FUDMA Journal of Sciences (FJS)
ISSN online: 2616-1370
ISSN print: 2645 - 2944
Vol. 3 No. 2, June, 2019, pp 67 - 76

EQUILIBRIUM ADSORPTION OF MALACHITE GREEN DYE ONTO DEFATTED PAPAYA SEED.

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ABSTRACT

Adsorption of malachite green (MG) dye onto defatted papaya seeds (DPS) was studied. Effects of initial dye concentration, contact time, pH and the solution temperature were investigated. It was found that adsorption of malachite green onto the defatted *Carica papaya* seeds was favorable in the acidic medium while the adsorption uptake increased with increasing initial dye concentration, solution temperature and contact time. Isotherm models such as Langmuir, Freundlich, and Temkin were used to analyze the data and Langmuir isotherm was found to be the best fit for the adsorption data with a monolayer adsorption capacity of 126.05 mg/g. The adsorption study showed that defatted papaya seed could be a viable alternative adsorbent in the removal of potentially toxic dyes from aqueous solution.

Keywords: Defatted papaya seeds, Low- cost adsorbent, Adsorption, Isotherm models, Malachite green

INTRODUCTION

Papaya (*Carica papaya*) is a herbaceous plant commonly found in tropical regions. It bears fruit which may be yellowish green, yellow or orange in color when ripe. The seeds are rich in oil and protein. The seeds and fruits of papaya are proven to enhance digestion, making it easier to digest proteins. It is also used to break up blood clots after surgery; this is due to the presence of enzyme papain in the plant's latex (Oduola *et al.*, 2007). The seeds were reported to contain 24.3 g protein, 25.3 g fatty oil, 32.5 g total carbohydrate, 17.0 g crude fiber, 8.8 g ash, and 0.09 % volatile oil. With reversed phase partition column chromatography, the oil from the seeds of C. papaya was found to contain the following acids: lauric, 0.4 %; myristic, 0.4 %; palmitic, 16.2 %; stearic, 5.0 %; arachidic, 0.9 %; behenic, 1.6 %; hexadecenoic, 0.8 %; oleic, 74.3 %; and linoleic, 0.4 % (Badami and Daulatabad, 1996; Morales-Payan and Stall, 2003). With the papaya seed oil being established as a potential source of high oleic oil, its full potential needs to be exploited (Puangsri *et al.,* 2005). Usefulness of the lipids mentioned above makes it very crucial that the seeds that are discarded as wastes be defatted first before use as biosorbent.

Malachite green (MG) is a popular dye that is extensively used. Its application extends to medical sciences but is highly toxic to flora and fauna. It induces the risk of cancer, acts as a liver tumor-enhancing agent and many other diseases (Garba *et al.*, 2014; Gong *et al.*, 2006). In addition to this, expelling MG into the hydrosphere can cause environmental degradation as it gives undesirable color to water as well as reducing sunlight penetration *(Ahmad and Kumar, 2010)* Therefore, it is imperative to remove MG from aqueous effluents before they are discharged into bodies of water.

Several processes like chemical, biological and physical processes have been applied for the treatment of dyes from wastewater (Alslaibi *et al.,* 2013). In recent years, the physical method through adsorption process based on activated carbon material has been considered to be superior techniques as compared to others (Adetokun *et al.*, 2019; Afidah and Garba, 2015; Afidah and Garba, 2016; Ait Ahsaine *et al.*, 2018; Alizadeh *et al.*, 2017; Darweesh and Ahmed, 2017; Garba and Afidah, 2014; Garba and Afidah, 2016; Garba *et al.*, 2019a; Garba *et al.*, 2019b; Prashanthakumar *et al.*, 2018). The first thing for an efficient adsorption process is the search for a low-cost adsorbent with high adsorption capacity. Economically, discovering alternative adsorbent to replace the expensive activated carbon is necessary. The use of cheap and abundant agricultural waste such as defatted *Carica papaya* seed as low-cost adsorbent will decrease the waste disposal and also convert this waste into a value-added product.

The aim of this paper, therefore, is to report the use of defatted papaya seeds (DPS) for the adsorption of malachite green (MG) dye from aqueous solution. Some variables that affect the adsorption capacity of the defatted papaya seed adsorbent such as particle size, pH, initial dye concentration, adsorbent dosage, temperature and time of adsorption were studied.

MATERIALS AND METHODS

Material

The papaya seeds were collected locally in Nigeria and left under the sun for about 10 days to dry. Malachite green, 4-[(4dimethylaminophenyl)-phenyl-methyl]-N,N-dimethyl-aniline supplied by Sigma-Aldrich (M) was used as an adsorbate. Deionized water was used to prepare all solutions. MG has a chemical formula of $C_{23}H_{25}N_2Cl$ with a molecular weight of 364.911 g/mol.

Preparation of the defatted papaya seeds (DPS)

The seeds were collected and then defatted by the Soxhlet extraction method using hexane. In order to remove residual hexane from the sample, it was allowed to dry in a fume cupboard. The sample collected thereafter was referred to as defatted papaya seeds (DPS).

Batch adsorption studies

Bath adsorption was carried out for the removal efficiency of malachite green dye (MG) in aqueous solution with different initial concentrations (30 to 180 ppm). A known amount of the DPS was added into 250 mL reagent bottles containing 100 mL of different concentrations of MG. Each sample was kept in an isothermal shaker at a fixed speed and temperature to reach equilibrium states. To analyze the solutions, all samples were filtered to separate DPS from the MG adsorbate. Ultraviolet-Visible (UV-vis) spectrophotometer ((JASCO V-530) was used for the determination of the remaining concentrations of MG at a wavelength of 617nm. The amount of MG adsorbed at equilibrium, q_e (mg/g) was calculated as:

$$q_e = \frac{(C_o - C_e)V}{W} \qquad 1$$

where C_o and C_e (mg/L) are the initial and equilibrium concentrations of the dyes respectively, V (L) is the volume of the solution, and W (g) is the weight of the dry adsorbent used. The percentage removal of the MG was calculated as:

$$\%\,Removal = \frac{C_o - C_e}{C_o} \times 100 \qquad 2$$

Effect of initial concentration and contact time

In order to study the effect of contact time and initial concentration on the MG uptake, 100ml of MG solutions with an initial concentration of 30 - 180 mg/l were prepared in a series of 250 ml Erlenmeyer flasks at different time intervals (0-24 hrs). A known amount of the DPS was added into each flask covered with a glass stopper and the flasks were then placed in an isothermal water bath shaker at a constant temperature of 303 K, with a rotation speed of 120 rpm.

Effect of solution pH

The effect of solution pH on the MG removal was studied by varying the pH from 2 to 12. The pH was adjusted by adding either HCl or NaOH. The pH measurement was conducted using a pH meter (Martini instrument, Mi 150). A known amount of the adsorbent was added to each 100 mL of MG solution, the agitation rate and temperature were set. The MG percent removal was calculated by using equation (2).

Effect of adsorbent dosage

The effect of adsorbent doses on the removal of the dye was studied by varying the adsorbent dosages from 25 to 200 mg. Different masses of the adsorbent were added to each 100 mL of MG solution; the agitation rate and temperature were set. The MG percent removal was calculated by using equation (2).

Effect of temperature

In order to study the effect of temperature on the adsorption process, the experiment was conducted at 303, 313 and 323 K by adjusting the temperature controller of the water bath shaker, while other operating parameters such as the volume of MG solution, pH, DPS dosage and rotation speed were kept constant. The residual MG concentrations were analyzed at 617nm.

RESULTS AND DISCUSSION

Effect of initial concentration and contact time

The effect of contact time on the adsorption capacity of MG using DPS at different initial concentrations is shown in Fig. 1. It can be seen that the adsorption capacity of MG increased with contact time rapidly and thereafter it proceeded at a lower rate and finally reached equilibrium. The result suggests that adsorption takes place rapidly at the initial stage on the external surface of the adsorbent followed by a slower internal diffusion process, which may be the rate-determining step (Gialamouidis *et al.,* 2010). In addition, the fast adsorption at the initial stage also may be due to the fact that a large number of surface sites are available for adsorption but after r a lapse of time, the remaining surface sites are difficult to be occupied. This is because of the repulsion between the solute molecules of the solid and bulk phases, thus, make it take a long time to reach equilibrium.

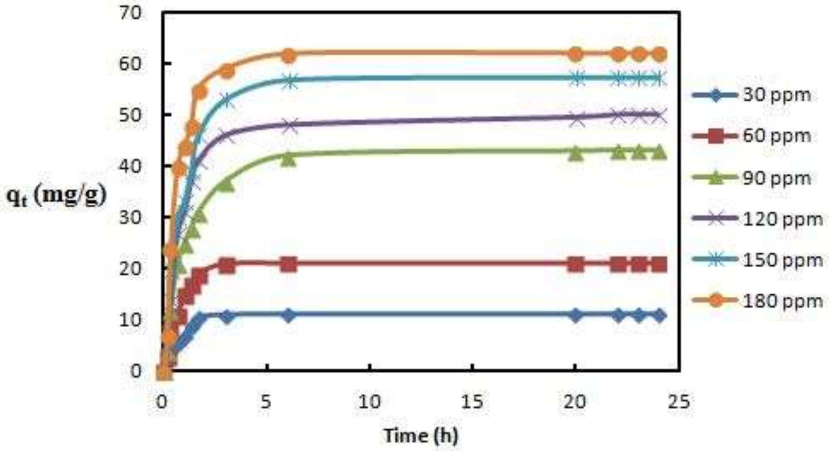

Fig. 1. Effect of contact time on the adsorption of MG onto DPS at different concentrations

It was also observed that an equilibrium time of about 3 hours was needed for the MG dye solution with initial concentrations of 30 and 60 ppm to reach equilibrium but longer equilibrium times of 20 – 24 hours were needed for initial concentrations of 90 - 180 ppm. It was clear that the adsorption capacity of the MG dye depended on its initial concentration. Adsorbate molecules have to initially encounter the boundary layer effect, it will then diffuse from boundary layer film onto the adsorbent surface and finally into the porous structure of the adsorbent (Idris *et al.*, 2011). In fact, the ratio of the initial number of dye molecules to the available surface area is low at lower initial concentration compared to higher initial concentration. Therefore, MG solutions with higher initial concentrations would take relatively longer contact time to attain equilibrium due to the higher amount of MG molecules. The equilibrium time for the adsorption of Reactive Orange 12, Reactive Red 2 and Reactive Blue 4 on coir pith - activated carbon was reported to be 4 h for an initial concentration of 40 ppm (Santhy and Selvapathy, 2006). (Leechart *et al.*, 2009) reported that for Red Reactive 141 with an initial concentration of 400ppm, the adsorption amount was gently increased in the first 6 h and gradually reached equilibrium within 24 h. This showed that the adsorption performance of the DPS in this study compared well with the findings of previous researchers where the adsorption capacity increased with increasing the initial dye concentration ending to the saturation at higher dye concentrations.

Effect of solution pH

The pH of the system exerts an overwhelming influence on the adsorption capacity of the adsorbate molecule presumably due to its influence on the surface properties of the adsorbent and ionization or dissociation of the adsorbate molecule. Fig. 2 shows the variations in the MG percent removal at pH values of 2-12. It can be seen that the percentage removal of MG increased from pH 2 to 6 before it decreased gradually until pH 12. The maximum percentage removal of MG (89 %) was observed at pH 6. A similar trend of pH effect was observed for the adsorption of MG onto the rubber seed coat based activated carbon (Idris *et al.*, 2011).

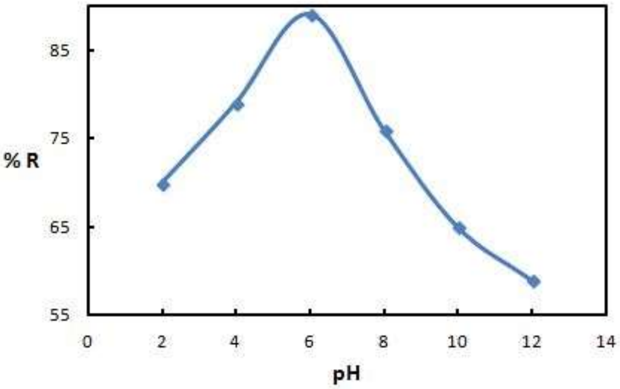

Fig. 2. Effect of solution pH on the removal of MG onto DPS

Effect of adsorbent dosage

Adsorption dosage is an imperative parameter in adsorption studies because it ascertains the capacity of the adsorbent for a given initial concentration of MG onto the DPS adsorbent as shown in Fig. 3.

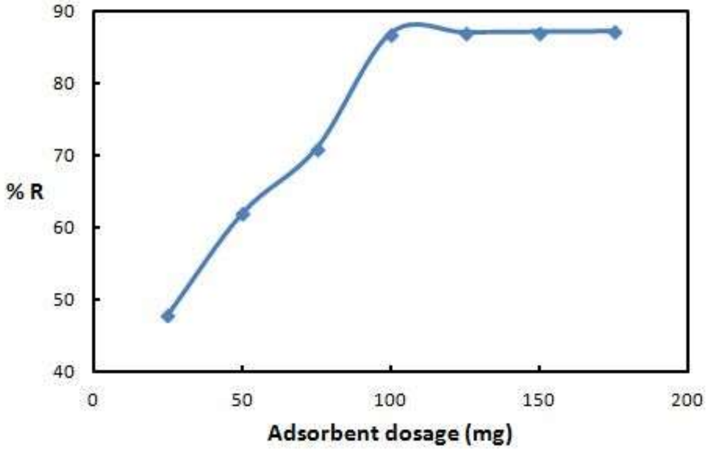

Fig. 3. Effect of adsorbent dosage on the removal of MG onto DPS

The percentage of MG removed from the solution increased with increase in DPS adsorbent dosage from 25 mg to 175 mg as shown in Fig. 3. This was attributed to the increase in the availability of the carbon surface active sites resulting from the increased dosage and accumulation of the adsorbent. The percentage removal increased from 48 to 87.15% with increasing adsorbent dosage from 25 to 175 mg for MG. When saturation was reached at the outer surface with the addition of 100 mg of the adsorbent, the MG molecules entered the pores of the adsorbent and were adsorbed by the inner surface of the particles.

Effect of temperature

The equilibrium adsorption capacity of MG increased with increase in the solution temperature from 303 to 323 K for all initial MG dye concentrations (30 – 180 ppm) as shown in Fig. 4. This may be a result of an increase in the mobility of the large dye ion with temperature.

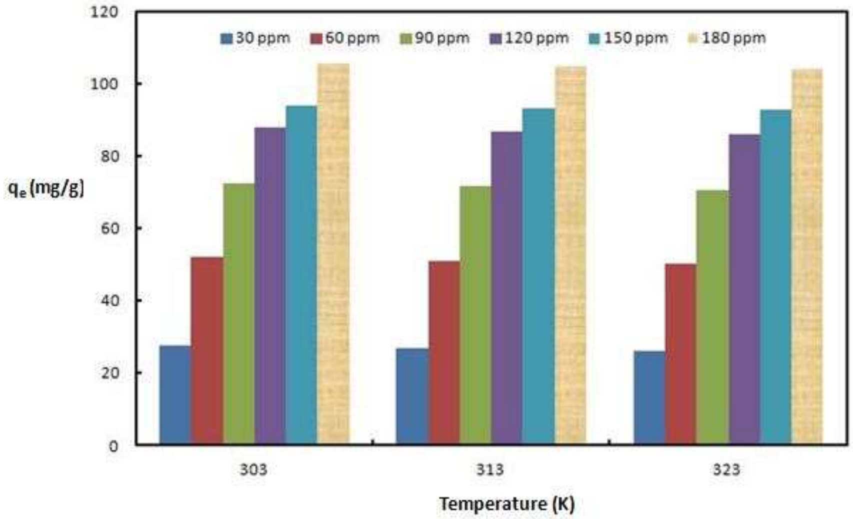

Fig. 4. Effect of solution temperature on the adsorption capacity at different initial concentrations

An increasing number of molecules may also acquire sufficient energy to undergo an interaction with active sites at the surface; furthermore, the increasing temperature may produce a swelling effect within the internal structure of the activated carbon, enabling large dyes to penetrate further. A similar trend for the adsorption of Remazol Brilliant Blue, Remazol Red 133 and Rifacion Yellow Red by fly ash as well as for the removal of Malachite Green on rubber seed coat based activated carbon was reported by other researchers (Dizge *et al.*, 2008; Idris *et al.*, 2011).

Adsorption Isotherms

The Langmuir, Freundlich, and Temkin isotherm models were applied to depict the relationship between the amount of MG adsorbed and its equilibrium concentration. Langmuir isotherm is one of the most popular isotherms reported by scientific workers for the removal of heavy metals, dyes and other organic pollutants by adsorption onto activated carbon. It assumes monolayer adsorption onto a surface containing a finite number of adsorption sites of uniform strategies (Langmuir, 1916). The linear form of the Langmuir isotherm equation is given as:

$$\frac{C_e}{q_e} = \frac{1}{K_L \cdot Q_a^0} + \frac{C_e}{Q_a^0} \qquad (5)$$

where C_e (mg/L) is the equilibrium concentration of the CPs, q_e (mg/g) is the amount of CPs adsorbed per unit mass of adsorbent. Q_a^0 (mg/g) and K_L (L/mg) are Langmuir constants related to adsorption capacity and rate of adsorption, respectively.

The essential characteristics of the Langmuir equation can be expressed in terms of dimensionless separation factor, R_L, defined as:

$$R_L = \frac{1}{1 + K_L C_o} \qquad (6)$$

where C_o is the highest initial solute concentration. The R_L value indicates the type of isotherm as in Table 1

Table 1: R_L values indicating types of isotherm

R_L Value	Type of Isotherm
$R_L > 1$	Unfavorable
$R_L = 1$	Linear
$0 < R_L < 1$	Favorable
$R_L = 0$	Irreversible

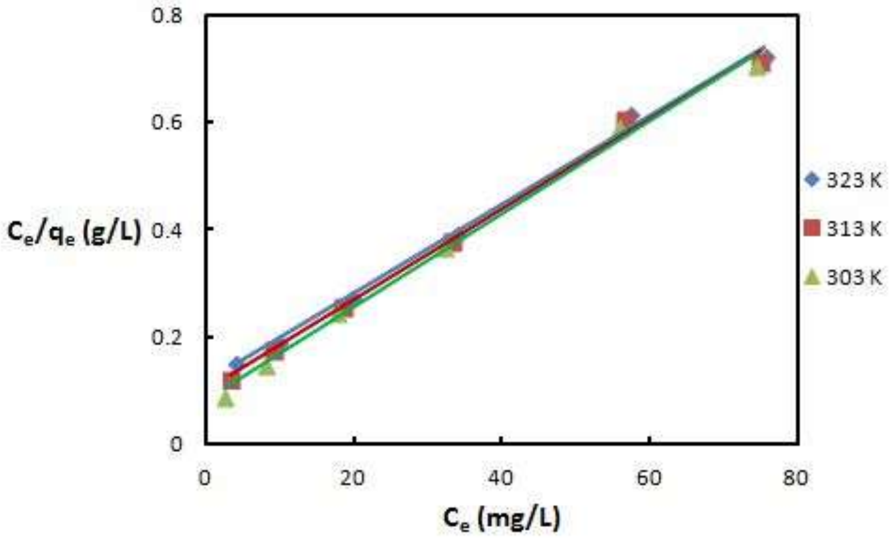

Fig. 5. Langmuir isotherm plot for the adsorption of MG dye onto DPS

Table 2 summarizes all the constants and R^2 values obtained from the three isotherm models applied for the MG adsorption on DPS. The Q_a^0 values obtained from the linear Langmuir plot (Fig. 5) increased with an increase in temperature, suggesting that the adsorption is endothermic. Raise in temperature increases mobility of the solute molecules which lead to increasing the number of molecules that will have enough energy to bind to the active sites of the DPS. According to the results shown in Table 2, the correlation coefficients of Langmuir isotherm model appeared to be higher than those of Temkin and Freundlich isotherms. The fitness of the Langmuir model to the adsorption process connotes that the MG from bulk solution were adsorbed on specific monolayer which is homogeneous in nature.

Table 2. Isotherm parameters for MG adsorption onto DPS at various temperatures

	Temperatures		
	303 K	313 K	323 K
Langmuir isotherm			
Q_a^0 (mg/g)	126.05	129.92	137.81
K (L/mg)	0.109	0.104	0.091
R^2	0.997	0.995	0.998
Freundlich isotherm			
K_F (mg/g) $(L/mg)^{1/n}$	21.88	18.595	16.151
n	2.06	2.84	2.19
R^2	0.971	0.962	0.973
Temkin isotherm			
A (L/g)	1.401	0.971	0.870
B (J/mol)	20.204	26.294	30.931
R^2	0.986	0.990	0.992

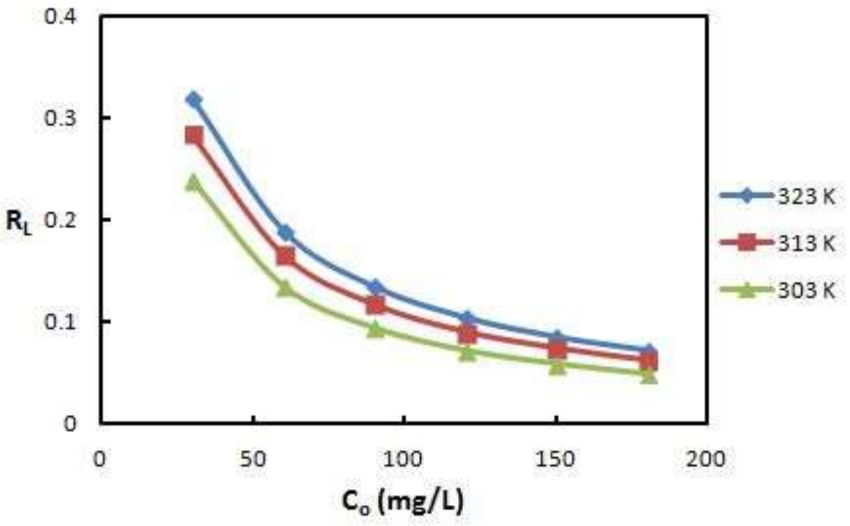

Fig. 6. Effect of initial MG concentration on dimensionless separation factor R_L

The lower the R_L value, the more irreversible the adsorption process. It can be seen from Fig. 6 that as the MG initial concentration increases from 30 to 180 ppm, the R_L values decreased and were all greater than 0 but less than 1 ($0 < R_L < 1$) indicating that the adsorption was more favorable at higher MG concentration.

Freundlich isotherm, on the other hand, assumes heterogeneous surface energies, in which the energy term in Langmuir equation varies as a function of the surface coverage. The logarithmic form of the Freundlich isotherm is given by the following equation (Freundlich, 1906):

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

where K_F and n are Freundlich constants with n being a measure of the deviation of the model from linearity of the adsorption and K_F indicates the adsorption capacity of the adsorbent. In general, n > 1 suggests that adsorbate is favorably adsorbed on the adsorbent. The higher the n value, the stronger the adsorption intensity.

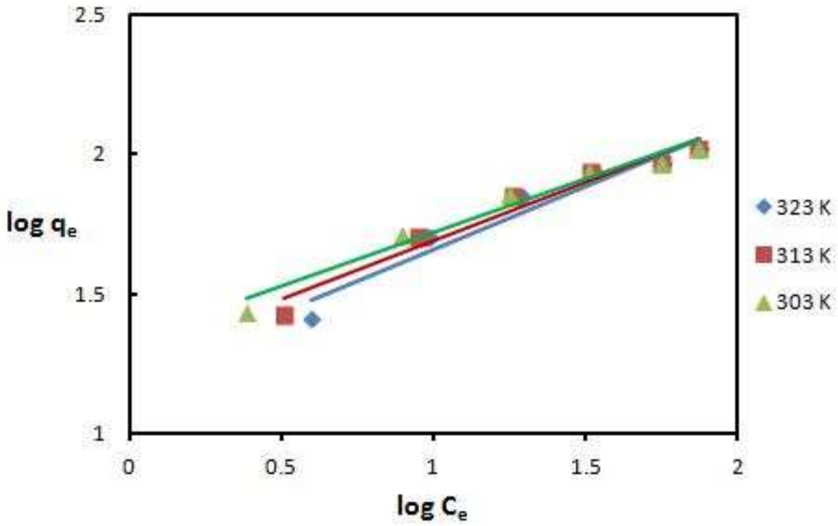

Fig. 7. Freundlich isotherm plot for the adsorption of MG dye onto DPS

The Freundlich parameters were calculated from Fig. 7 as shown in Table 2. The n values indicate physical process and favorable adsorption conditions for MG in aqueous solution. The coefficient of correlation for the adsorption of MG was less than that of both Langmuir and Temkin isotherms and was, therefore, the less favorable.

Temkin and Pyzhev considered the effects of indirect adsorbent/adsorbate interactions on adsorption isotherms. The heat of adsorption of all the molecules in the layer would decrease linearly with coverage due to adsorbate/adsorbate interactions. The linear form of Temkin isotherm is as follows (Temkin and Pyzhev, 1940):

$$q_e = \frac{RT}{b}\text{In A} + \frac{RT}{b}\text{InC}_e \qquad (8)$$

where $\frac{R_T}{b}$ = B (J/mol) and A (L/g) are Temkin constants, which are related to the heat of sorption and maximum binding energy, respectively. R is the gas constant (8.31J/mol K) and T (K) is the absolute temperature.

The constants A and B were determined by plotting q_e vs. ln Ce, as shown in Fig. 8.

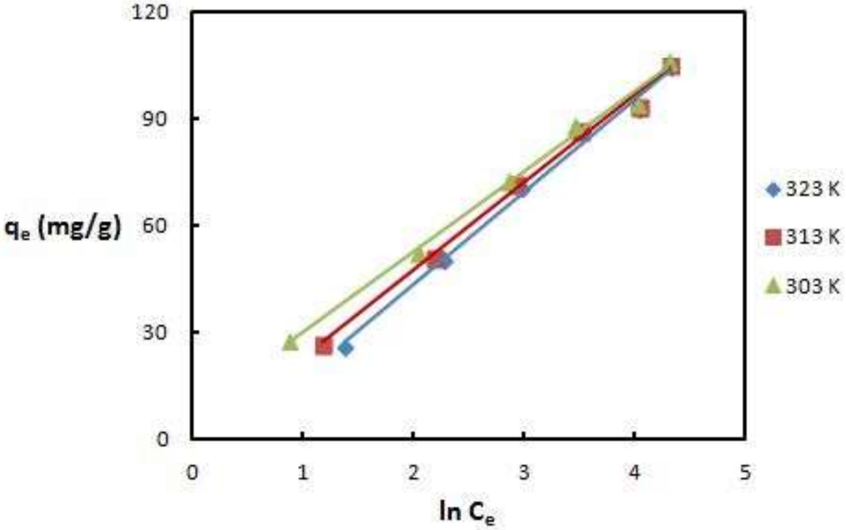

Fig. 8. Temkin isotherm plot for the adsorption of MG dye onto DPS

The Temkin coefficient correlation (R^2) values for the adsorption MG were slightly lower than those of Langmuir isotherm at all temperatures indicating that Langmuir isotherm gave a better fit.

The conformation of the experimental data into the Langmuir isotherm equation proved that the surface of DPS for adsorption of MG is made up of homogeneous adsorption patches than heterogeneous adsorption patches (Tunc *et al.*, 2009). The results agreed with the works carried out by previous researchers for other reactive dyes such as reactive blue dyes, which reported that the Langmuir model gave a better fit than both Freundlich and Temkin models on the adsorption using different adsorbents such as commercial activated carbon from bentonite (Ozcan *et al.,* 2007) and sepiolite (Demirbas and Nas, 2009). This suggested that some homogeneity on the surfaces or pores of the activated adsorbents played the role in MG adsorption.

Table 3 lists a comparison of the adsorption capacity of the DPS with those obtained in the literature for the adsorption of MG at 303K. It can be seen that the DPS were very effective for this purpose even when compared with activated carbons. The adsorption capacity of DPS for MG was 126.05 mg/g, higher than the adsorption capacities of various low-cost adsorbents and activated carbons for MG adsorption reported from the literature.

Table 3. Comparison of adsorption capacities, Q_a^0 of MG onto different adsorbents at 303 K

Adsorbents	Q_a^0 (mg/g)	References
Defatted papaya seed	126.05	This work
Coconut coir activated carbon	27.44	(Uma *et al.,* 2013)
Borassus aethiopum activated carbon	219.67	(Garba *et al.,* 2014)
Activated carbon from waste apricot	116.27	(Basar, 2006)
Ashoka leaf powder	83.3	(Gupta *et al.,* 2012)
Rubber seed coat based activated carbon	72.73	(Idris *et al.,* 2011)
Lab grade activated carbon	42.18	(Mall *et al.,* 2005)
Sea shell powder	42.3	(Pan *et al.,* 2012)
Lemon peel	51.73	(Kumar, 2007)
Hen feathers	26.1	(Mittal, 2006)

CONCLUSION

The adsorption of malachite green (MG) dye from aqueous solution was investigated using defatted papaya seeds (DPS) as low-cost adsorbent under different experimental conditions. Langmuir adsorption isotherm was the best fit to the experimental data with a maximum adsorption capacity of 126.05 mg/g. Adsorption of MG onto DPS was found to increase with an increase in contact time, MG initial concentration and solution temperature and was favored at pH 6 which is *slightly* acidic medium. The outcome of the investigation signaled that DPS, a low-cost adsorbent could be employed as an alternative to commercial activated carbon for the removal of MG from aqueous solutions but further studies on characterization, kinetic and thermodynamics are necessary to fully understand the adsorption processes.

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