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Removal of Brilliant Green Dye from Aqueous Solutions Using Multi-walled Carbon Nanotubes (MWCNTs): Linear and Nonlinear Isotherm Models and Error Analysis

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ABSTRACT

Current research explains the comparison of linear and nonlinear regression methods for finding the optimal isotherm study using experimental data for the adsorption of BG on multi-walled carbon nanotubes MWCNTs. BG dye maximum adsorption onto MWCNTs occurred at pH 2 and 35°C, with the apparent equilibrium reached after 15 min. In this study, five error functions were used: ERRS, Hybrid, Chi-square (χ^2), ARE, and EABS. The values of error functions suggest that the Langmuir Linear type 3 is a suitable isotherm to describe the adsorption of BG on MWCNTs. The results showed that the Langmuir isotherm is a fit good isotherm to describe the adsorption process. The coefficient of non-determination (K^2) showed Hybrid, and ERRS were the preferable error functions used to predict the fit of linear and nonlinear isotherm models. Compared with other studies, MWCNTs can be used as a low-cost adsorbent with low contact time for the removal of BG dye from an aqueous solution.

INTRODUCTION

Water pollution is one of the major causes of environmental pollution today. Dyes are colored organic compounds that are discharged into wastewater from many industries and cause serious pollution to the environment and humans (Al-Tohamy et al. 2022). Brilliant Green (BG) dyes are cationic, odorless green crystals and belong to the triphenylmethane family (Singh et al. 2022). This dye is dangerous and toxic to humans and animals, as it causes irritation in the digestive system and is a carcinogenic and mutagenic substance. When exposed for a long time, it leads to damage to organs, including the lungs (Mansour et al. 2021, 2020, Abbas 2020). It generates carbon dioxide, sulfur oxides, and nitrogen oxides during its decomposition. Therefore, removing this dye from the aqueous solution is very important because of its complete solubility (Salem et al. 2016, Rehman et al. 2013). It is used for a variety of purposes, including veterinary medicine, dyeing textiles, printing paper, and as an additive in poultry feed to prevent the formation of parasites and fungi (Mariah & Pak 2020, Fiaz et al. 2020). Adsorption is a common technique used to remove dyes and harmful substances from aqueous solutions. This technique has the advantages of low operating costs, simplicity, and high efficiency (Mansour et al. 2021, 2020, Ali 2018). Many researchers used a variety of sorbents to remove this dye, such as red clay (Rehman et al. 2013), areca nut husk (Baidya & Kumar 2021), sawdust (Mane & Babu 2011), Luffa Cylindrical Sponge (Segun Esan et al. 2014), rice husk ash (Dahlan et al. 2019) and Ficus (Gul et al. 2023). Carbon nanotubes (CNTs) can be divided into two general types: single-walled carbon nanotubes(SWCNTs) and multi-walled carbon nanotubes (MWCNTs) (Alkaim et al. 2015). CNTs have properties similar to those reported for fullerenes, but other novels are of great importance in the fields of biology, electricity, and the environment (Rodríguez et al. 2020). CNTs have the potential to deliver drugs to cancer sites in a targeted and sustained manner (Raza et al. 2016). MWCNTs are inexpensive surfaces that have many properties as new and powerful adsorbents due to their large specific surface area, small size, and negative surface charge (Saxena et al. 2020, Abujaber et al. 2019). MWCNTs are more effective at removing cations due to the attractive force between the surface and the positively charged dye molecules with very short contact times, less amount, and high adsorption capacity (Karimifard & Alavi Moghaddam 2016, Ghaedi et al. 2016).

MWCNTs have gained great appeal due to unique properties such as electrical conductivity, large surface area, high mechanical strength, and chemical stability. Water-soluble functional groups such as carboxyl and amino groups are used for making the surface of MWCNTs hydrophilic due to their surface being hydrophobic (Hayat et al. 2022). It was widely used in electronic transistors (Gupta et al. 2009), biosensors (Crini et al. 2006), and optical components (Rida et al. 2013) because of its unique chemical, mechanical, and electronic properties (Wang et al. 2012). This study aimed to use MWCNTs as a low-cost, highly efficient, and available absorbent for the removal of BG dyes from aqueous solutions. The effect of different adsorption factors, such as solution pH, contact time, and temperature, was studied. In the present work, the initial behavior of BG dye removal onto MWCNTs was analyzed by the Langmuir and Freundlich isotherm models with linear and nonlinear regressions with error functions applied.

MATERIALS AND METHODS

Equipment

UV-visible (1800 Shimadzu) spectrophotometer, A shaking water bath (BS-11 digital, JETO Korea, TECH), and a pH meter (Hanna) were used in this study.

Chemicals and Solutions

Multiwall Carbon Nanotubes (purity 90%) was obtained from Cheap Tubes Inc. (Grafton, VT 05146 USA) with a tube length of 10–30µm and outer diameter (o.d.) of 10–30 nm, brilliant green (BG) (Sigma-Aldrich), NaOH (BDH) and HCl (Fluka) were analytical reagent. 0.01 gm of BG dye was dissolved in 100 mL distilled water(100 $mg.L^{-1}$). (0.5-3.5 mL) of BG dye were transferred from 100 $mg.L^{-1}$ solution BG dye followed by dilution using 25 ml of distilled water to prepare a series of various concentrations from 2 to 14 $mg.L^{-1}$. The pH effect study of BG solutions was adjusted using (0.003 $mol.L^{-1}$) of HCl and NaOH.

Adsorption Studies

A set of tubes containing 10 mL of 10 $mg.L^{-1}$ of BG dye solutions were used to carry out adsorption experiments. Three affected factors were investigated, namely: pH (2-9), temperature (35°C, 45°C, 55°C), and contact time (10-30 min.). The calibration curve of the BG solution showed that a maximum absorbance appeared at λ_{max} of 624 nm and the molar absorptivity (Є = 0.131 $L.g^{-1}.cm^{-1}$). Three different temperatures (25°C, 35°C, 45°C, and 55°C) were tested as the isotherm for the adsorption of BG dye onto MWCNTs for 15 min. The efficiency adsorbed amount qe of adsorption BG dye and removal% defined as in equations (1) and (2) (Song et al. 2018, Das et al. 2018):

$$q_e = ((C_O - Ce) \times V/m \quad \ldots(1)$$

$$\text{Removal\%} = (C_o - Ce)/C_o \times 100 \quad \ldots(2)$$

Where Co and Ce are the initial and final concentrations of BG dye ($mg.L^{-1}$), respectively, m is the weight of MWCNTs surfaces (gm), and V is the volume of BG dye (L) (Rostamian & Behnejad 2018). The adsorption of a dye molecule onto a surface can be described by different models (like linear and nonlinear Langmuir and Freundlich), depending on the characteristics of the system being studied (Belhachemi & Addoun 2011). Langmuir isotherm described that there is a single layer of dye molecules on the surface and that the adsorption process is reversible and homogeneous. The Freundlich isotherm model is multilayer adsorption, where the adsorbate molecules form layers on top of each

Table 1: Equations of adsorption isotherm.

Isotherm	Equation	Linearized form	Plot	Parameters
Langmuir type1	$q_e=q_m K_L C_e/(1+KLCe)$	$\frac{Ce}{qe}=\frac{1}{qmKL}+\frac{Ce}{qm}$	(Ce/qe) vs. Ce	$qm = (\text{slope})^{-1}$, K_L = (slope/intercept)
Langmuir type2		$\frac{1}{qe}=\frac{1}{qmKLCe}+\frac{1}{qm}$	$1/q_e$ vs. $1/Ce$	$qm = (\text{intercept})^{-1}$, K_L =(intercept/slope)
Langmuir type3		$qe = qm - (\frac{1}{KL})\frac{qe}{Ce}$	qe vs. qe/Ce	qm = intercept, $K_L = -(\text{slope})^{-1}$
Langmuir type4		$\frac{qe}{Ce} = KLqm - Klqe$	qe/Ce vs. qe	qm=–(intercept/slope), K_L =–slope
Freundlich	$q_e = KFCe^{1/n}$	Log (q_e) =1/n log (C_e) +log(K_F)	$Log q_e$ vs. $Log C_e$	K_F = exp(intercept), $n = (\text{slope})^{-1}$

q_m ($mg.g^{-1}$) and K_L ($mL.mg^{-1}$) are Langmuir constants; K_F ($mL.mg^{-1}$) is the Freundlich constant, and n is the intensity of adsorption of the Freundlich isotherm model

Table 2: Equations of error functions.

Error functions	Equations
Square error function (ERRS)	$\sum_{i=1}^{n}(q_{\text{cal.}} - q_{exp.})^2$
Hybrid fractional error function(HYBRID)	$\frac{100}{\text{n} - \text{p}}\sum_{i=1}^{n}\frac{(q_{\text{cal.}} - q_{exp.})^2}{q_{\text{cal.}}}$
Chi-square(X^2)	$\sum_{i=1}^{n}\frac{(q_{\text{cal.}} - q_{exp.})^2}{q_{\text{cal.}}}$
The average relative error (ARE)	$\frac{100}{\text{p}}\sum_{i=1}^{n}\frac{(q_{\text{cal.}} - q_{exp.})^2}{q_{\text{cal.}}}$
the sum of the absolute errors (EABS)	$\sum_{i=1}^{n}\lvert(q_{\text{cal.}} - q_{exp.})\rvert$

other. Freundlich supposed that the adsorption surface is heterogeneous (Table 1) (Sala et al. 2014).

Error Analysis

Error functions are mathematical functions that are used to determine how well a particular isotherm model fits the experimental data. In this study, three error functions were calculated using Microsoft Excel and the origin lab® 16 (Table 2) (Subramanyam & Das 2014, McKay et al. 2014, Shahmohammadi-Kalalagh & Babazadeh 2014).

RESULTS AND DISCUSSION

Effect of pH

The acidity of the medium is the most important factor in adsorption (Machado et al. 2011). Different pH in the range (2-10) was studied (Fig. 1). All solutions were prepared at 0.003 mg of MWCNT dosage, 10 mg/L of initial BG dye concentration, and 35°C for 15 min. The maximum percentage of BG dye removal when pH 2 was 92.8% (Fig. 2). An acidic medium (pH2) increases the removal of BG dye because of the electrostatic attraction between BG dye and the positive charge on the MWCNTs surface (Abualnaja et al. 2021).

Effect of Contact Time and Temperature

The contact time influence of BG with 0.003 gm of MWCNTs was investigated for solutions with pH 2 and 10 mg.L^{-1} concentration. The tests were achieved at 25, 35, 45, and 55°C (Fig. 3). The high removal percentage of BG onto the MWCNTs was achieved at 15 min and 35°C to sponsor the high adsorption and completed reaction. At first, the high rate of removal percentage is due to the greater number of active sites present on the surface of MWCNTs at 15 min and 35°C. After that, it becomes constant due to the lesser number of active sites left on the adsorbent surface (Figs. 4 and 5).

Linear Isotherm Models

Five error functions- ERRS, HYBRID, Chi-square(χ^2), ARE, and EABS have been used as evidence to establish the best isotherm in this study. The linear forms type 2 of

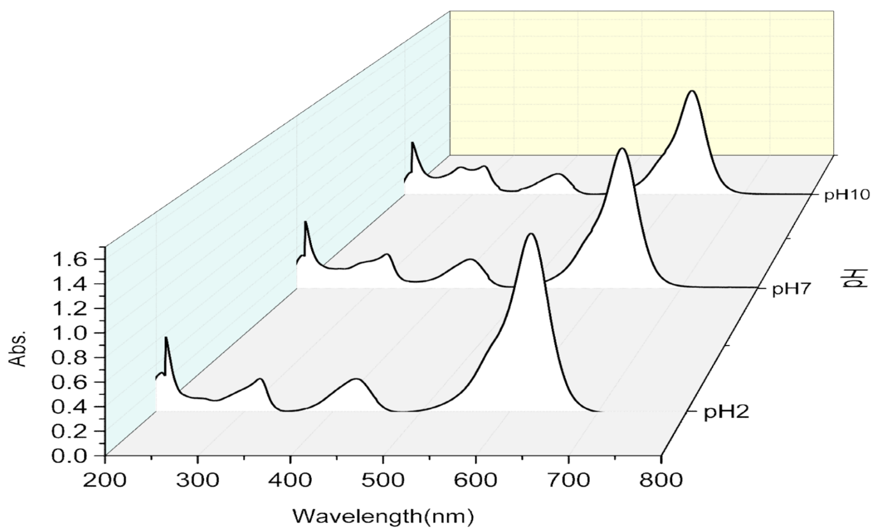

Fig. 1: pH stability of BG dye.

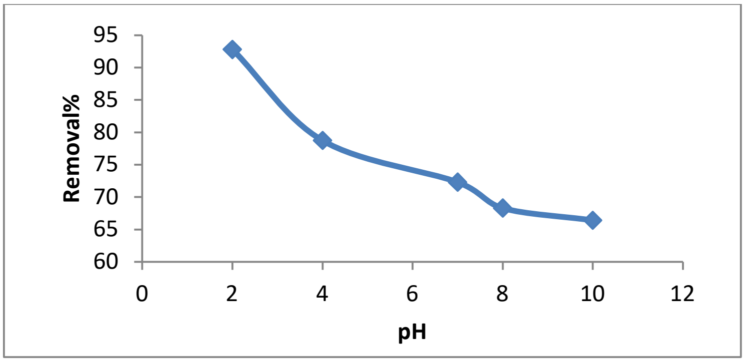

Fig. 2: Effect of pH on adsorption of BG dye onto MWCNTs.

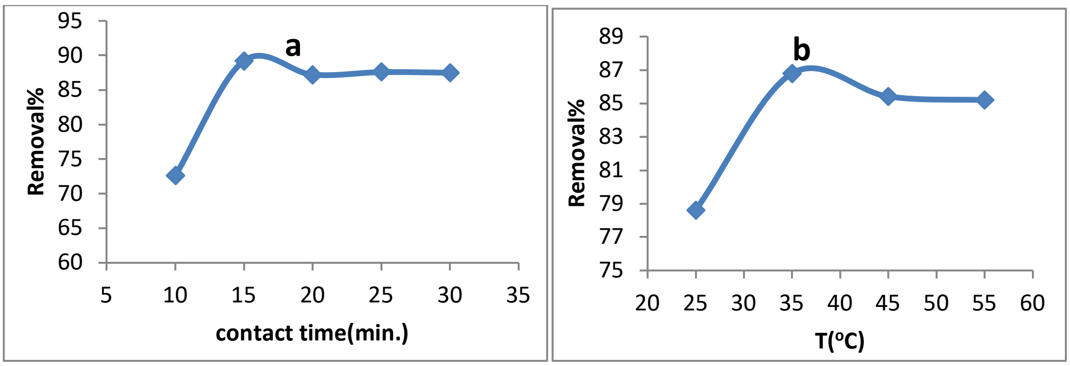

Fig. 3: Effect of (a) contact time and (b) temperature on adsorption of BG dye adsorption by MWCNTs.

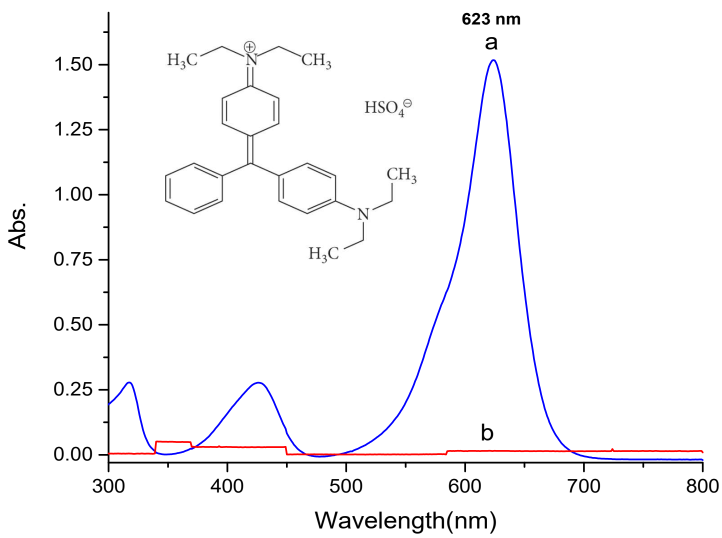

Fig. 4: UV-Vis absorption spectrum of 12 mg.L^{-1} BG dye solution: a- before adsorption and b- after adsorption with 0.003 g of MWCNTs at pH 2, 15 min. and 35°C.

Langmuir isotherm models have shown a lower value of error function and the highest values of (R^2 0.963) compared to other forms (Fig. 6). The low Langmuir error function value suggests that the adsorption of the BG dye onto the MWCNTs surface is a monolayer process. The surface is homogeneous with a limited number of identical adsorption sites, and the adsorption process is monolayer and reversible (Ayawei et al. 2017). According to the low value of the error

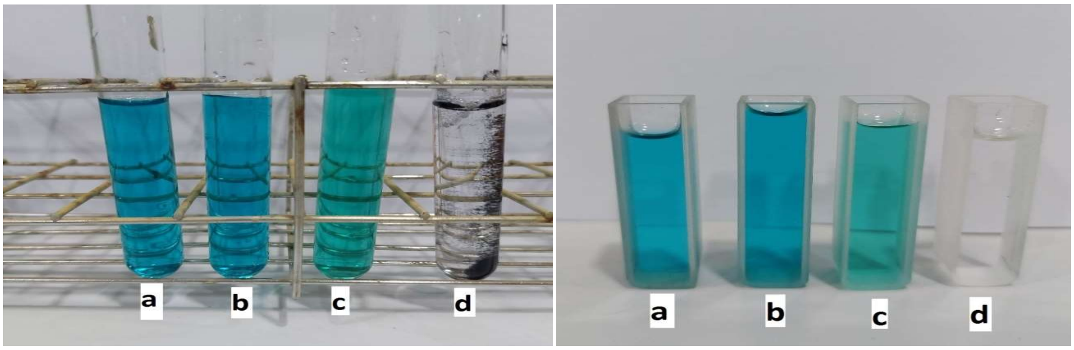

Fig. 5: photograph of 12 mg.L^{-1} brilliant green dye before adsorption in a - pH 9, b - pH 7, c - pH 2, and d - after adsorption process with 0.003 g of MWCNTs at pH 2, 15 min, and 35°C.

functions, it could be considered that the linear Langmuir model is a more suitable isotherm for this study than the linear Freundlich model (Fig.7) (Table 3) (Piccin et al. 2011, Kumar & Sivanesan 2005, 2006).

Nonlinear Isotherm Models

Origenlab®16 was used to find the nonlinear forms of Langmuir and Freundlich isotherm (Fig. 8). Nonlinear

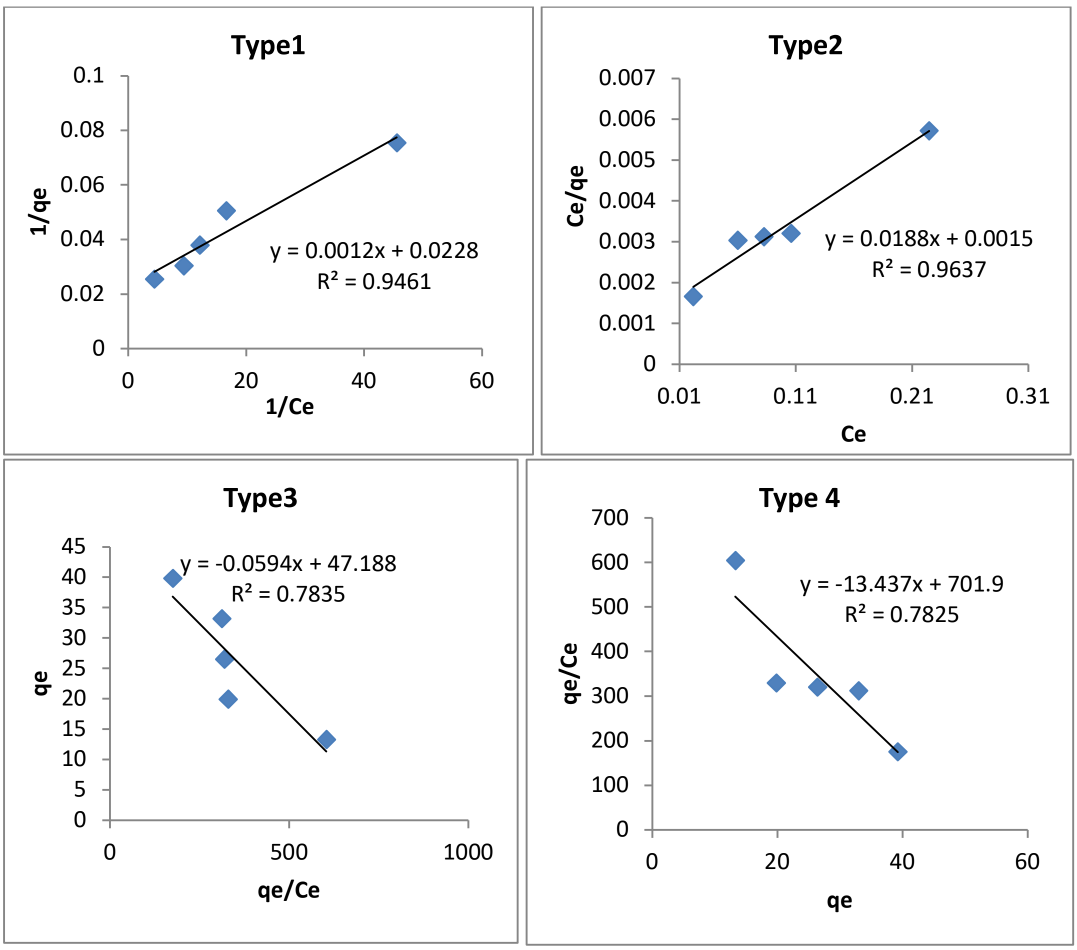

Fig. 6: Linear Langmuir isotherm models for removal of BG dye at 35°C.

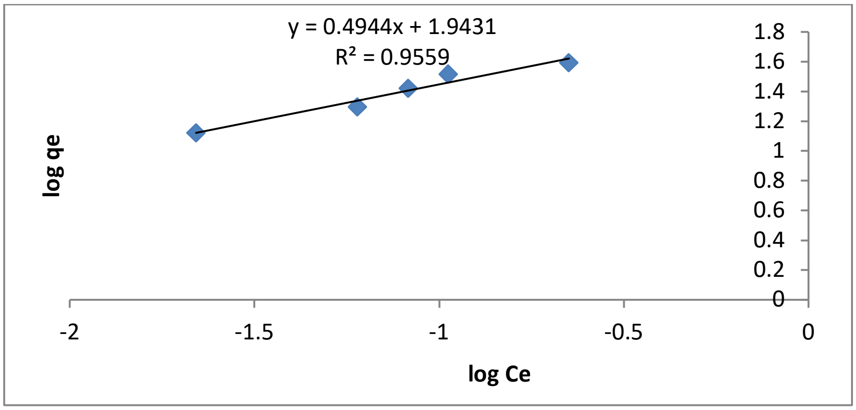

Fig. 7: Linear Freundlich isotherm models for removal of BG dye at 35°C.

Table 3: Adsorption parameters of BG dye onto MWCNT at 35^{o}C for linear Langmuir and Freundlich isotherms.

Adsorbent	Isotherm models	Parameters value		ERRS	HYBRID	χ^2	ARE	EABS
MWCNTs	Langmuir Linear type1	KL	0.045	2.602	**0.011**	0.647	42.452	4.375
		q_m	1000					
		R^2	0.946					
	Langmuir Linear type2	K_L	0.055	3.892	81.643	79.624	49.997	5.267
		q_m	1000					
		R^2	**0.963**					
	Langmuir Linear type3	K_L	16.949	0.028	0.019	**0.081**	4.094	**0.002**
		q_m	47.18					
		R^2	0.783					
	Langmuir Linear type4	K_L	52.263	**0.020**	0.016	0.090	**3.922**	0.037
		q_m	13.430					
		R^2	0.782					
	Freundlich linear	K_f	87.700	3.851	0.448	24.921	49.737	5.239
		n	2.024					
		R^2	0.955					

Langmuir isotherm is the best nonlinear isotherm due to the higher value of R^2 and the lowest value of the error function compared to the nonlinear Freundlich isotherm (Table 4).

R^2 is limited in its ability to identify the better fitting of the models, essentially when the models under consideration have different numbers of variables or different functional forms. Therefore, the error function gives a good fitting of data to isotherm models. The isotherm models of this study could be arranged as follows: Langmuir Linear type 3>Langmuir Linear type 4>Langmuir non-linear> Langmuir Linear type 1> Freundlich nonlinear>Freundlich linear> Langmuir Linear type 2.

The Relationship Between Error Functions

The coefficient of non-determination (K^2) measures the amount of unexplained variance between two variables or between a set of predictors and an outcome variable (like error functions). When the relationship between the variables or the predictors and the outcome variable becomes weaker, the K^2 value is higher. On the other hand, when the relationship between the variables or the predictors and the outcome variable became stronger, the K^2 value is smaller (Hami et al. 2021, Sivarajasekar & Baskar 2019)

$$K^2 = \frac{\text{Unexplained variance}}{\text{Total variance}}$$

$$= 1 - \frac{Unexplainedvariance}{\text{Total variance}} = 1 - r^2$$

HYBRID had shown minimal unexplained isotherm for Langmuir linear type 1, 3, 4, and Freundlich linear,

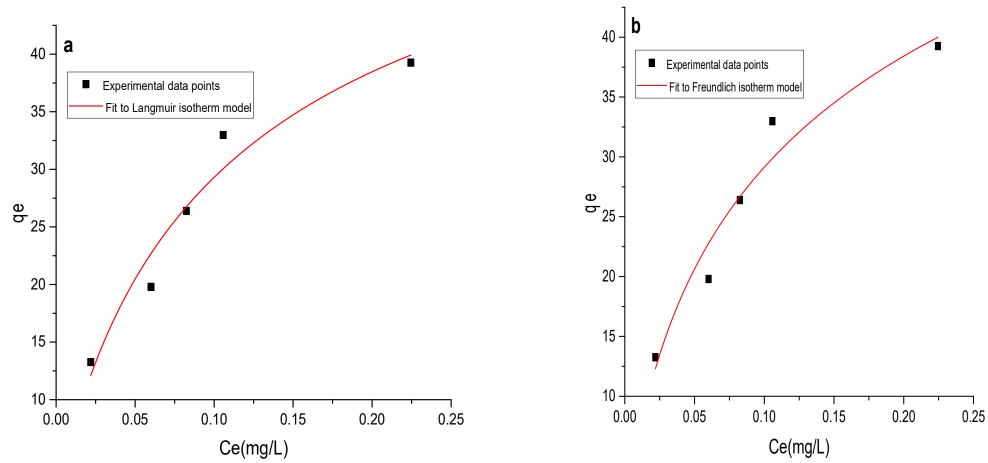

Fig. 8: Adsorption parameters of BG dye onto MWCNT at 35°C for nonlinear (a) Langmuir and (b) Freundlich isotherms.

Table 4: Adsorption parameters of BG dye onto MWCNT at 35°C for nonlinear Langmuir and Freundlich isotherms.

Adsorbent	Isotherm	Parameters value		ERRS	HYBRID	□^{2}	ARE	EABS
MWCNTs	Langmuir nonlinear	K_L	6.3552	0.129	**0.014**	0. 082	10.350	0.932
		q_m	62.550					
		R^2	**0.955**					
	Freundlich nonlinear	K_f	0.201	2.873	**0.016**	0.911	44.195	4.581
		n	0.187					
		R^2	0.952					

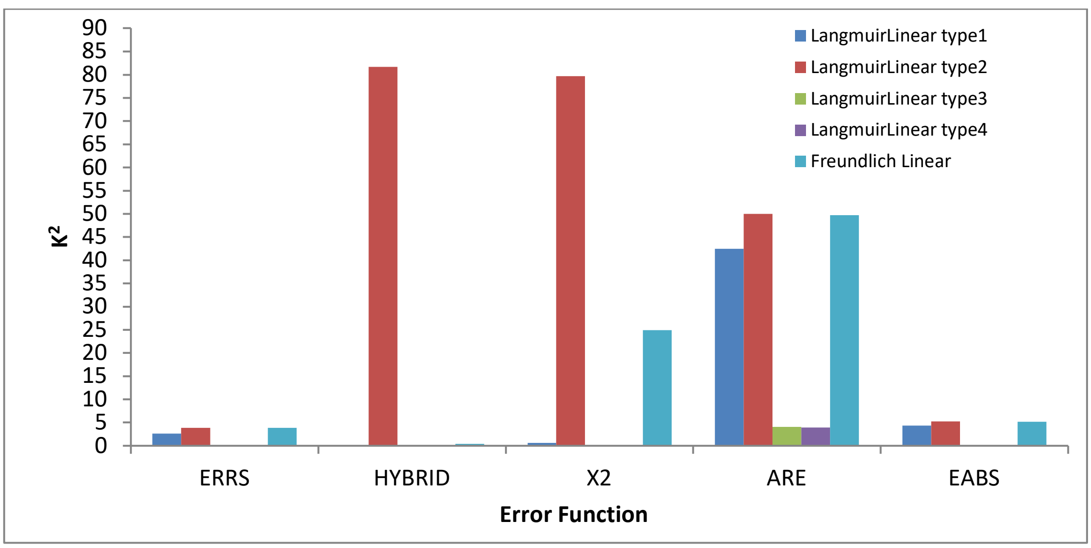

Fig. 9: The relationship between linear Langmuir and Freundlich isotherms with error functions of BG dye adsorption onto MWCNTs.

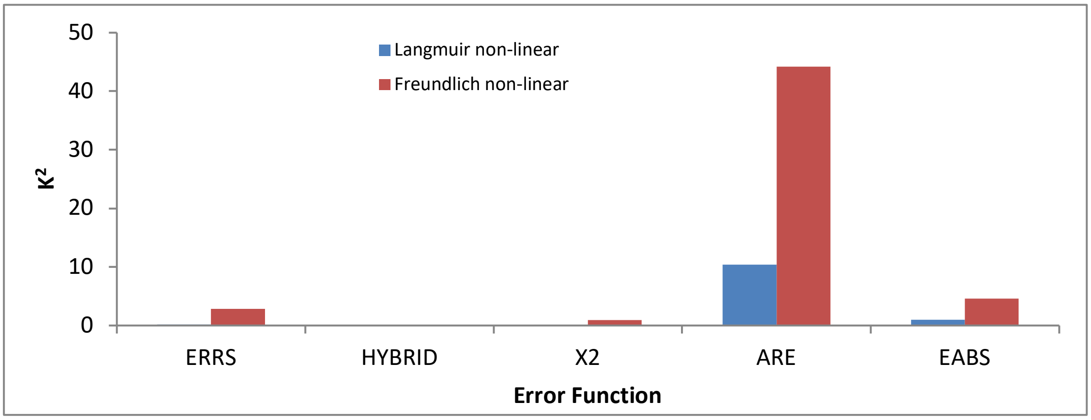

Fig. 10: The relationship between nonlinear Langmuir and Freundlich isotherms with error functions of BG dye adsorption onto MWCNTs.

Table 5: Comparison with other studies.

Adsorbent	pH	Contact time	isotherm	Ref.
Red clay	7	4 h	Redlich–Peterson	(Rehman et al. 2013)
Peat soil in Brunei Darussalam	4.9	60 min.	Redlich–Peterson	(Chieng et al. 2015)
Activated carbon derived from guava tree wood	7	20 min.	Freundlich	(Mansour et al. 2020)
Modified *Bambusa Tulda*	7	60 min.	Langmuir	(Laskar & Kumar 2019)
Surfactant Doped Polyaniline/MWCNTs Composite	3	240 min.	Langmuir	(Kumar et al. 2014)
MWCNTs	2	15 min.	Langmuir Linear type 3	This study

but ERRS had shown minimal unexplained isotherm for Langmuir linear type 2 (Table 3) (Fig. 9). HYBRID was shown to minimize the distribution of error between the empirical and expected nonlinear isotherm (Table 4) (Fig. 10).

Comparison with Other Studies

The adsorption of BG dye on the MWCNTs surface had a better contact time than other surfaces of other works (Table 5).

CONCLUSION

In this work, the linear Langmuir isotherm type 3 performed better compared to the nonlinear isotherm model for the adsorption of BG dye onto MWCNTs. As a result, the optimal conditions for adsorption of BG dye with pH 2, 15 min of shaking time, and 35°C of temperature. The coefficient of non-determination (K^2) showed Hybrid, and ERRS were the preferable error functions used to predict the fit of linear and nonlinear isotherm models.

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