.25 g; 50 mL; initial dye concentration: 12.2394 mg/L; 25°C).

### 3.5 Influence of temperature variation.

Temperature influences the movement of dye, solution viscosity and favorability of energy of adsorption (Hamdaoui, 2006). In the current experiment, the adsorption capacity decreased with raising the temperature (Figure 6), which showed the exothermic mode of adsorption. The increased temperatures probably led to looseness of binding forces and resulted in partial desorption (Dogan et al., 2009). Likewise exothermic behavior has been observed in CV adsorption of different biomass-based sorbents (Ahmad et al., 2017; Nasuha et al., 2010).

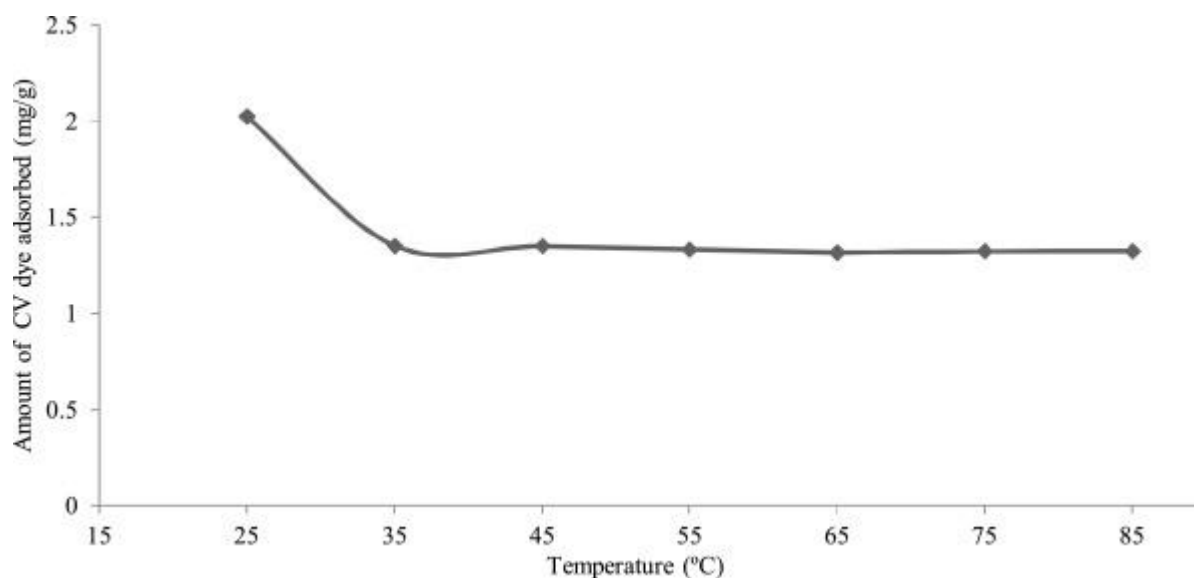


Figure 6: Influence of temperature on crystal violet adsorption (WCH: 0.25 g/50 mL; initial dye concentration: 12.2394 mg/L; particle size: 0.15–3.0 mm).

### 3.6 Adsorption kinetics analysis.

In wastewater applications, it is important to also have rapid kinetics besides the necessary capacity (Forgacs et al., 2004). Pseudo-first-order (PFO) and pseudo-second-order (PSO) models [33 and 34] were used to analyze the kinetics data and are common and used significantly to explain adsorption rate behavior (Weber & Morris, 1963).

The linear PFO form is:

$$\log(q_e - q_t) = \log(q_e) - \frac{k_1}{2.303} t, \quad (3)$$

where  $k_1$  is the PFO rate constant, and  $q_t$  and  $q_e$  are adsorption capacities at time  $t$  and at equilibrium.

The PSO model is:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e}, \quad (4)$$

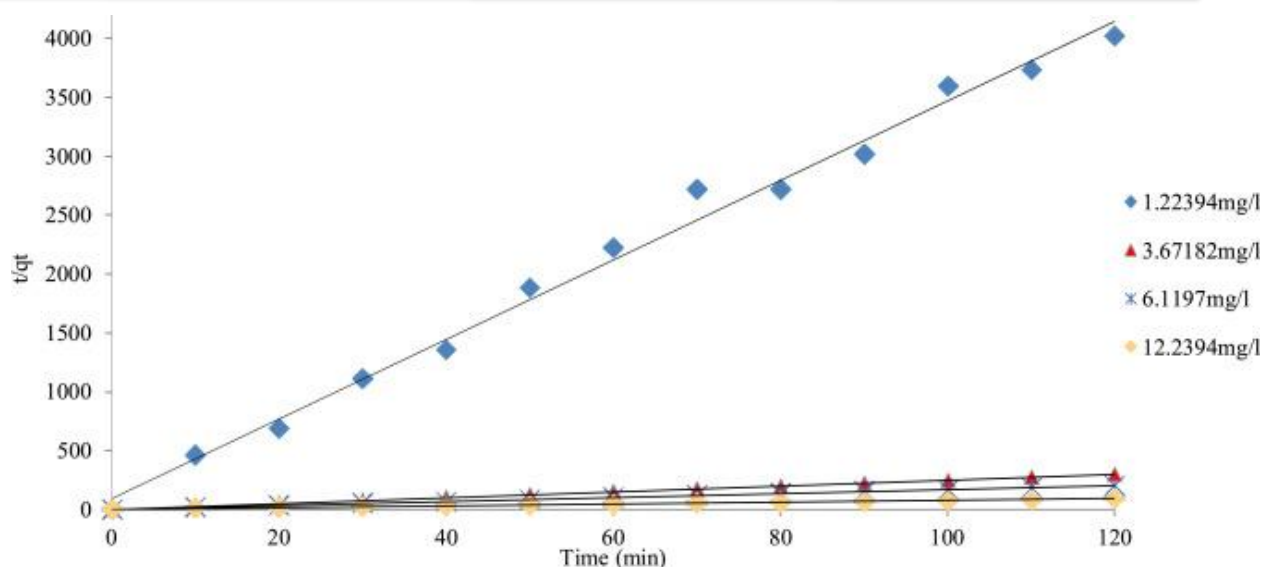
where  $k_2$  is the PSO rate constant. The PSO model provided excellent agreement with the experimental data ( $R^2 > 0.99$ ), supporting a rate description consistent with stronger surface interactions (Weber & Morris, 1963; Wang & Li, 2013). The fitted parameters are summarized in Table 1. The PSO plots are shown in Figure 7.

**Table 1: Kinetic model data for crystal violet removal using waste coffee husk biomass.**

CV conc. (mg/L)	Pseudo-first-order				Pseudo-second-order		
	$q_{e,exp}$ (mg/g)	$q_e$ (mg/g)	$k_1$	$R^2$	$q_e$ (mg/g)	$k_2$	$R^2$
5 (lr)2-6-8							
1.224	0.030	0.023	0.004	0.005	0.030	0.0001	0.992

.672	0.406	6.761	0.048	0.768	0.410	0.155	0.999
.120	0.589	9.099	0.030	0.013	0.591	2.520	0.999
.016	1.220	5.848	0.048	0.771	1.227	2.962	0.999
.239	1.368	4.406	0.037	0.299	1.397	0.495	0.999





**Figure 7: Pseudo-second-order kinetic plot for CV adsorption onto WCH.**

### 3.7 Adsorption equilibrium analysis.

The adsorption isotherms explain how molecules of the dye are distributed between the solid and a liquid phase at equilibrium and are needed in optimizing the process (Weber & Morris, 1963). Langmuir (Langmuir, 1918) and Freundlich (Freundlich, 1906) models were studied in determining the equilibrium data.

The linear Langmuir form is:

$$\frac{1}{q_e} = \frac{1}{q_0 b} \cdot \frac{1}{C_e} + \frac{1}{q_0} \quad (5)$$

where  $q_0$  is the monolayer capacity and  $b$  is the Langmuir affinity constant. The separation factor is:

$$R_L = \frac{1}{1 + bC_0} \quad (6)$$

where  $0 < R_L < 1$  generally indicates favorable adsorption [39].

The linear Freundlich form is:

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \quad (7)$$

where  $K_F$  and  $n$  are the adsorption capacity and intensity correspondingly (Hall et al., 1966). Figures 8 and 9 are the plots of the isotherms and Table 2 is the summary of the fitted constants. Both models were highly linearized within the concentration range that was tested and it has been reported to be so in similar systems of biosorption (Dubinin & Radushkevich, 1947).

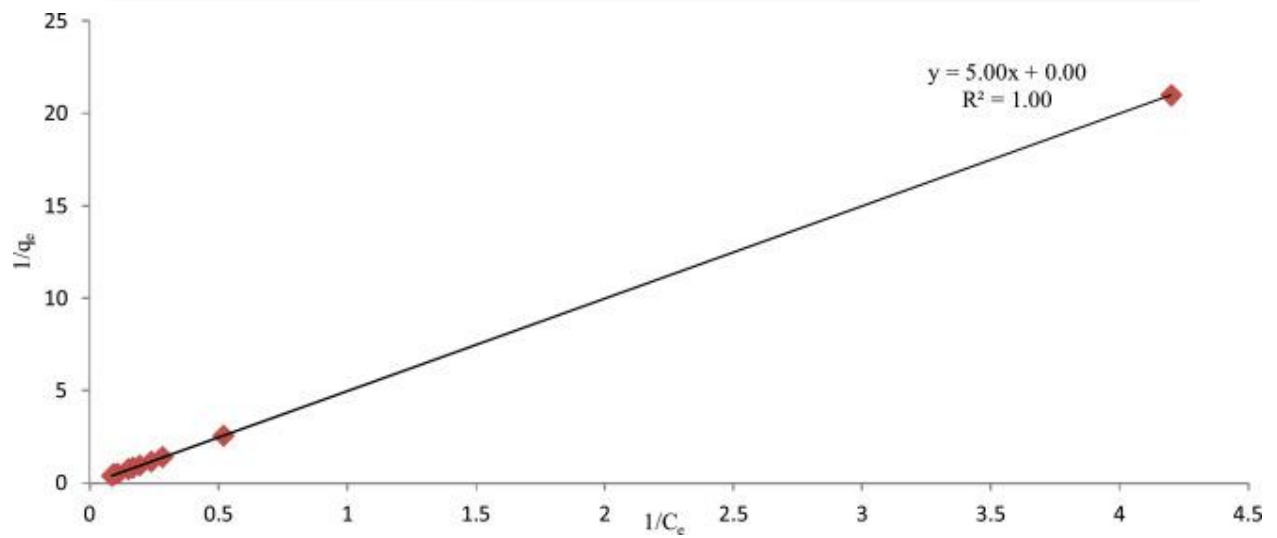


Figure 8: Langmuir isotherm plot for CV adsorption onto WCH.

Table 2: Langmuir and Freundlich isotherm parameters for CV adsorption onto WCH.

Langmuir adsorption isotherm				Freundlich adsorption isotherm		
(lr)1-4	$b$	$R^2$	$R_L$	$K_F$	$n$	$R^2$
(lr)5-7 $q_0$						
0	0	1	1	4.998	1	1

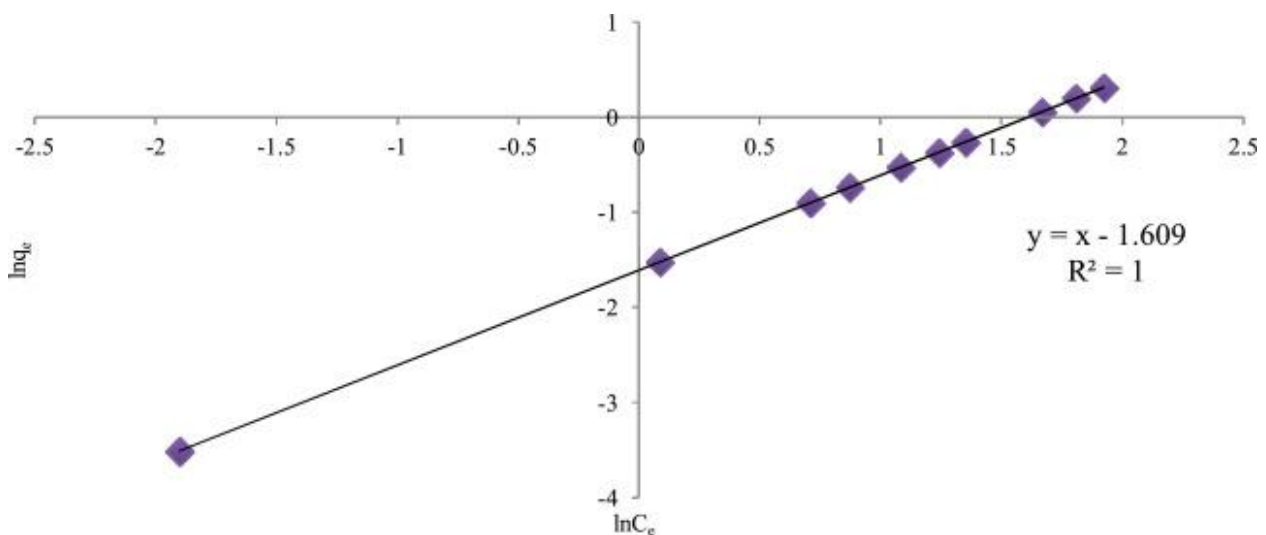


Figure 9: Freundlich isotherm plot for CV adsorption onto WCH.

### 3.8 Thermodynamic analysis

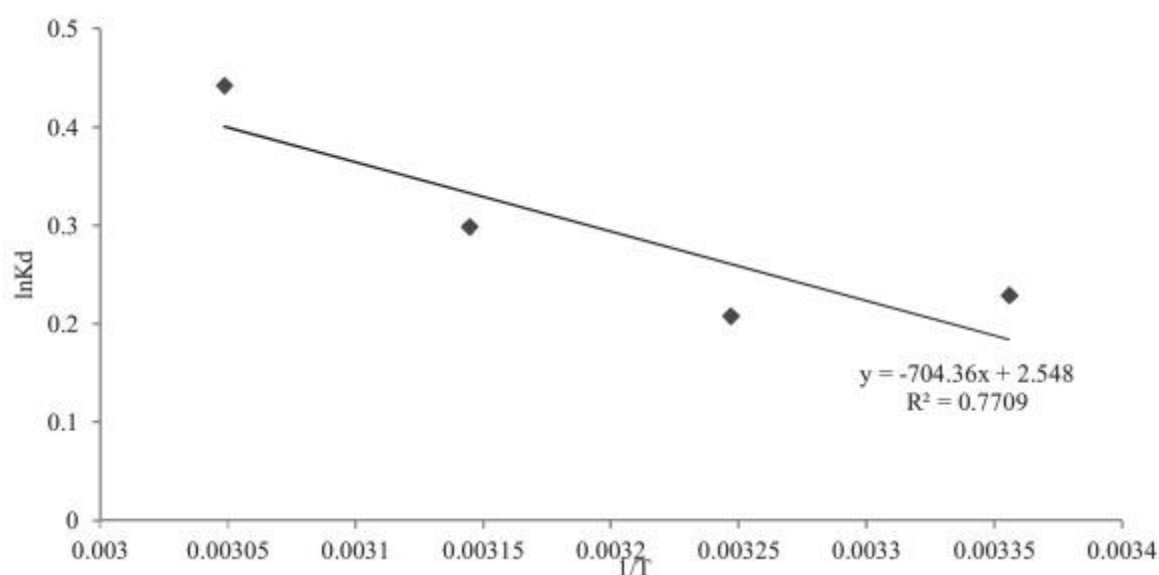
Thermodynamic parameters were estimated to interpret feasibility and energetic characteristics of adsorption. The standard Gibbs free energy is:

$$\Delta G^\circ = -RT \ln K_d, \quad (8)$$

where  $R$  is the gas constant and  $T$  is absolute temperature. A Van't Hoff relationship was used:

$$\ln K_d = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT}. \quad (9)$$

The distribution coefficient was expressed as  $K_d = C_{solid}/C_{liquid}$  at equilibrium, and  $\Delta H^\circ$  and  $\Delta S^\circ$  were obtained from the slope and intercept of the  $\ln K_d$  versus  $1/T$  plot (Figure 10). Then,  $\Delta G^\circ$  was computed using Eq. (8). The resulting parameters (Table 3) indicate spontaneous adsorption (negative  $\Delta G^\circ$ ), exothermicity (negative  $\Delta H^\circ$ ), and increased interfacial disorder (positive  $\Delta S^\circ$ ), consistent with related CV adsorption reports (Nasuha et al., 2010; Temkin & Pyzhev, 1940).



**Figure 10:** Graph of  $\ln K_d$  versus  $1/T$  (K<sup>-1</sup>) for CV sorption on WCH (0.25 g per 50 mL; starting CV level: 12.239 mg/L).

**Table 3:** Thermodynamic indicators describing CV uptake by WCH.

$R^2$	$\Delta H^\circ$ (kJ/mol)	$\Delta S^\circ$ (J/mol·K)
0.770	-5.856	+21.184

Note: Reported  $\Delta G^\circ$  values across the tested temperature range were negative (approximately  $-0.002$  to  $-0.004$  kJ/mol), indicating feasibility and spontaneity under the examined conditions.

#### 4 Conclusion

Waste coffee husk (WCH) biomass showed a high potential in terms of being a cost-effective biosorbent in removing CV in aqueous media. It was preferred to be removed at acidic (optimal pH 3) temperature and ambient temperature, and fast uptake during the initial period of contact. Higher dose led to increased removal efficiency at the cost of reduced capacity/gram, whereas smaller particle sizes led to an improved adsorption ability because of increased surface accessibility. Higher temperature decreased adsorption and thermodynamic was used to support an exothermic and spontaneous process. Kinetic modeling indicated that there was a significant better fit between kinetic modeling and pseudo-second-order model as compared to the pseudo-first-order model and equilibrium data was also fit to the Langmuir and Freundlich description across the concentration range examined. In general, WCH provides a low-cost renewable method of treating dye-contaminated effluents.

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## "إزالة صبغة الكريستال البنفسجي الخطرة من الأوساط المائية باستخدام الكتلة الحيوية لقشور القهوة: سلوك الاتزان، حركية الامتزاز، والتقييم الترموديناميكي"

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### الملخص:

تعد صبغة الكريستال فيوليت (Crystal violet – CV) صبغة كاتيونية من نوع ثلاثي فينيل الميثان قابلة للذوبان في الماء، وتمتلك القدرة على البقاء في الأنظمة المائية، كما تم ربطها بآثار صحية وبيئية سلبية. تتناول الورقة البحثية استخدام كتلة حيوية مشتقة من قشور القهوة العادمة (Waste coffee husk – WCH) والتي تم فحصها كـممتز حيوي (biosorbent) منخفض التكلفة ومتجدد لإزالة صبغة CV من الأوساط المائية، وذلك من خلال تجربة امتزاز دفعية (batch adsorption) أجريت في جامعة بغداد. تم تقصي العوامل التالية بشكل منهجي: وقت التلامس، التركيز الأولي للصبغة، جرعة الممتز، بالإضافة إلى دراسة حجم الجسيمات، الرقم الهيدروجيني (pH) الأولي، ودرجة الحرارة. وقد لوحظ أن امتصاص صبغة CV كان سريعاً عند فترات التلامس القصيرة، كما لوحظت كفاءة عالية في إزالة اللون في الوسط الحامضي وعند درجة حرارة الغرفة. أدت زيادة جرعات قشور القهوة (WCH) إلى زيادة نسبة الإزالة وتقليل القدرة النوعية لكتلة الممتز، بينما أدت أحجام الجسيمات الأصغر إلى زيادة الإزالة نظراً لامتلاكها مساحة سطح أكبر وطول انتشار أقل. أظهرت النتائج أنه كلما ارتفعت درجة الحرارة انخفض الامتزاز، مما يرجح أن التفاعل طارد للحرارة (exothermic). وأشارت النمذجة الحركية إلى أن نموذج الرتبة الثانية الكاذبة (pseudo-second-order) كان أفضل بكثير في تمثيل النتائج التجريبية مقارنة بنموذج الرتبة الأولى الكاذبة. كما أظهرت كل من أيزوثرمات لانغمير وفريندليتش (Langmuir and Freundlich) حالة من التوازن ضمن نطاق التركيز المدروس. سجلت المعايير الديناميكية الحرارية جدوى وتلقائية العملية (قيمة سالبة لطاقة جيبس الحرة  $\Delta G$ )، وكون التفاعل طارداً للحرارة (قيمة سالبة للمحتوى الحراري  $\Delta H$ )، وزيادة في العشوائية البينية (قيمة موجبة لدرجة العشوائية/الإنتروبي  $\Delta S$ ). وتبرر هذه النتائج استخدام قشور القهوة العادمة كـممتز حيوي رخيص وغير سام في عملية معالجة مياه الصرف الصحي الملوثة بالأصبغ.