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# Removal of toxic textile dyes from aqueous solution through adsorption onto coconut husk waste: Thermodynamic and isotherm studies

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## ABSTRACT

In this research, activated carbon (coconut husk waste) is prepared using sulfuric acid activation from coconut husk waste which is a cheap material that shows agreed scavenging actions by adsorption for eliminating the toxic textile dyes (methylene blue MB, crystal violet CV, as well as Brilliant Blue BB) from the aqueous solutions. In a shaker water bath, different physio-chemical factors like contact time, adsorbent dose, pH, temperature of the dye solution and initial concentration of the dye have been measured and the adsorption time is 120 minutes. The results show that adsorption of MB, CV is favorable at a high pH value, but at acidic pH, the brilliant blue BB dye is favorable. The activated carbon thermodynamic analysis is conducted using three dyes: The Gibbs free energy, entropy and also enthalpy. According to the results, the adsorption is a Physical (endothermic). It is also found that the activated carbon is regulated by the equations of Freundlich and Temkin. Finally, field emission scanning electron microscopy (FE-SEM) and Fourier-transform infrared spectroscopy (FT-IR) have been used to show adsorption.

**Keywords:** Adsorption, Coconut husk, Activated carbon, Textile dyes, isotherm, Thermodynamic parameters.

## INTRODUCTION

Many industries such as plastics, textiles, cosmetics, pharmaceutical, paper and so on have commonly used the pigment and dye for coloring the resulting goods. In fact, textile dyeing process is an essential source of contamination that is responsible for continuous environmental pollution. (Adeyemo *et al*. 2017; Albadarin *et al.* 2018). Moreover, MB as cationic dyes has a common utilization in colouring paper, cotton dyeing, wool, leather, silk, and paper stock coating. (Aljeboree & Alshirifi 2001; Aljeboree 2015; Zaied *et al*. 2019). In spite of the commercial value of MB dye, for both toxicological and esthetical purposes, the existence of above materials in the natural environment like water and in trace amounts would be largely helpful. (Attia *et al.* 2008; Aljeboree & Alshirifi 2018; Aljeboree *et al.* 2019). Moreover, one of the popular colorants called crystal violet (CV) has been often used as one of the dermatological agents, biological stains, veterinary medicines, poultry feed additives to prevent fungi, intestinal parasite, and mold from proliferation, textile dying, paper printing, and so on. (Fabryanty *et al*. 2017). Brilliant Blue (Blue 1), else famous under commercial names, is a colorant for foods and other substances. It has the manifestation of the powder reddish-blue. It is soluble in water, Brilliant Blue belongs to toxic and recalcitrant organic pollutants (Gecgel 2012). It is often used in the production of polymeric colorants as a starting material in the textile industry. Techniques such as adsorption on agro-industrial waste materials (wheat bran), Fenton oxidation, ozonization, hydrolysis, electrochemical and bio-sorption on Candida sp. are adopted in order to remove Brilliant Blue (Saleh *et al.* 2019; Sadeghi & Nasehi 2019). The chemical structures of (MB), (CV), (BB) are shown in Fig .1.

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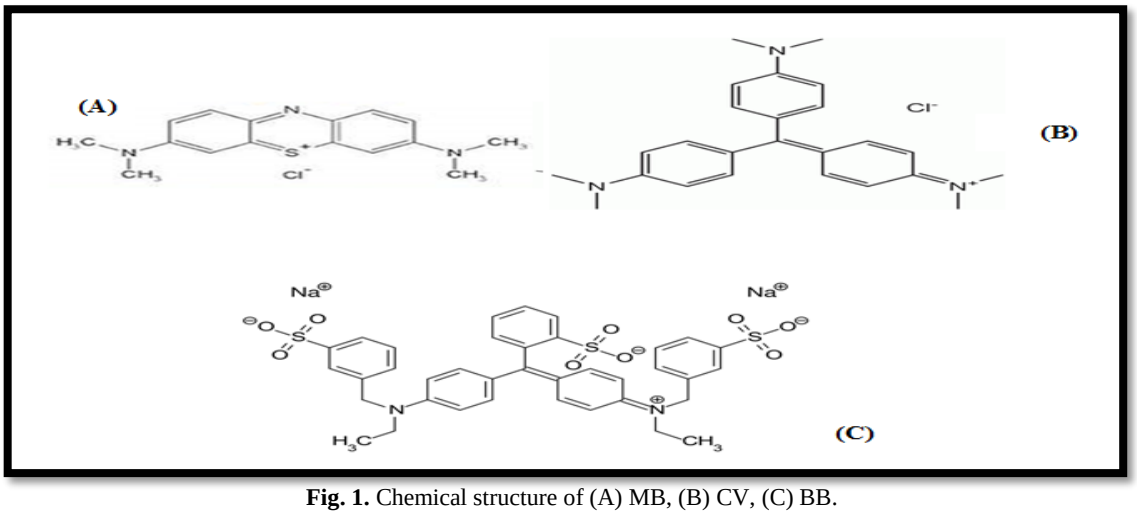


**Fig. 1.** Chemical structure of (A) MB, (B) CV, (C) BB.

## MATERIALS AND METHODS

According to the research design, we provided coconut husk from Hilla Market in Babylon, Iraq). It is air-dried, mashed, and then screened for obtaining 2 fractions with the geometrical mean dimension between 63 µm and 2.5 mm. In the next step, 100 g of the chosen fraction is impregnated with the concentrated HCl (40%) and then oven-dried at 75 ºC for 24 h. It is subsequently triggered at 400 ºC (3 h) in a hot-air oven. Finally, distilled water would be used to wash this carbonized substance for removing free acid till the activated carbon pH reaches 6.2-6.5 and is dried at 108 ºC mechanically.

### Effect of Different Parameter

Preparing the stander solution is done thought dissolving 1 gm of analytical dyes MB, CV, BB in A 1000 mL volumetric flask of distilled water. Solutions of three dyes (100 mL) is from 25-100 mg $L^{-1}$. The quantity of activated carbon in adsorption of three dyes ranges from 0.05, 0.1, 0.25, 0.5,0.8 ,0.1 and 1.5 g 100 $mL^{-1}$ .The pH range is of ( 2, 4, 6.4, and 10), the beast pH 6.4 is added to the closed bottles containing 0.05g of AC. with different solution temperature (17, 30, and 45 ºC). All the tests are performed at the optimal pH of 6.4 and the weight of the waste glass (0.5 g 100$mL^{-1}$) values placing all the solutions in the shaker in a thermostatically controlled water bath and starting to shaking for 120 minutes of stability time. Therefore, centrifuge solution separation would be performed for 15 min at 3500 rpm to measure the absorption after adsorption using the UV-Visible spectroscopic.

Amount of AC adsorbed $q_e$ and the removal percent of the three dye are determined by the following equations:

$$Q_e = (C_o - C_e) \times V\,sol \;/\; W \qquad \textbf{(1)}$$

$$Adsorption\ \% = (C_o - C_e)/C_o \times 100 \qquad \textbf{(2)}$$

where Ce and Co are the equilibrium and initial concentration of dye in the solution in mg $L^{-1}$ respectively, m is the hydrogel mass in mg and v is the solution's amount (volume)

## RESULT AND DISCUSSIONS

### FT-IR Analysis

FTIR The technique is used to analyze the surface groups responsible for three (MB, CV & BB) dyes adsorption. Adsorbent surfaces (coconut shell) and dyes-loaded adsorbent sample are put into an oven at a temperature of 80 ° C for 5 hours after adsorption. Samples are shaped as pellets and then three dyes of infrared spectra on adsorbent are observed in the range 4000–400 $cm^{-1}$ prior to and following the process of adsorption on a Bio Rad FTS 175C spectrophotometer (Aljeboree *et al*. 2020). Hydroxyl groups are usually attributed to broadband at about 3500 cm1. Axial deformation of carbonyl groups (C = O) is due to the spectrum area of 1612 $cm^{-1}$. Fig. 2 shows that

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absorption peaks do not change, but the high of FT-IR spectra beak increases after absorption, which is seen as a physical adsorption. (Cazetta *et al*. 2011; Karim & Jasim 2019; Aljeboree *et al*. 2020).

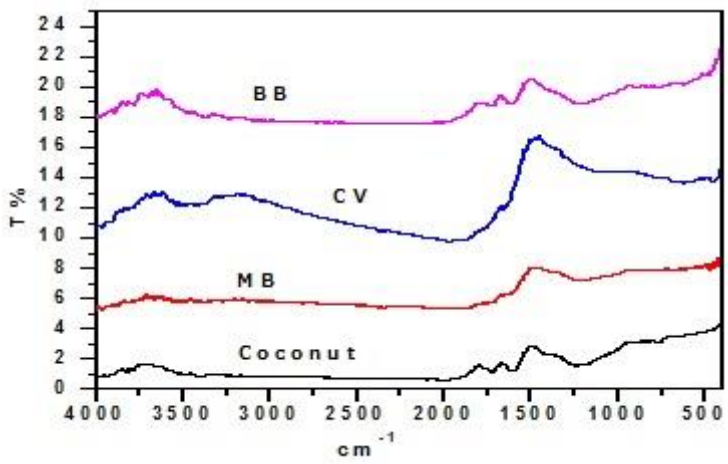


**Fig. 2.** The FT-IR spectra of coconut (a) prior to and (b) following MB (c) CV (d) BB adsorption (adsorbent dosage = 0.1 g, temperature = 290 K, rate of shaking = 120 rpm, pH=6.4, & contact time = 24 h).

**SEM characterization for adsorbent/adsorbate**

A key method for determining the characteristics of the surface morphology as well as basic physical features of the absorbent has been SEM. SEM of adsorbent content is taken on coconut shell surfaces before and after dye adsorption. (Fig. 3; Radhy & Jasim 2019).

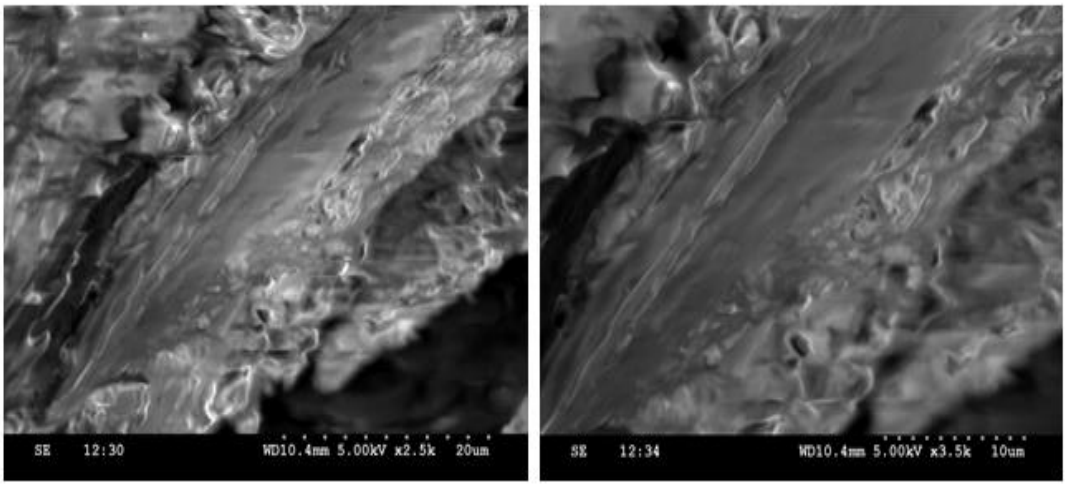


**Fig. 3.** FE-SEM for coconut shell (activated carbon) after and before adsorption.

**Adsorbent dosage**

different weight of coconut shall (activated Carbone) is taken (0.001-0.15 g), and put in initial dye concentration of 50 mg $L^{-1}$ from three dyes with volumes (100 mL), a temperature of 30˚C, at 120 rpm as shaking rate and ideal pH of 6.0 .to determine the effective weight for adsorption. All samples are left 24 h to equilibrium and all solutions are separated to measure absorption, then absorption is enhanced by elevating the weight of activated Carbone because of the increasing of functional group intel (0.05 g). After that the adsorption is approximately constant because the solution is saturated (Fig. 4; Hayati 2012). As concentration of the dye enhances, the dye amount eliminated increases. As an example, when the percent enhances from 25.1-99.5, concentration also enhanced from 0.01-0.15 g of BB dye, but by increasing concentration from 0.01-0.15 g of MB dye, the percent would enhance from 12.1-49.5%, but by enhancing concentration from 0.01-0.15 g of CV dye, an increase from 36.1 to 48.5% is observed. (Saeed & Iqbal 2010; Aksakal 2010).

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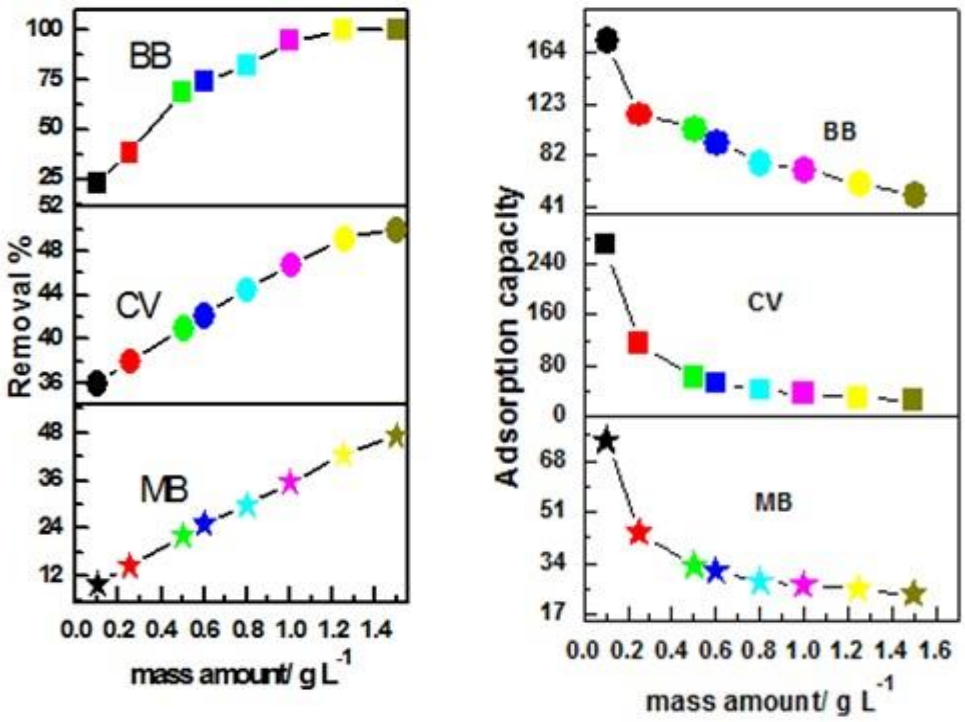


**Fig. 4.** Mass dosage effect on (MB, CV, BB) adsorption by coconut surface.
(PH = 6.4, T = 30 ºC, [dye] = 50 mg $L^{-1}$, shaking rate =120 rpm, shaking time = 24 h).

**PH effect on dye adsorption**

According to the studies, pH of the solution has been considered as one of the prominent monitoring parameters for the absorption system. PH affects the degree of adsorbate ionization as well as the surface charge of the adsorbent specimens in the solutions. Therefore, we applied 50 mg $L^{-1}$ dyes concentration with a pH between 2 and 10 at a temperature of 30 ºC, results are reported by table 1. Moreover, we determined the impact of pH on absorption. 4. Out of table 1, with increasing pH, the dye uptakes (qe) and E percent is found to increase. The decreased absorption of MB, CV at the acidic pH is possibly due to the presence of excessive H+ions vying for the sites of absorption with the cation groups on the dye (Aljeboree *et al.* 2016). The coconut surface can receive negative charges at a greater pH, which increases dye cations with the positive charges by electrostatic attraction powers. (Ehsan & Iqbal 2017; Aljeboree 2019) The maximum BB adsorption is reached at the minimum pH of 3 whereas the minimum absorption is at lowest pH of 10. Adsorption of dye BB has been shown to decrease by enhancing the solution pH increases; this is attributed to absence of $H^+$ ions, electro-static repulsion between the negative dye charges and the coconut surface thus facilitates (Aljeboree 2019). While the activated carbon groups [carboxyl groups (COOH), chronic groups & phenolic groups ($H^{+2}$)] are protonated at acidic pH (Aljeboree *et al.* 2016). The coconut surface charge becomes more positively charged at pH 2, which increases (BB) adsorption by electrostatic attraction (Aljeboree & Alkaim 2019).

**Table 1.** Effect of pH solution on Adsorption on three dyes.

| PH | E% MB | E%CV | E%BB |
|---|---|---|---|
| 2 | 22.11 | 12.76 | 99.21 |
| 4 | 33.35 | 25.54 | 82.54 |
| 6.4 | 44.12 | 35.3 | 75.21 |
| 10 | 66.68 | 50.92 | 11.98 |

**Absorption isotherm**

Dye distribution could be described by absorption isotherm when the balance is achieved. It is at a specific temperature between the solid phase and the solution. Moreover, Freundlich, Temkin and Langmuir models are

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applied to fit the balance results. Relative constants characterizing the features of surface and indicating the absorption potential of such a substance are expressed in each isothermal model. Therefore, a non-linear form of the isothermal Langmuir model (Langmuir, 1918) can be shown by:

$$q_e = \frac{q_0 K_L C_e}{1+K_L C_e} \quad \textbf{(3)}$$

Here KL (L $mg^{-1}$) represents the constant of Langmuir absorption associated with the adsorption energy, the maximum as well as equilibrium adsorption power are qo and qe (mg $g^{-1}$) respectively. Table 2 summarizes the Langmuir constant produced by the adsorption data chart of qe against Ce (Fig. 5). It is widely accepted that Freundlich isotherm follows this assumption: adsorption takes place and is therefore non-identical on unusual heterogeneous surfaces with different adsorption energy locations. For examining adhesion of the absorption mechanism to the model, the nonlinear version of the Freundlich isotherm would use.

$$qe = K_f C_e^{1/n} \quad \textbf{(4)}$$

Researchers have described Kf as a distribution or adsorption coefficient that refers to the amount of dyes absorbed to the absorbent for the unit concentration of equilibrium. Moreover, 1/n represents the factor of heterogeneity and n refers to the measure of variance of the adsorption linearity. The value set for n represents the degree of non-linearity of the solution concentration with adsorption that includes: In case of the equality of n-value to the unit, adsorption will be linear but if it is less than that of the unit, the absorption process would be chemical. In addition, if it is above the unit, the absorption process would be a desirable physical procedure. Furthermore, values of the model parameter are obtained from qe vs. Ce plot (Fig. 5) (Table 1). The deviation from linearity of Temkin isotherm model would be defined by Equation (5):

$$q_e = RT/b \ \log (K_T C_e) \quad \textbf{(5)}$$

Where the Tempkin constant (b) is adsorption heat related (kJ $mol^{-1}$), T=temperature (K), KT= maximum binding energy equilibrium binding constant associated with the Emprical Temkin constant (L $mol^{-1}$) as well as R = gas constant (8.314 J $mol.K^{-1}$). Therefore, it is possible to analyze absorption data based on Equation 5. The findings have been illustrated by Fig. 5. Table 2 displays 8 and the isothermal constants KT and b. The best isothermal fit is selected on the basis of the highest (closest to unity) value of correlation coefficient ($R^2$), which defines the isothermal experimental quality of results.

**Table 2.** Effect of different parameters isotherms for of MB, CV, BB adsorptions onto coconut surface at different temperatures.

| Isotherm | Isotherm Parameter | 290K | | | 303K | | | 318K | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | MB | CV | BB | MB | CV | BB | MB | CV | BB |
| Langmuir | Qm(mg $g^{-1}$) | 93.364 ± 1.673 | 103.188 ± 2.799 | 78.908 ± 4.701 | 43.351 ± 1.614 | 104.318 ± 7.181 | 82.126 ± 4.442 | 48.983 ± 2.993 | 110.037 ± 7.641 | 108.229 ± 2.035 |
| | KL(L $mg^{-1}$) | 0.111 ± 0.020 | 0.0254 ± 0.0013 | 0.214 ± 0.060 | 0.188 ± 0.037 | 0.0376 ± 0.006 | 0.544 ± 0.184 | 0.464 ± 0.228 | 0.045 ± 0.007 | 1.277 ± 0.199 |
| | $R^2$ | 0.9439 | 0.9982 | 0.909 | 0.9326 | 0.9877 | 0.8856 | 0.7477 | 0.9791 | 0.9863 |
| Freundlich | KF | 9.999 ± 0.553 | 5.548 ± 0.788 | 26.170 ± 1.017 | 14.283 ± 0.870 | 8.4882 ± 0.652 | 33.65 ± 1.413 | 22.122 ± 0.756 | 11.161 ± 0.801 | 55.706 ± 5.413 |
| | 1/n | 0.309 ± 0.014 | 0.601 ± 0.038 | 0.275 ± 0.01 | 0.262 ± 0.016 | 0.5393 ± 0.021 | 0.252 ± 0.009 | 0.205 ± 0.009 | 0.501 ± 0.02 | 0.209 ± 0.035 |
| | $R^2$ | 0.9870 | 0.9801 | 0.9917 | 0.9772 | 0.9923 | 0.9887 | 0.9887 | 0.9906 | 0.8609 |
| Temkin | b/J $mol^{-1}$ | 7.663 ± 0.317 | 21.246 ± 0.832 | 11.975 ± 0.826 | 7.763 ± 0.303 | 199.83 ± 90.83 | 6.719 ± 0.447 | 60.548 ± 1.046 | 0.761 ± 0.189 | 16.117 ± 1.402 |
| | KT | 1.546 ± 0.244 | 0.285 ± 0.023 | 8.723 ± 2.939 | 3.305 ± 0.575 | 0.172 ± 0.094 | 26.829 ° ± 11.2 | 0.515 ± 0.015 | 20.202 ± 1.809 | 28.692 ± 12.09 |
| | $R^2$ | 0.9039 | 0.9914 | 0.9782 | 0.9998 | 0.9876 | 0.9765 | 0.98886 | 0.9548 | 0.9543 |

**Thermodynamic parameters and temperature effects**

At different temperatures, removal of three dyes has been studied. 290 K, 303 K and 318 K to establish isothermal and thermodynamic adsorption parameters. The experiment has investigated that the amount of dye uptake increases with temperatures rising from 290 K to 318 K, diffusion rate of dye molecules is a process regulated by

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temperature. Temperature variation alters the adsorbent's equilibrium potential for a specific adsorbate. The increase in temperature in the current study results in a rapid diffusion of the dye molecules through external boundary layer of the adsorbent particles and internal pores due to less resistance in the aqueous phase of viscous forces.

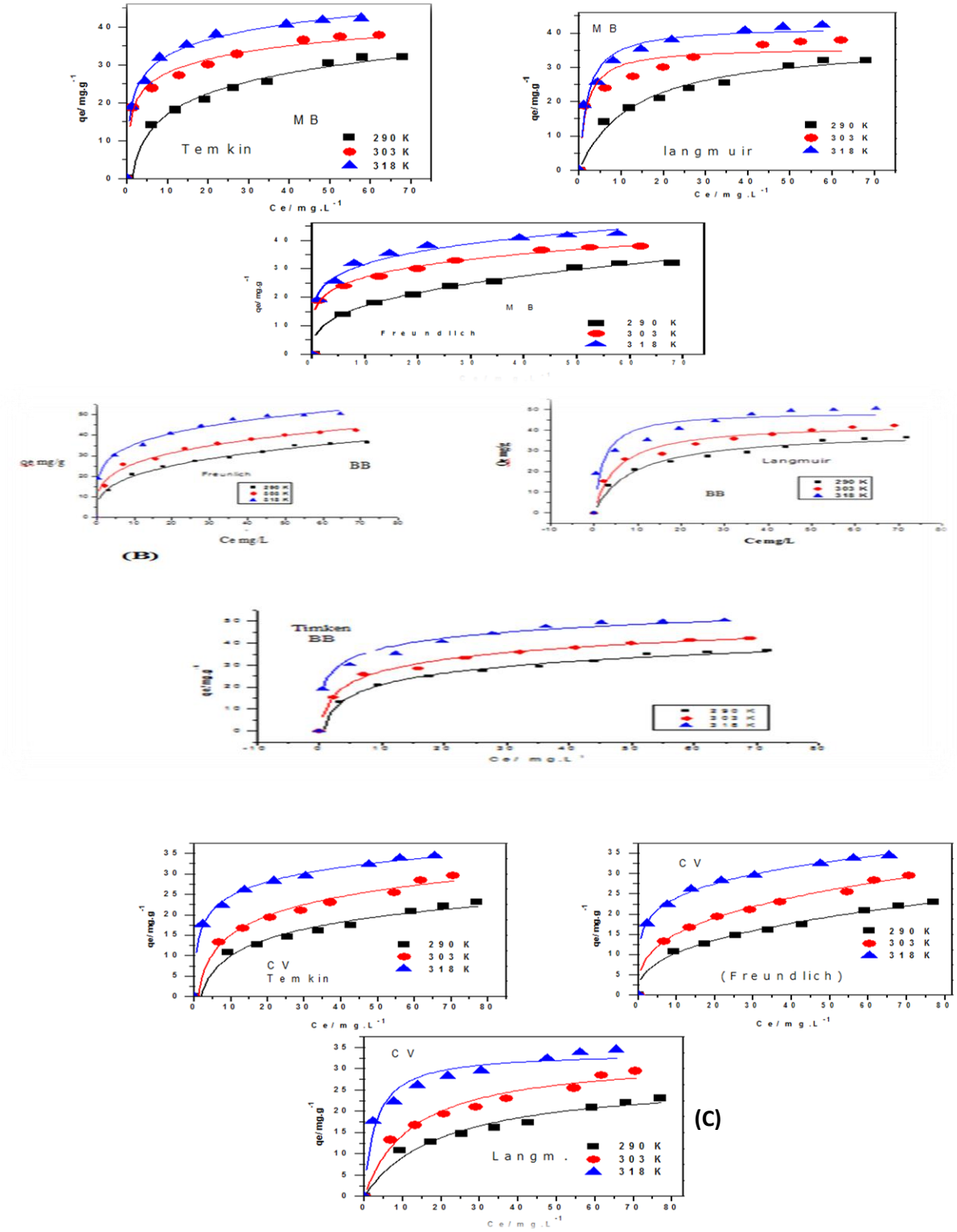


**Fig. 5.** Isotherm model plot three dyes (A) MB (B) BB (C) CV adsorption (adsorbent dosage = 0.1 g, T = 290 K, Vibration average 120 rpm, and connect time 24 h).

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The solubility of the adsorbate molecules is also compromised in certain situations, which eventually has a major impact on the removal process. Increased potential of adsorption of adsorbents (Langmuir 1918; Aljeboree & Alshirifi 2012) have been measured using the following equations, and thermo-dynamic variables like changes in the free energy (ΔG), changes in enthalpy (ΔH) as well as changes in entropy (ΔS) (Aljeboree & Alshirifi 2012)

$K_d = qe / C_e$ **(5)**

Where $K_d$ is constant equilibrium, Ce and qe, concentrations of the equilibrium dye ion, are solution (mg $L^{-1}$) and absorbent (mg $g^{-1}$) concentrations, respectively. However, changes in the free energy may be calculated by the following equation:

$\Delta G = RT \ln Ke$ **(6)**

But from the following equation, the change in enthalpy (ΔH) is calculated:

$\ln Ke = \Delta H / RT + \Delta S / R$ **(7)**

**Thermodynamic parameters and temperature effects**

At different temperatures, removal of three dyes has been studied. 290 K, 303 K and 318 K to establish isothermal and thermodynamic adsorption parameters. The experiment has investigated that the amount of dye uptake increases with temperatures rising from 290 K to 318 K, diffusion rate of dye molecules is a process regulated by temperature. Temperature variation alters the adsorbent's equilibrium potential for a specific adsorbate. The increase in temperature in the current study results in a rapid diffusion of the dye molecules through external boundary layer of the adsorbent particles and internal pores due to less resistance in the aqueous phase of viscous forces. The solubility of the adsorbate molecules is also compromised in certain situations, which eventually has a major impact on the removal process. Increased potential of adsorption of adsorbents (Langmuir, 1918; Aljeboree & Alshirifi 2012) have been measured using the following equations, and thermo-dynamic variables like changes in the free energy (ΔG), changes in enthalpy (ΔH) as well as changes in entropy (ΔS) (Aljeboree & Alshirifi 2012)

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**Table 3.** ΔG, ΔH and ΔS, for MB, CV, BB absorbed on the coconut.

| T/K | Ka | $\Delta G^o$/KJ.mol$^{-1}$ | $\Delta H^o$/KJ.mol$^{-1}$ | $\Delta S^o$/JK$^{-1}$.mol$^{-1}$ |
|---|---|---|---|---|
| **Coconut adsorbent /CV adsorbate** | | | | |
| 290 | 1154.89 | -17.002 | 7.344 | 84.059 |
| 303 | 1366.56 | -18.188 | | |
| 318 | 1511.12 | -19.354 | | |
| Coconut adsorbent /BM adsorbate | | | | |
| 290 | 568.86 | -15.294 | 9.597 | 85.775 |
| 303 | 660.58 | -16.357 | | |
| 318 | 807.36 | -17.699 | | |
| Coconut adsorbent /BB adsorbate | | | | |
| 290 | 2006.16 | -18.333 | 11.434 | 102.497 |
| 303 | 2331.18 | -19.533 | | |
| 318 | 3043.14 | -21.205 | | |

Therefore, there should be a straight line plot of ln Ke versus 1/T (Fig. 8). S- and H-values have been determined respectively through intercept and slope of this map. The equations. are used to obtain the standard changes in the

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free energy (ΔG), standard changes in entropy (ΔS) as well as standard changes in enthalpy (ΔH). Table 3 lists 6 and 7 and their values correlated with the adsorption of MB, CV, BB to coconut. Moreover, negative values of G suggest viability of this procedure and random nature of the high-performance adsorption. MB, CV, BB for coconut. Additionally, ΔH positive value refers to the method's endothermic component, while S positive value implies the adsorbents' affinity, including MB, CV, BB and BB. (Al-Hayder & Al-Hussainawy 2016; Sajjadi & Moosavi 2018; Aseel *et al*. 2019; Hamilton-Ekeke 2019; Askari *et al.* 2020; Delavari *et al*. 2021; Baghernejad & Fiuzat 2021).

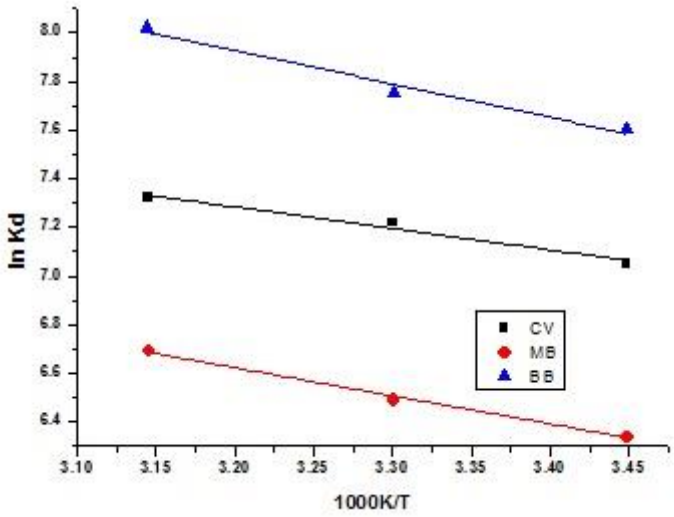


**Fig. 6.** Van't Hoff's plot for the coconut absorption of MB, CV, BB.

## CONCLUSION

It can be inferred that a coconut shell (AC) can be used for hazardous textile dyes (MB, CV and BB) with regard to the experimental findings of the current research. Absorption isotherms of these dyes on coconut have also been studied and modelled using Langmuir > Freundlich > Tempkin isothermal models. The Freundlich isothermal model will accurately explain the adsorption of three dyes. As confirmed by thermodynamic experiments, the adsorption process of dyes on AC is endothermic. For a device suggesting a random operation, the ΔG value is negative. The percentage of removal of the dyes depends on the mass of AC and solution temperature.

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