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ORIGINAL ARTICLE

The Adsorption of Malachite Green from Industrial Wastewater by Oxidized Black Tea Adsorbent

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KEYWORDS

Adsorption;
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ABSTRACT: An Eco-friendly and low-cost adsorbent surface was prepared from oxidized black tea leaves for adsorption of Malachite Green (MG) dye by batch equilibrium adsorption technique and investigated the optimum conditions that include dose of adsorbent, contact time, adsorbent particle size, pH, and initial concentration of the dye. All adsorption measurements were performed using spectrophotometry at 618 nm, the maximum wavelength of MG. Maximal adsorption was obtained at 1.0 g of oxidized black tea leaves with a contact time of 120 min. The optimal pH was 8. The maximum adsorption capacity was 97.8 mg g^{-1}. The physic processes were used to prepare the adsorbent surface from oxidized black tea leaves. These included boiling the oxidized black tea leaves for 10 hours using distilled water, filtering, washing, and drying at 80°C. Physic preparation gave a suitable surface for the adsorption process. The adsorption of MG on the surface of tea leaves obeyed the Freundlich and Langmuir equations in adsorption. Isotherm parameters of Freundlich and Langmuir modules were estimated.

INTRODUCTION

Dyes are vastly used in printing, textiles, cosmetics, paper, rubber, plastics, and the industry of leather for coloring their products leading to colored wastewater [1]. MG, one of these dyes, has a passive influence on organisms found in water, the chemical composition, and the visual characteristics of water [2]. MG is carcinogenic, highly toxic, and causes mutagenesis and its effects increase with concentration, temperature, and exposure time [3]. Accordingly, it is necessary to demo the different methods ready for mitigation and separation before discharging them into the water.

Several methods have been used to remove the green megalith dye. Photo-degradation by Graphene or its derivatives used to remove MG at a removal of 95 % and adsorption capacity of 17 mg l^{-1} [4]. Electrochemical oxidation polymerization can be used for MG removal from an aqueous solution [5]. The photocatalytic method is used to remove MG Dye by using ZnO or ZnO nano-powder with UV irradiation [6, 7]. Furthermore, MG was removed from wastewater by photocatalytic using nano-composites containing TiO_2 at a removal of 91% and pH 7 [8]. Nevertheless, these methods are expensive, require a catalyst, have high electricity costs, and generate polluted sludge with metals [9, 10]. The electro-coagulation technique is used for MG removal from contaminated water with 100% by using current density (76.5 A m^{2-1}) at pH 8 [11, 12]. The ionic flocculation method is used for the removal of MG by adding surfactant and calcium, and then the formed precipitate of adsorbed MG is separated by centrifugation[13].

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However, these techniques are not environmentally sustainable, generate colored coagulated waste, and produce toxic sludge [14]. The liquid membrane method was used for selective MG removal from an aqueous solution by polyvinylidene fluoride films which are used for liquid membrane as supports at high flux value and pH11 [15]. However, this technique has recurrent membrane clogging, operation costs, and high pressure [16, 17].

Among all the mentioned removal methods, adsorption still to be the most agreeable process for the treatment of many pollutants including MG from wastewater [18]. Adsorption supremacy over other methods is related to its comfortable operation, simplicity of design, low cost, and great susceptibility to removing an enormous number of pollutants [19]. Many adsorbents were used for the adsorption of MG. MG dye was adsorbed from an aqueous medium by A green approach using a chitosan-cellulose mixture with an adsorption capacity of 115.1 mg g^{-1} and a contact time of 30 min [20]. Corn leaves were used as cheap adsorbent material for decolorizing MG by adsorption process with a removal of 91% and a contact time of 4 days [21]. Shells of *Helianthus Annuus* seeds were used to remove MG dye as eco-friendly material by adsorption with an adsorption capacity of 7.69 mg g^{-1}, pH 7, and contact time of 60 min [22]. The Banana stem was used to remove MG dye as a cheap adsorbent by adsorption method with an adsorption capacity of 8.29 mg g^{-1}, pH 8, and contact time of 45 min [23]. Mesoporous inorganic clays were utilized to remove MG dye as an available adsorbent by adsorption technique with an adsorption capacity of 172.4 mg g^{-1}, pH 8, and contact time of 120 min [24]. MG dye was adsorbed by activated carbon prepared from *Catha Edulis* stem as a cheap bio‑adsorbent with an adsorption capacity of 4.55 mg g^{-1}, pH 10, and contact time of 60 min [25]. However, oxidized black tea leaf waste is one of the cheapest adsorbents that can adsorb many dyes.

This paper studies the adsorption of MG dye on oxidized black tea leaves as a low-cost commercial adsorbent by batch equilibrium adsorption technique. The adsorbent is prepared by the physics method which represented an unconsumed way. Two adsorption isotherm modules, Freundlich and Langmuir, were employed in this investigation, and optimal conditions were determined to reach maximal dye removal ability and adsorption capacity.

MATERIALS AND METHODS

Instruments

Analytical balance (Denver Instrument) was utilized to weigh the adsorbent surface and during the preparation of the MG dye solution. An Electric oven (MTI Corporation) was used to dry the physic processes to prepare the adsorbent surface. A shaker (Barnstead International, MaxQ 2000 Orbital Shaker Model SHKA2000) was used to shake different samples during the adsorption of MG with oxidized black tea leaves at 150 rpm agitating speed. pH meter (WTW3) was used to measure the pH values of various solutions. UV - Vis spectrophotometer (PG Instruments Ltd, APEL) was used to measure the MG dye spectrophotometry at 618 nm. Laboratory porcelain mortar with a pestle was used to crush and cut the adsorbent into different granular sizes. Laboratory gradation testing sieves were utilized to separate adsorbents into different granular sizes.

Materials

Black tea leaves were purchased from local markets and used after boiling with water. MG dye, hydrochloric acid, and sodium hydroxide were obtained from Sigma-Aldrich Company in a purified state. Hydrochloric acid and sodium hydroxide were used at 0.1 M to adjust the pH. All experiments were run out using deionized water. The physicochemical properties of MG dye shown in Table 1.

Table 1. Physiochemical properties of MG dye.

Parameter	Value
Molecular Formula	$C_{23}H_{25}ClN_2$
Molecular Weight	364.91g mole^{-1}
Maxima Wavelength	618 nm
Other Names	Basic green 4
Solubility	Water + Alcohol

Preparation of adsorbent surface

The oxidized black tea adsorbent surface was prepared by taking a large number of tea leaves and washing them several times with distilled water. The black tea leaves carbonized at 650°C and then oxidized by boiling nitric acid as previously described. After filtration,

Preparation of adsorbed dye

The MG dye, N, N, N′, N′-Tetramethyl-4,4′-diaminotriphenylcarbenium chloride, which was used in this research has the chemical structural formula as in Figure 1(18). 1000 mg l^{-1} dye was prepared by weighing 1.00 g of the dye and diluting to 1000 mL of deionized distilled water to be used as a stock solution for the next experiments. The working solutions were prepared by dilution in distilled water.

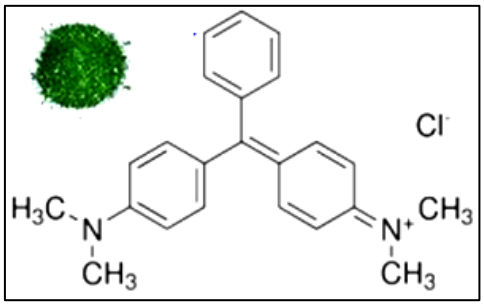

Figure 1. Synthetic structural formula of MG dye

Studies of batch adsorption [17]

The percentage removal efficiency of oxidized black tea leaves and adsorption capacity ($q_{e)}$ were calculated by using the equations 1 and 2, respectively. Where; C_o= Initial concentration of dye (mg l^{-1}), C_e = Concentration of the dye at equilibrium, and q e: is the adsorption capacity of MG (mg g^{-1}). V: the volume (L) of the solution. W: the oxidized black tea leaves dose (g).

$$Removal\ efficiency\ (\%) = \frac{C_o - C_e}{C_o} * 100 \qquad (1)$$

$$q_e = \frac{V}{W}(C_o - C_e) \qquad (2)$$

Equation of Langmuir isotherm [3]

The Langmuir isotherm is given by the equation (3)

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

where q_e is the amount of material absorbed per unit mass of adsorbent at equilibrium (mg l^{-1}), q_m is the adsorption capability (mg g^{-1}), C_e is the solution concentration at equilibrium (mg l^{-1}) and Ka is the adsorption constant. The Plotting of C_e/q_e against C_e in the last equation is linear which means the adsorption of material by adsorbent takes the Langmuir line. Langmuir model vital features can be given by a constant of dimensional called equilibrium coefficient (RL), which is known by equation 4

$$R_L = \frac{1}{1 + \mathrm{b}\, C_o} \qquad (4)$$

where b is the Langmuir constant and C_o is the initial concentration. The value of R_L referring to the link may be unfavorable ($R_L > 1$), permanent ($R_L = 0$), favorable ($0 < R_L < 1$), or linear ($R_L = 1$).

Equation of Freundlich Isotherm [3]

The Freundlich isotherm is given by the equation (5).

$$logq_e = \left(\frac{1}{n}\right) logC_e + logK_f \qquad (5)$$

Where qe is the amount of material absorbed per unit mass of adsorbent at equilibrium (mg g^{-1}), Ce is the solution concentration at equilibrium (mg l^{-1}). Kf and n refer to the Freundlich constants, n shows a gesture to the favorability, and Kf (mg g-1) (L mg-1) 1/n, Kf, and n values are acquired from the graph of log Ce against log qe and that can give the slope and intercept of the graph individually. The value of n lies between 2 and 10, which suggests smart adsorption.

RESULTS AND DISCUSSION

Calibration curve

After determining the wavelength of the MG dye by using a UV-visible spectrophotometer, which showed a maximum peak at 618 nm. Different solutions of the dye were prepared with concentrations within the range of (0.01 – 9.0) mg l^{-1} to determine the calibration curve. Figure 2 displays the calibration curve of the MG dye.

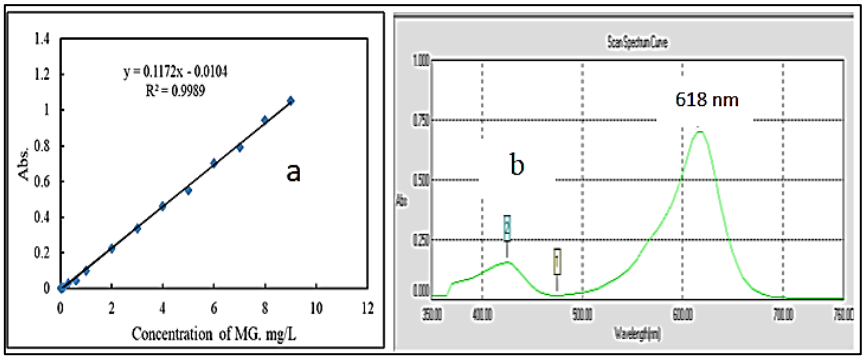

Figure 2. (a) Calibration curve and (b) absorption spectra of the MG dye.

Effect of dose weight

The effect of the surface capacity of tea leaves on MG dye was studied using different weights of 0.1 - 1.5 grams in 100 mL of dye at a concentration of 50 mg l^{-1}. After shaking for 3 hours, it was found that 0.1 g was the appropriate weight to obtain the best removal ratio. The removal ratio ranged from 76.2424% to 99.1515% in sequence with increased weight as Figure 3 shows. The percentage of the removal of the dye was calculated by Equation 1. The enhanced adsorption with the adsorbent dose may be caused by augmentation of surface area for dye molecule adsorption on the surface and more adsorption sites will occur and become high when the surface area increases [13]. Similar results were obtained by Israa [26] when studying the effect of adsorbent dose on removal.

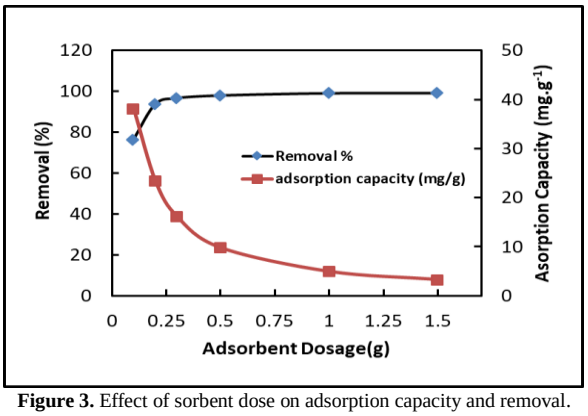

Figure 3. Effect of sorbent dose on adsorption capacity and removal.

The effect of contact time

The effect of the contact time was studied by running 6 identical experiments using 0.1 g of the adsorbent. 50 mg l-1 of dye was added to each flask and shaken for different durations of 20-180 min. Figure 4 displays the effect of the contact time on the percentage of removal and it was found that 120 minutes is the required time for optimal adsorption. It can be observed that the rate of adsorption is first high and then gradually slows down until saturation is reached. After 120 minutes from the beginning of the adsorption procedure, the dye's final concentration did not change considerably. It was observed that longer time showed constant results which indicates that the active sites on the surface of the adsorbent were saturated. As a result, equilibrium can be attained in 120 minutes. This outcome is foreseeable because there are initially a lot of surface sites accessible for adsorption, and subsequent 120 minutes, the residual surface sites are challenging to take because of the repelling interactions between bulk phases and the solute molecules of solute [17]. In comparison, Ashish et al. [27] found that contact time of 210 minutes when removing MG dye from aqueous solutions using Limonia acidissima shell as a low-cost absorbent while Emmanuel et al. [28] found that contact time of 90 minutes when removing MG dye from aqueous solutions using the pulverized teak leaf as absorbent.

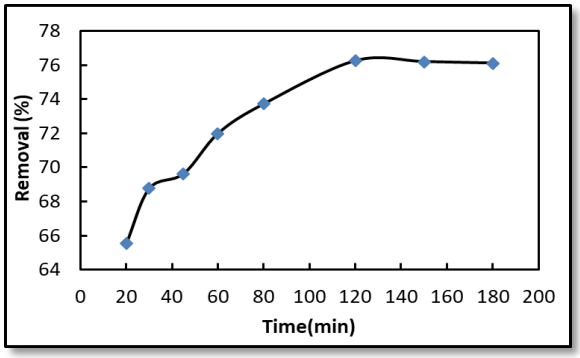

Figure 4. Influence of contact time on MG removal.

The particle size effect of adsorbent surface

The influence of the particle size of the tea leaves on the adsorption was studied at different particle sizes. 75 -850 micrometers were taken. Six identical experiments were run using 0.1 g of adsorbent with 100 mL of 50.00 mg l^{-1} MG dye shaken for 120 minutes. It was found that the removal ratio decreases when the particle size increases owing to the decreasing of the surface area of the particles, the percentage of removal was better at the size of 75 and 150 micrometers as can be seen in Figure 5. It is evident that as adsorbent particle size decreases, the adsorption capacity rises. This suggests that the smaller particle size of the oxidized black tea leaves for a given weight, the additional surface area, and the availability of binding sites that are accessible [3]. In comparison, Dakhil et al. [29] found that decreasing the particle size when removing direct red 28 dye using activated carbon which is prepared from rice husk as absorbent increases the removal.

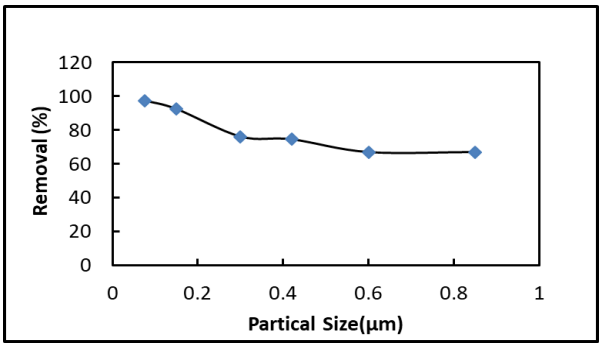

Figure 5. Particle size effect on MG removal.

The pH influence on adsorption process

The influence of the pH on the adsorption of the MG dye was studied by running six identical experiments at different pH of 2, 3.6, 4.3, 7.4, 8, and 9.5. The pH was adjusted using 0.1 M HCl and NaOH. The conditions of each experiment were 100 ml of the MG dye of 50.00 mg l-1, with 0.1 g minced oxidized black tea leaves with 150 micrometers particle size and shook for 120 minutes. Figure 6 indicates that the effect of the acidic function on the adsorption was clear at pH 8, which is due to the tendency of the dye to bind to the surface of the adsorbent more than remaining in the solution. In an acidic medium, the negative charge of the dye molecules disappears, and the adsorption is weak due to the increased strength of repulsion between identical charges, i.e. electrostatic repulsion of the dye with the surface [25]. A comparable tendency when the studying of pH factor was reported for MG adsorption by using Avena sativa as an adsorbent surface [30]. Similarly, Yongmei et al. [31] found that the pH was 8 when removing the MG dye from aqueous solutions by photocatalytic degradation using titanium dioxide under UV radiation. A similar value was reported by Marco et al.[32] when using chemical precipitation to remove MG dye from aqueous solutions by the magnetic adsorbent radiation.

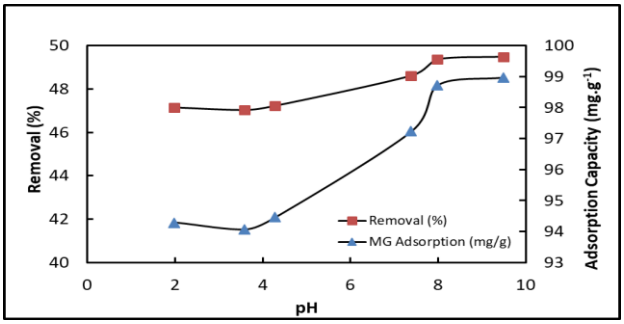

Figure 6. Influence of pH on adsorption capacity and removal.

The effect of the initial concentration of MG dye

The initial concentration effect of the MG dye on the adsorption capacity of the tea leaves' surface was studied. Different concentrations of the dye were taken 10.0, 30.0, 50.0, 70.0, 90.0, and 100.0 mg l^{-1} in 100 mL distilled water and added to six flasks containing 0.1 g oxidized black tea leaves with particle size 150 micrometers. After that shaking for 120 minutes and at pH 8. Figure 7 indicates that the capacity of sorption increased with the increase of dye concentration, and reached 97.887 mg g^{-1}.

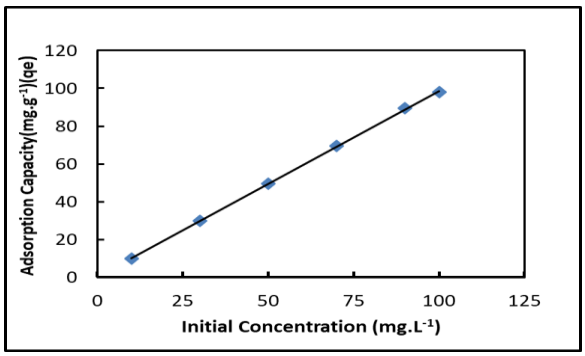

Figure 7. Initial concentration effect on adsorption capacity and removal.

The concentration acts as a key impetus for overcoming the dye's mass transfer impedance between solid phases and the aqueous [19]. As a similar result, Ashish et al. [27] found that increasing the initial concentration of MG dye when it's removed by the Limonia acidissima shell as a low-cost absorbent causes an increase in the adsorption capacity. Table 2 shows a comparison of the adsorption

capacity of MG by oxidized black tea leaves with other adsorbents and with different removal techniques.

Table 2. Adsorption capacity comparison of MG by adsorbents with different techniques.

Adsorbent	Techniques	Adsorption capacity (mg g^{-1})	Ref.
Banana stem	Adsorption	8.29	[33]
***Catha edulis* stem**	Adsorption	4.55	[25]
Avena sativa	Adsorption	80	[30]
Keggin polyoxometalate	Co-precipitation	37.51	[34]
carbon nanotube	Adsorption	80.64	[35]
Diatomite	Adsorption	23.64	[36]
ZnO-activated carbon	Ultrasonic	66.68	[37]
Deinococcus radiodurans R1	Biological decolorization	7.63	[38]
Oxidized black Tea Leaves	Adsorption	97.8	Current work

Adsorption kinetics

Langmuir line for the sorption of MG dye above oxidized black tea leaves is demonstrated in Figure 8a while Figure 8b expresses the Freundlich line of the sorption of MG dye. The calculated isothermal MG sorption parameters of Freundlich and Langmuir are tabulated in Table 3. The Langmuir isotherm's applicability indicated a monolayer coverage of MG dye on the surface of oxidized black tea leaves.

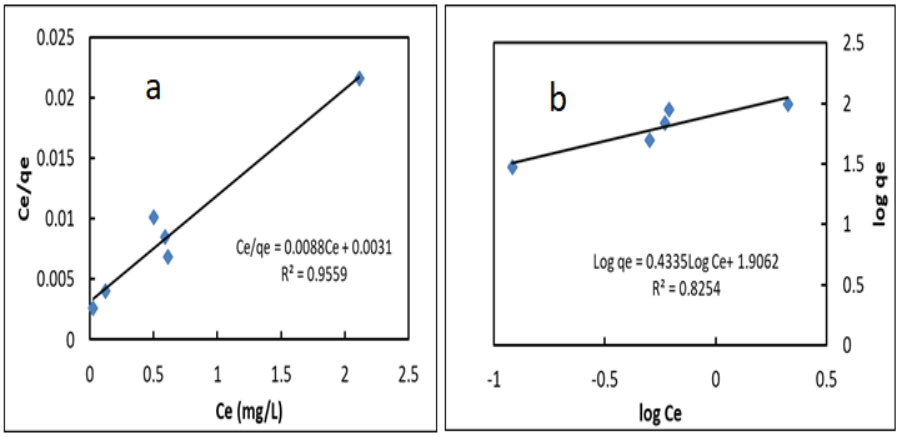

Figure 8. (a) Linearized isotherm of Langmuir model (b) Freundlich model of MG.

Table 3. Sorption isotherm features removal for MG dye for the two models.

Langmuir		Freundlich	
q_m (mg g^{-1})	113.6363	K_F (mg g^{-1})(L mg^{-1})$^{1/n}$	80.5749
Ka (L mg^{-1})	2.839	1/n	0.4335
R^2	0.9559	R^2	0.8254
R_L	0.0340 – 0.00351		

The separation issue RL values show in Figure 9, that they were within the range of 0 – 1, which indicates that this sorption method was satisfactory. Also, the value 1/n obtained from the Freundlich isotherm was between 0 – 1. Furthermore, this confirms that the sorption is acceptable and suitable. The greater the value of KF, the greater the removal of dye by adsorption, because this worth elucidates an exceedingly heterogeneous surface when it is near zero [17].

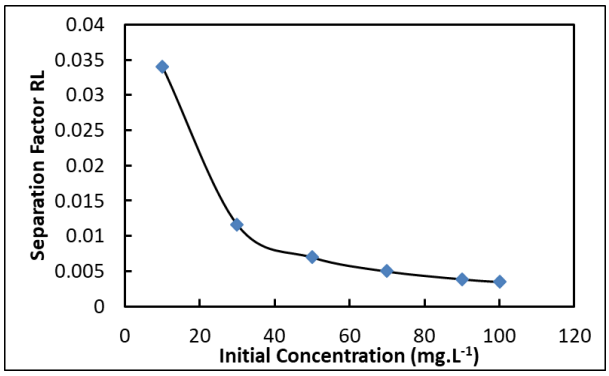

Figure 9. The separation factor initial of MG concentration.

The deviation of those models from the achieved data clarifies Figure 10. It appears that the MG sorption may fit well on oxidized black tea leaves by Langmuir isotherms.

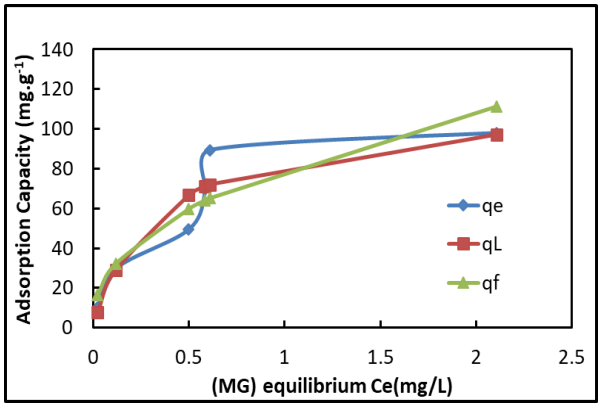

Figure 10. Comparison of calculated and experimental data of the equilibrium isotherm by Langmuir and Freundlich models.

CONCLUSIONS

The current study reveals that oxidized black tea leaves can be simply used as a good removal material for MG depending on the data of removal percentage. This method is characterized by the ease and low cost of preparing the adsorbent surface as well as its high efficiency of MG dye adsorption. A 99.15% of 50 mg l^{-1} removal amount of MG dye was found at 1.5 g of oxidized black tea leaves, at 120 min shaking time and pH 8. This work demonstrates that the sorption of the dye could be boosted on oxidized black tea leaves by obeying Langmuir better than Freundlich isotherm models depending on the R^2 value.

CONFLICT OF INTERESTS

The authors declare no conflict of interest.

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