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Linear and Non-linear Regression Methods to Study the Adsorption of Cr(VI) from an Aqueous Solution using Pomegranate Peel

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ABSTRACT

In this study, the previous data of the adsorption of Chromium (VI) ions on the pomegranate peels (MPGP) as adsorbents were used. linear and non-linear equations for Langmuir, Freundlich, Dubinin-Radushkevich, Tempkin, Redlich-Peterson, Sips, and Toth adsorption isotherm models were applied. The five error functions ERRSQ, HYBRD, MPSD, ARE, and EABS were analyzed for non-linear isotherm equations and in each case a set of isotherm parameters were determined. The algorithms for the simulation of linear and non-linear isotherm models using error functions were achieved with the aid of Microsoft Excel solver Add-Ins for minimizing the respective error function across the concentration range studied. Linear and non-linear methods show comparable data for two-parameter models, also high R^2 values were obtained for Langmuir, Freundlich, and Temkin models while the Dubinin-Radushkevich model reveals low R^2 values, on the contrary, three-parameter models show different data between linear and non-linear methods with high R^2 value for all. Which indicates that the error does not obey the Gaussian distribution.

Keywords: Chromium (VI), pomegranate peel, linear regression, non-linear regression, error functions, adsorption.

الخلاصة

في هذه الدراسة استخدمت بيانات سابقة لامتزاز ايون الكروم السداسي على قشور الرمان وتم تطبيق المعادلات الخطية وغير الخطية لموديلات ايزوثيرمات الامتزاز Langmuir, Freundlich, Dubinin-Radushkevich, Tempkin, Redlich-Peterson, Sips, and Toth وتم تحليل البيانات للمعادلات غير الخطية باستخدام خمسة دوال من دوال الخطأ وهي ERRSQ, HYBRD, MPSD, ARE, and EABS , وفي كل حالة يتم تعيين مجموعة المعاملات أو الثوابت لمعادلة الايزوثيرم وتمت هذه العملية باستخدام برنامج الاكسل Excel من شركة مايكروسوفت باستخدام الدالة solver Add-Ins لتقليل الخطأ او لايجاد التقارب بين القيم العملية و القيم النظرية المستخرجة من معادلات ايزوثيرمات الامتزاز. أظهرت الطريقة اعلاه نتائج متطابقة مع المعادلات الخطية لايزوثيرمات الامتزاز التي تمتلك معاملين أو ثابتين two-parameter وكذلك تم الحصول على قيم عالية من دالة الخطأ معامل التحديد (R2) coefficient of determination لموديلات الامتزاز Langmuir, Freundlich, and Temkin بينما أظهر موديل Dubinin-Radushkevich قيم واطئة لدالة الخطأ (٢R). وعلى العكس اظهرت الايزوثيرمات التي تمتلك ثلاث معاملات او متغيرات Redlich-Peterson, Sips, and Toth نتائج مختلفة عن بعضها البعض للمعادلات الخطية وغير الخطية مع الحصول على قيم عالية لدالة الخطأ (٢R). ان اختلاف قيم ثوابت ايزوثيرمات الامتزاز للمعادلات الخطية وغير الخطية يثبت بان الخطأ (الانحراف بين القيم العملية والنظرية) لا يطيع قانون التوزيع لكاوس Gaussian distribution.

INTRODUCTION

The absorption process is a very effective way to remove pollutants from an aqueous solution [1-5]. Sorption processes include the selective transfer of solute components in the fluid phase onto the surface or the bulk of solid adsorbent materials. Many isotherm models try to describe the adsorption mechanism and to determine which the best isotherm equation is can describe the interaction between the adsorbent and the solute, two parameters (Freundlich, Langmuir, Temkin, Dubinin-Radushkevich) and three-parameters

(Redlich-Peterson, Toth, and Sips) isotherm models were used to fit the experimental data. Because of the main shortage of linear regression method in fitting models and their parameters evaluation which include: Error changes discrepancy [6] and unsuitability for models with more than two parameters [7], both linear and non-linear regression methods were used to test the fitness of these models and their parameters evaluation using the Sum of the Squares of the Errors (ERRSQ), A Composite Fractional Error Function (HYBRD), A Derivative of Marquardt's Percent Standard Deviation (MPSD) [8], The Average Relative Error (ARE) [9], The Sum of the Absolute Errors (EABS) and coefficient of determination (R^2) error functions, to obtain best experimental data fitness and estimation of models coefficients using Microsoft Excel® solver Add-Ins. The R^2 error function was maximized while the others were minimized.

MATERIALS AND METHODS

The pomegranate peel was gathered from the northern region of Iraq, dried and then ground into particle size of 500µm after being sieved. The resulting biomass was mixed with distilled water and shaken overnight. This process was repeated 10 times or until the color was removed. The mixture was filtered and air dried at 80˚C overnight. The final product was named as modified pomegranate peel.

Error Functions

A number of error functions were used. The expressions of these error functions are illustrated below [8][11]:

$$\text{ERRSQ} = \sum_{i=1}^{p} \left(q_{e,meas} - q_{e,calc}\right)_i^2 \quad (1)$$

$$\text{HYBRD} = \sum_{i=1}^{p} \left[\frac{(q_{e,meas} - q_{e,calc})^2}{q_{e,meas}}\right]_i \quad (2)$$

$$\text{MPSD} = \sum_{i=1}^{p} \left(\frac{q_{e,meas} - q_{e,calc}}{q_{e,meas}}\right)_i^2 \quad (3)$$

$$\text{ARE} = \sum_{i=1}^{p} \left|\frac{q_{e,meas} - q_{e,calc}}{q_{e,meas}}\right|_i \quad (4)$$

$$\text{EABS} = \sum_{i=1}^{p} \left|q_{e,meas} - q_{e,calc}\right|_i \quad (5)$$

$$R^2 = \frac{\sum(q_{e,meas} - \underline{q}_{e,calc})^2}{\sum(q_{e,meas} - \underline{q}_{e,calc})^2 + \sum(q_{e,meas} - q_{e,calc})^2} \quad (6)$$

For the non-linear method, a trial-and-error technique was used to find the isotherm parameters by minimizing the error functions, while maximizing R^2, using the solver add-in with Microsoft's spreadsheet, Microsoft Excel [12][13]. In each case a set of isotherm parameters were determined; it is likely to be different in each case; by minimizing the respective error function across the concentration range studied [11]. To prevent a bias in which set we adopted, we used the sum of the normalized errors (SNE), for each set of parameters and isotherms. The (SNE) can find it by dividing the values of error function sets (except R^2 values) over their respective maximum value [13][14]. Table 1 appears in this method by using our data for the adsorption of Cr(VI) onto MPGP at 20 ℃ applying Freundlich isotherm.

Isotherm Models

The isotherm models represent the distribution of solute molecules between solid phase (adsorbent) and the liquid phase at an equilibrium state. Table 2 shows the entire isotherm models used which include two and three parameters.

Table 1. The Freundlich isotherm parameters for Adsorption of Cr(VI) onto MPGP at 20 ℃ using (SNE) method for non-linear and R^2 for linear.

METHOD/ERROR FUNCTION (PARAMETER SET)						
MPGP	Linear	Non-linear				
		ERRSQ	HYBRD	MPSD	ARE	EABS
K_F (MG/G) (L/MG)	1.515384	1.83965	1.64539	1.51249	1.50952	1.84497
N	1.506444	1.71011	1.59420	1.51201	1.56826	1.75317
R^2	0.95453	0.96796	0.96437	0.95651	0.93883	0.95899
SUM OF ERRORS^2 (ERRSQ)	1.29098	**0.89946**	1.00393	1.23587	1.82319	1.^{16}849
HYBRID ERROR FUNCTION (HYBRD)	0.15114	0.15767	**0.13569**	0.14784	0.23516	0.18450
MARQUARDT'S PSD (MPSD)	0.01955	0.03500	0.02202	**0.01933**	0.03128	0.03567
AVERAGE RELATIVE ERROR (ARE)	0.27677	0.31191	0.28953	0.27550	**0.26830**	0.30931

SUM OF ABSOLUTE ERRORS (EABS)	2.04898	1.79444	1.92299	2.03657	1.93261	**1.74653**
SUM OF NORMALIZED ERRORS (SNE)	------	4.02620	**3.61745**	3.73181	4.68606	4.27472

Table 2. Isotherm models equations used for the Cr(VI) adsorb onto MPGP.

Two Parameter Models				
Model	**Linear eq.**	**Non-linear eq.**	**Plot**	**Slope and intercept**
Freundlich	$logq_e = logK_f + \frac{1}{n}\ log\ log\ C_e$	$q_e = K_f\ C_e^{\frac{1}{n}}$	log q_e vs $log\ log\ C_e$	slope= $\frac{1}{n}$, Intercept $= logK_f$
Langmuir	$\frac{1}{q_e} = \frac{1}{K_L q_{max}}\frac{1}{C_e} + \frac{1}{q_{max}}$	$q_e = \frac{q_m K_L C_e}{1 + K_L C_e}$	$\frac{1}{q_e}$ vs $\frac{1}{C_e}$	slope=$\frac{1}{K_L q_{max}}$, Intercept= $\frac{1}{q_{max}}$
Dubinin-Redushkevich	$ln\ ln\ q_e = lnq_m - K_{DR}\varepsilon^2$, $\varepsilon = RTln\left(1+\frac{1}{C}\right)$, $E = \frac{1}{\sqrt{2K_{DR}}}$	$q_e = q_m\ exp\ (-K_{DR}\varepsilon^2)$	lnq_e vs ε^2	slope=$-K_{DR}$, Intercept= lnq_m
Timken	$q_e = B_t\ ln\ ln\ A_t + B_t\ lnC_e$	$q_e = \frac{RT}{b}\ ln\ ln\ (A\ C_e)$	q_e vs lnC_e	slope= B_t, Intercept = B_t $ln\ ln\ A_t$
Three Parameter Models				
Model	**Linear eq.**	**Non-linear eq.**	**Plot**	**Slope and intercept**
Redlich-Peterson	$ln\ ln\left\{K\frac{C_e}{q_e} - 1\right\} = blnC_e + lna$	$q_e = \frac{KC}{1 + aC_e^b}$	$ln\ ln\left\{K\frac{C_e}{q_e} - 1\right\}$ vs lnC_e	slope= b, Intercept= lna
Sips	$ln\left[\frac{q_e}{q_m + q_e}\right] = \frac{1}{n}\ lnC_e + ln\ (b)^{\frac{1}{n}}$	$q_e = \frac{q_m\ (C_e b)^{\frac{1}{n}}}{1 + (C_e b)^{\frac{1}{n}}}$	$ln\left[\frac{q_e}{q_m + q_e}\right]$vs lnC_e	slope=1/n, Intercept= $ln\ (b)^{\frac{1}{n}}$
Toth	$ln\ ln\ \frac{q_e^n}{q_m^n - q_e^n} = n\ ln\ ln\ C_e + n\ ln\ ln\ K_t$	$q_e = \frac{q_m K_t C_e}{[1 + (K_t C_e)^n]^{\frac{1}{n}}}$	$ln\ ln\ \frac{q_e^n}{q_m^n - q_e^n}$ vs $ln\ ln\ C_e$	slope= n, Intercept=$n\ ln\ ln\ K_t$

Table 3. Parameters of linear and nonlinear isotherm models for the adsorption of Cr(VI) on MPGP at 20 °C.

Two Parameters Isotherms										
Isotherm	**The parameters for the Linear equation**				**The parameters for nonlinear eq.**					
Freundlich	K_F (mg/g)(L/mg $)^{1/n}$	n		R^2	K_F (mg/g)(L/mg $)^{1/n}$	n		R^2	SNE	Error functions
	1.515	1.506		0.974	1.6454	1.5942		0.964	3.6174	HYBRD
Langmuir	K_L (L/mg^{-1})	q_{max} (mg/g)		R^2	K_L (L/mg^{-1})	q_{max} (mg/g)		R^2	SNE	Error functions
	0.054	21		0.997	0.066	18		0.991	3.7322	HYBRD
	K_{DR} (mol/J)^2	q_{max} (mg/g)	E (J/mol)	R^2	K_{DR} (mol/J)^2	q_{max} (mg/g)	E (mol/J)	R^2	SNE	Error functions

Dubinin-Radushkevich	2.87E-06	9	4.18E+02	0.95	2.74E-06	9	4.27E+02	0.918	4.2377	HYBRD,MPSD, ARE
Temkin	b_t (J/mol)	A_t (L/mg)	B_t	R^2	b_t (J/mol)	A_t (L/mg)	B_t	R^2	SNE	Error functions
	606	0.648	4.021	0.996	608	0.6508	4.004	0.995	4.4614	MPSD

Three Parameters Isotherms

Isotherm	**The parameters for the Linear equation**				**The parameters for nonlinear eq.**					
Redlich-peterson	K(L/mg)	a (L/mg) b	b	R^2	K(L/mg)	a (L/mg) b	b	R^2	SNE	Error functions
	36.9569	23.417	0.352	0.908	0.98	5.8 E-03	1.69	1	3.8289	HYBRD
Sips	qmax (mg/g)	b(L/mg)	n	R2	qmax(mg/g)	b(L/mg)	n	R^2	SNE	Error functions
	2.30E+09	4.83E-14	1.45	0.974	14	1.18 E-01	0.77	0.998	3.6176	MPSD
Toth	q_{max}(mg/g)	K_T(L/mg)	n	R^2	q_{max}(mg/g)	K_T(L/mg)	n	R^2	SNE	Error functions
	11	0.058	2.52	1	12	0.0826	2.17	0.999	3.9667	HYBRD

RESULTS AND DISCUSSION

In this work two types of Isotherm models were examined; two and three-parameter models. Table 3 shows the result obtained from the linear and non-linear methods for different adsorption isotherms.

Two Parameters Model:

Freundlich Isotherm

Figures S1 and S2 in supplementary data and Tables (3 and 1) show the results of linear and nonlinear Freundlich isotherm for adsorption of Cr(VI) at 20 ℃. Investigation of these results leads to the linear regression of Freundlich isotherm having high R^2 values (approach to 1.0), so it is suitable for application on this adsorption system. The values of Freundlich Isotherm's parameters (K_f, n) for linear and non-linear at 20 ℃ are shown in Table 3. The results appear that these parameters are consistent and comparable to each other for linear and non-linear regression. Despite the MPSD is not the best according to the sum of the normalized error (SNE), the MPSD parameters are closer to that for linear regression at 20 °C; see Table 1; The minimum value for the Sum of normalized errors (SNE) is determined using HYBRD error function.

Langmuir Isotherm

The values of Langmuir Isotherm's parameters (K_L, q_{max}) for linear and non-linear at 20 ℃ are shown in Table 3. The results show these parameters are remarkably consistent and comparable to each other for linear and non-linear regression. For the linear form of Langmuir isotherm, we plot ($1/q_e$) against ($1/C_e$), these plots are illustrated in supplementary Figures (S3, S4). The results show high values of R^2 for ascorbate systems under study, these results indicate that the Langmuir isotherm is a good model for our data. The five error functions for the Langmuir model and its parameters are illustrated in Table 1. From this table, we find that the parameters (K_L, q_{max}) are consistent with that for linear regression and to each other for different error functions.

Dubinin-Radushkevich isotherm

Supplementary Figures S5, S6 and Tables (3, S2) show the results of linear and non-linear Dubinin-Radushkevich isotherm and Error functions values for adsorption of Cr(VI) at 20 ℃. Investigation of these results leads to the linear regression of Dubinin-Radushkevich isotherm having the least R^2 values, and high error functions values so it is unsuitable for application on this adsorption system.

Temkin Isotherm

For the linear form of Temkin isotherm, we plot (q_e) against (ln C_e), these plots are illustrated in Figure S8, while the Figure S7 represents the nonlinear regression based on the SNE method. The values of Temkin Isotherm's parameters (A_t,

B_t, b_t) for non-linear regression; which is adopted according to the SNE method; and linear are shown in Table 3. The results show these parameters are so close and comparable to each other for linear and non-linear regression. The results revealed high values of R^2 for ascorbate systems under study, these results indicate that the Temkin isotherm is a good model for the data. The five error functions for the non-linear Temkin model and its parameters are illustrated in Table S3. From this table we find that the parameters (A_t, B_t, b_t) are consistent with that for linear regression and to each other for different error functions.

Three Parameter Model

Figures 2-5 represent a linear and non-linear plot of linearized and non-linearized three isotherm models Redlich-Peterson, Sips, and Toth for adsorption of Cr(VI) onto MPGP at 20˚C using trial and error in the solver add-in with Microsoft's spreadsheet, Microsoft Excel.

The Redlich-Peterson Isotherm

For a linear form of Redlich-Peterson isotherm, we plot $\ln(K\ (C_e\backslash q_e)\ -1)$ against ($\ln C_e$), this plot was illustrated in Figure S10, while Figure S9 represents the nonlinear regression based on the SNE method. The values of Redlich-Peterson Isotherm's parameters (K, a, b) for non-linear regression; which is adopted according to the SNE method; and linear at 20 ºC are shown in Table 3. The results show these parameters are so different from each other for linear and non-linear regression moreover, the linear data get low values for R^2 Which indicate the linear form of Redlich-Peterson is unsuitable for application to this adsorption system and the error does not obey the Gaussian distribution [7]. On the contrary, the non-linear form gives high values of R^2(approach to 1.0) at 20 ℃, this indicates that the non-linear form is a good fit for our system. The five error functions for the non-linear Redlich-Peterson model and its parameters are illustrated in Table S4. From this table we find, for non-linear, the parameters (K, a, b) are consistent with each other for different error functions.

Sips Isotherm

For the linear form of Sips isotherm, we plot $\ln(q_e\backslash q_m+q_e)$ against ($\ln C_e$), these plots are illustrated in Figure S12, while Figure S11 represents the nonlinear regression based on the SNE method. The values of Sips Isotherm's parameters (q_{max}, n, b) for non-linear regression that are adopted according to the SNE method and linear at 20 ℃ are shown in table S3. The results show these parameters are so different from each other for linear and non-linear regression. Despite this, the linear form of Sips isotherm showed high values for R^2, but it is unsuitable for applying to this adsorption system because the parameters' values (q_{max}, n, b) are so high and q_{max} far away from that experimentally obtained. On the contrary, the non-linear form gives high values of R^2 (approach to 1.0) and reasonable parameters' values (q_{max}, n, b), this indicates that the non-linear form is a good fit for our system. The five error functions for the non-linear Sips model and its parameters are illustrated in table S5. From this table we find, for non-linear, the parameters (q_{max}, n, b) are consistent with each other for different error functions.

Toth Isotherm

For the linear form of the Toth isotherm, we plot $\ln(K\ (C_e\backslash q_e)\ -1)$ against ($\ln C_e$), these plots are illustrated in Figure S14, while Figure S13, represents the nonlinear regression based on the SNE method. The values of Toth Isotherm's parameters (q_{max}, n, K_T) for non-linear regression that is adopted according to the SNE method and linear at 20 ºC are shown in Table 3. The parameters values for linear and non-linear are acceptable and consistent with each other, in addition, they have high values for R^2 Which indicate the linear and non-linear form of Toth is suitable for application in this adsorption system. The five error functions for the non-linear Toth model and its parameters are illustrated in table S6. From this table we find, for non-linear, the parameters (q_{max}, n, K_T) are consistent with each other for different error functions.

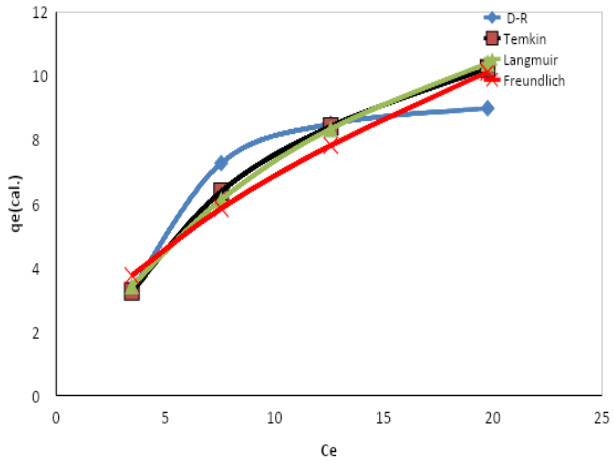

Figure 1. Non-linearized two parameter models for the adsorption of Cr(VI) on MPGP at 20 °C.

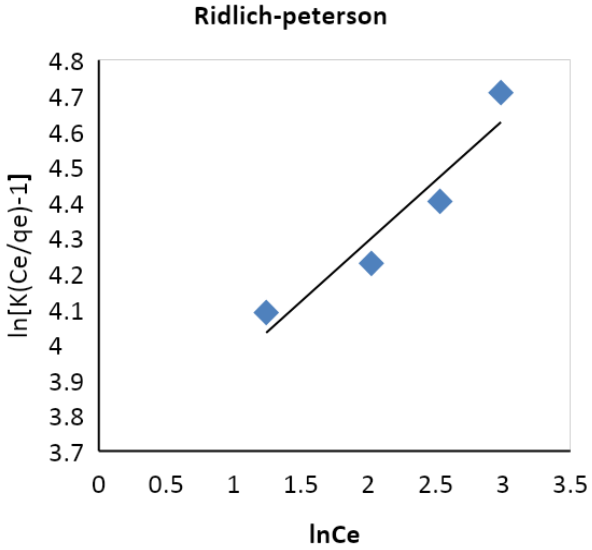

Figure 2. Linear Ridlich Peterson models for the adsorption of Cr(VI) on MPGP at 20 ℃.

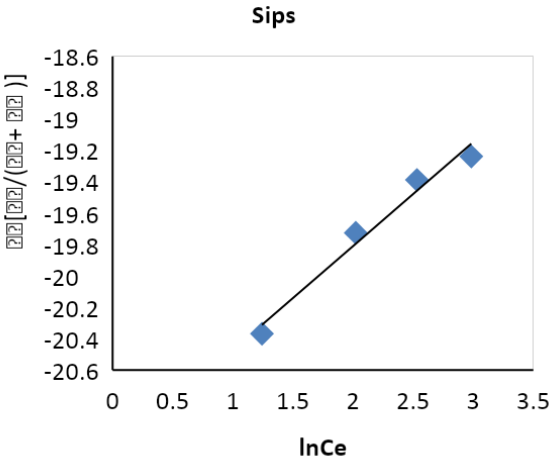

Figure 3. Linear Sips models for the adsorption of Cr(VI) on MPGP at 20 ℃.

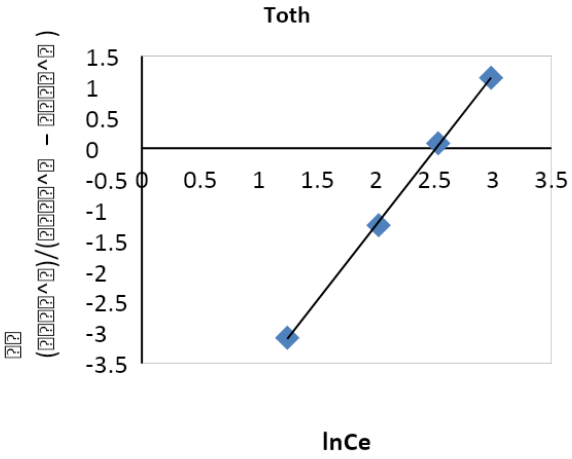

Figure 4. Linear Toth models for the adsorption of Cr(VI) on MPGP at 20 ℃.

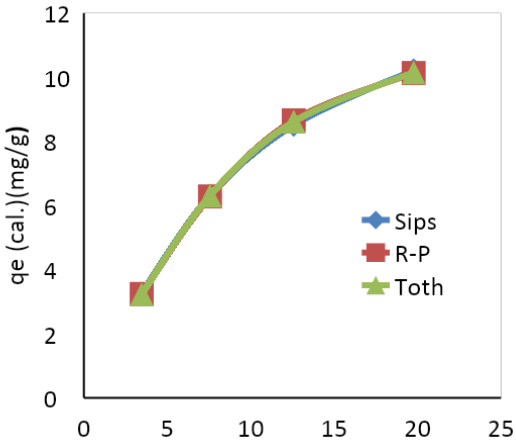

Figure 5. Non-linearized three parameters models for the adsorption of Cr(VI) on MPGP at 20 ℃.

Error Function

The result in table 3 signed HYBRD>MPSD= EABS>ARE>ERRS Has the best error functions with the respective lowest sum of normalized error (SNE) for non-linear -two and three parameters-isotherm models.

CONCLUSIONS

Four expressions of two-parameter isotherm models were used, Freundlich, Langmuir, Dubinin-Radushkevich, and Timken. Despite the linear regression of these isotherm models being investigated in previous work [10], the parameter values of these linear models were recalculated and certain values were corrected as necessary. Table 3 shows all those values and the corrected values are remarked by underlining. In non-linear regression of the two-parameter models, we chose the best fitting to depend on the sum of normalized error

(SNE) with minimum value. The established data from the linear and non-linear methods are comparable to each other which indicates the efficiency of the non-linear regression method [13][15]. The results reveal the best fitting isotherm is Freundlich ≈ Langmuir ≈ Temken with a high R^2 value. The D-R model was the least fitted to describe the adsorption, which means the unsuitability characteristics. The Tempkin constant, BT, increases with temperature rise, emphasizing that the adsorption of Cr(VI) onto MPGP is an endothermic process. There are differences between the values of parameters for linearized and non-linearized three-parameter isotherm equations (Redlich-Peterson, Sips, and Toth). Depending on the R^2 value, the non-linear three isotherms models are very good fitting to the adsorption system under study. The results appear to have different values for calculated parameters between the linear and non-linear equations of Redlich-Peterson and Sips isotherm, the results led to an improvement in the main drawback of the linear isotherm equation for these models. Specifically, the findings suggested that the error did not follow a Gaussian distribution.

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Disclosure and Conflict of Interest: The authors declare that they have no conflicts of interest.

Data Availability

The figures and data used to support the findings of this study are included within separated supplementary data file, other data are available on request.

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