

Organic pollutants removal from oilfield produced water using nano magnetite as adsorbent

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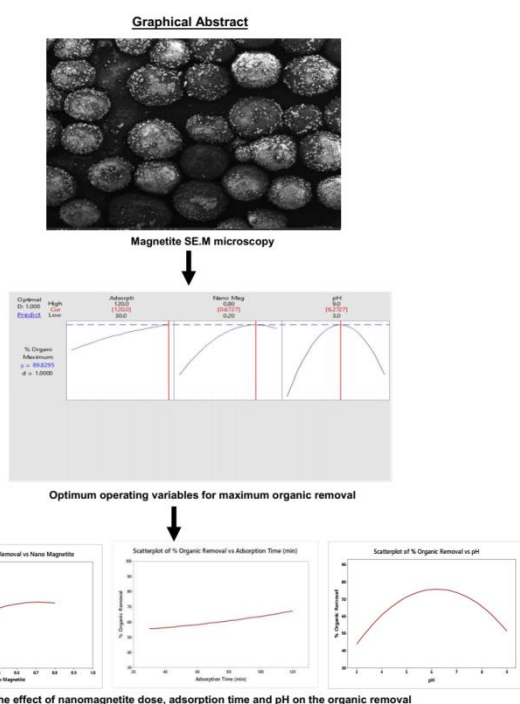
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Graphical abstract



Abstract

The current study investigates the ability of Nano magnetite as an adsorbent to remove organic materials from oilfield-produced water (PW). The effect of several variables on the removal procedure was investigated, such as nano magnetite dose, pH of solution and adsorption period. The adsorbent was carefully examined and completed using several approaches FTIR, SEM and surface area analyzer. The results show that the best organic removal reached more than 89% at 0.7 g Nano magnetite dose in 120 min through pH = 6.0 at room temperature. Three mathematical isotherm models of adsorption were used in this study: Langmuir, Freundlich and Brunauer-Emmett-Teller. It seems that the Langmuir model and Brunauer-Emmett-Teller was best fitted model over the experimental variety with a correlation coefficient of 0.994 and 0.995 respectively.

Keywords: Produced Water; adsorbents; nano magnetite; dose; RSM; isotherms.

1. Introduction

For many countries, oil is the primary source of wealth and energy (Igunnu and Chen, 2012). The oil and gas manufacturing regular growths include a vast quantity of injected water toward the oil recovery that remains active toward the surface lengthways with organic compounds, salt, and extra solutes and remains frequently known by way of “produced water” (Rashid *et al.*, 2020). Oil trades produced wastewater is real multifaceted, and comprises many organic and inorganic substances, such as free and dissolved oil, phenol and heavy metal through products (AlJaberi *et al.*, 2020). For the reason that the wastes of oilfields cause ecological contaminations, treatment remains vital beforehand elimination. The action of these wastes will maybe consequence in improved oil retrieval, oil/water separation, recycling of water, defense of downstream facilities, and finally follows health and environmental safety guidelines (Hassan and Mousa Al-zobai, 2019). The treatment of wastewater goals remains toward recover the organic content and edge the previous waste aimed at recycling or reject. The wastewaters are gutted through numerous physical and chemical incomes beforehand the release and rules place severe limits on stages of contaminants which can remain cleared to the sea (Ibrahim *et al.*, 2019). The environment needs the development and implementation of appropriate action services capable of eliminating dangerous toxins from many of these industrial waters. Due to the high level of environmental attention paid to these watercourses, they remain difficult to treat (Ali *et al.*, 2016) and (Alakoul *et al.*, 2021). Organic pollutants and their derivatives have a dangerous effect on human life for a long time, so it needs special processing not depends on traditional methods only (Ali *et al.*, 2018). Numerous modern treatments has been applied to remove the organic content from produced water : Advanced oxidation processes (Naeem *et al.*, 2018) and (Al-zobai *et al.*, 2020), coagulation -adsorption (Rosenblum *et al.*, 2016), Electro-oxidation (Ahmed *et al.*, 2021; Alturki *et al.*, 2021), Electrocoagulation (Manilal *et al.*, 2020) and separation of a membrane (Zsirai *et al.*, 2018). Amongst these approaches, the treatment of adsorption gained a high effect of the most attractive in terms of financial and ecological friendly (Ibrahim *et al.*,

2010). Nevertheless, whenever the wastewater watercourses are sufficient, such treatments request great costs. Adsorption remains one of the organic elimination approaches that had learned cumulative thought through the asset of its possible efficiency and its extra advantages similar being refillable, high selectivity at molecular level, cheap by way of per energy demands, flexibility in dealing with numerous substances, protection towards poisonous constituents and comfort of handling (Naeem and Hassan, 2018). Adsorption keeps its place generally by considering it a way of one of the most extensive approaches aimed at eliminating pollutants from wastewater solutions. Profitable activated carbon has been deliberate by way of an adsorbent for the elimination of contaminants from waters for several years because of the great exact surface area and pore structure. To overwhelm this drawback, substantial care has remained given toward make activated carbons resulting from self obtainable and squat cost waste materials (Depci *et al.*, 2012). The good-looking property of iron oxide nanoparticles authorizes informal disseverment of adsorbents from the outline and might remain recycled for the additional request (Namdeo, 2018). Furthermore, as a cost-effective and environmentally friendly material, magnetite has high adsorption capabilities for a wide range of species, including dissolved metals, particulate matter, organic and biological components. (Petrova *et al.*, 2011). The purpose of this work is to select the perfect PW activity by utilizing the nano magnetite as an adsorbent. The Nanomagnetite dosage in addition to the pH is principal effects to affect the general action capability and these were thoughtful in this work and study the kinetic model for adsorption processes.

2. Materials and methods

2.1. Produced water

Samples of produced water (PW) were collected as from Al-Ahdab oil field at Wassit governorate, Iraq. The properties of wastewater and other constituent's in this PW was registered in Tables 1 and 2 respectively:

Table 1. Properties of wastewater

Property	Concentration
Organic content	103.5 ppm
pH	6.77
TDS	137800 ppm
Conductivity	170.5 ms cm ⁻¹
Turbidity	84 NTU
TSS	20.25 ppm

Table 2. Constituents of produced water

Constituent	Concentration (ppm)
Calcium	7835
Magnesium	2335
HCO ₃	258
Dissolved oxygen content	0.063
Manganese	3

2.2. Nano magnetite characterization

A type of microscopy that uses electrons to (SEM) inspection was attained to examine the adsorbent's

morphology. In this research, JSM-6100 was used to analyze the nanomagnetite before adsorption. (FT-IR) utilized for identifying surface useful collections of FT-IR spectroscope (Bruker), wherever a range from 4000 to 500 cm⁻¹.

2.3. Materials

Nano Magnetite adsorbent made of Merck, Sodium chloride, CCL₄, hydrochloric acid in addition Sodium hydroxide both made of Merck-India, distilled water, adjust the pH of the solution with HCL 1N or NaOH 1N.

2.4. Adsorption procedure

All procedures of adsorption experiments were achieved at the volume of nano magnetite with 150 milliliter PW in a 250-milliliter glass bottle. The adsorptions speed at a value of two hundred revolutions per minute through shaking device at room temperature with pH (3-9). The significance of adsorbent amount from 0.2 to 0.8 g of nano magnetite specific amount of nano magnetite with 103.5 ppm produced water. The pH ranged from three to nine, and the adsorption time interval from 30 until 120 minutes was controlled by the magnetite particles and dosage. The mass balance for the following equation was being used to evaluate the amount of PW at equilibrium. (Gupta, 2011):

$$q_e = V(C^o - C_e)/W \quad (1)$$

Wherever q_e (mg g⁻¹): represents the organic content in PW per unit of adsorbent mass at known volume, time and magnetite mass, W (g): acts the magnetite weight, while symbols C^o , C_e (ppm): exemplify initial concentration, final concentration of produced water at time t respectively. The organic removal efficiency by nano magnetite was calculated by the following equation:

$$\text{Adsorption(\%)} = (C^o - C_e/C^o) \quad (2)$$

2.5. Experimental design

PW mineralization by the nano magnetite execution is better by using the technique of Central -Composite - Design (CCD) under Response Surface Methodology as software of experimental design, data analysis and scheme charting. The factors, such as time adsorption (X_1), the dosage of magnetite (X_2) and pH (X_3), were coded in the CCD with their different grades, such as time adsorption (X_1) from half-hour to two hours, a dosage of magnetite (X_2) from 0.2 until 0.8 and pH (X_3) from 3 to 9.

2.6. Organic concentration determination by UV-spectrophotometer analysis

0.2 gm of sodium chloride was added to fifty mL PW by using a funnel, 5 ml of CCL₄ was added and shaking for 2 minutes. After 25 min., the wastewater was separated into two special layers, the lower organic layer especially used for the absorbance measurement, and organic content was computed using the calibration curve. The turbidity was monitored and read using a turbid meter (Lovi-bond, SN 10/1471) (Ernestina Elizabeth Banda-Cruz *et al.*, 2016).

3. Results and discussions

3.1. Description of biosorbent

3.1.1. FT-IR Examination

FT-IR spectra at a range of (4000–500) cm^{-1} for nano magnetite were exposed in Figure 1. FTIR test documented the being harmfully charged functional hydroxyl groups, carboxyl group, in addition amine group at the nano magnetite's surface. Because of hydroxyl group expansion or amine (-NH_2) bending of polymeric compounds, the entire band at 3317.67 powder stays attributed. The strength of the (-CH_2) corresponding spreading vibrations remains in the range shown in Figure 1.

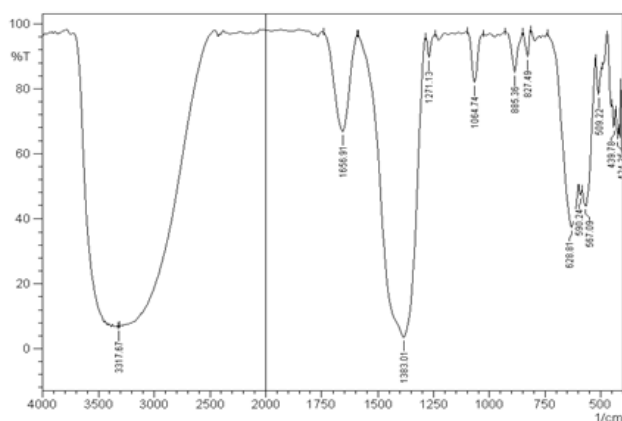


Figure 1. FT-IR testing of Nanomagnetite.

3.1.2. SEM examination/specific surface area

Entire surface and shape of the adsorbent are explained by the SEM image. The surface morphology of nano magnetite was properly produced into a filamentous pattern Figure 2. It has a porous and irregular construction with a vast number of pores that could still operate as adsorbing zones, making it significant. This property distinguishes **Table 3**. Careful variables significances

nano magnetite's surface as a good adsorbent for removing organic content. The nano magnetite has a surface area of about $98.11 \text{ m}^2 \text{ g}^{-1}$.

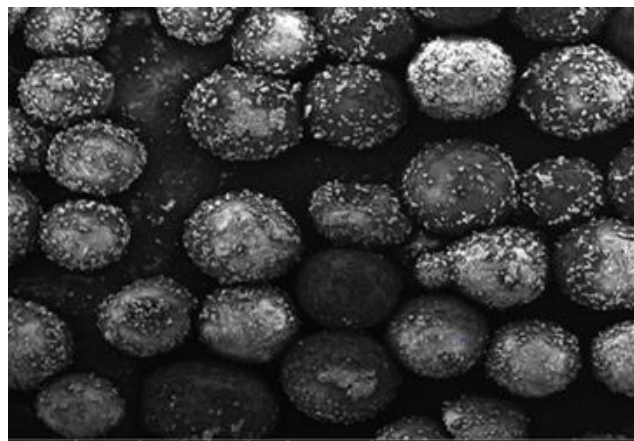


Figure 2. Magnetite SEM microscopy.

3.2. Regression models

The responses and independent variables might be studied at least-squares technique by a 2nd. order model by (Davarnejad *et al.*, 2014):

$$Y = B_0 + \sum_{i=1}^q B_i X_i + \sum_{i=1}^q B_{ii} X_i^2 + \sum_{i,j} B_{ij} X_i X_j + \varepsilon \quad (3)$$

Wherever a symbol y acts the elimination responses, while X_1, X_2, \dots, X_q symbolize the employed variables. The constant of regression is referred to as B_0 , while the linear regression constant is referred to as B_i . However, B_{ii} acts the squared regression constant and B_{ij} acts the cross-product reversion constant, so the error is symbolized by a symbol ε . Table 3 displays the result of the variables used in the study based on CCD design, as well as the percentage of organic elimination in PW.

Run	StdOrder	Run Order	Pt Type	Blocks	X_1 :Adsorption time (min.)	X_2 :Nanomagnetite dose (g)	X_3 : pH	Organic removal%
1	1	1	2	1	30	0.2	6	52.36
2	2	2	2	1	120	0.2	6	63.45
3	3	3	2	1	30	0.8	6	77.96
4	4	4	2	1	120	0.8	6	89.66
5	5	5	2	1	30	0.5	3	40.32
6	6	6	2	1	120	0.5	3	51.32
7	7	7	2	1	30	0.5	9	51.36
8	8	8	2	1	120	0.5	9	64.58
9	9	9	2	1	75	0.2	3	35.21
10	10	10	2	1	75	0.8	3	48.96
11	11	11	2	1	75	0.2	9	34.66
12	12	12	2	1	75	0.8	9	55.36
13	13	13	2	1	75	0.5	6	82.31
14	14	14	2	1	75	0.5	6	81.99
15	15	15	2	1	75	0.5	6	82.05

The mathematical formula (Equation 4) explain terms of actual factors as a real values connecting the organic

elimination response to the operating variables reveals the linkages between these variables, based on untried consequences:

$$\begin{aligned} \text{Organic Elimination} = & -91.5+0.208X_1+132.4X_2 \\ & +38.34X_3-0.00072X_1^2-108.9X_2^2-3.196X_3^2 \\ & +0.011X_1X_2+0.0041X_1X_3+1.93X_2X_3 \end{aligned} \quad (4)$$

3.3. Nano magnetite solution effect

The organic content adsorption on Nano magnetite as an adsorbent quantity function was showed in Figure 3. The removal of organic in PW was increased with the cumulative quantity of Nano Magnetite under constant conditions reached 72.3%. As clearly seen in Figure 3, increasing the dose of nanomagnetite has a positive effect by increasing the organic removal from PW.

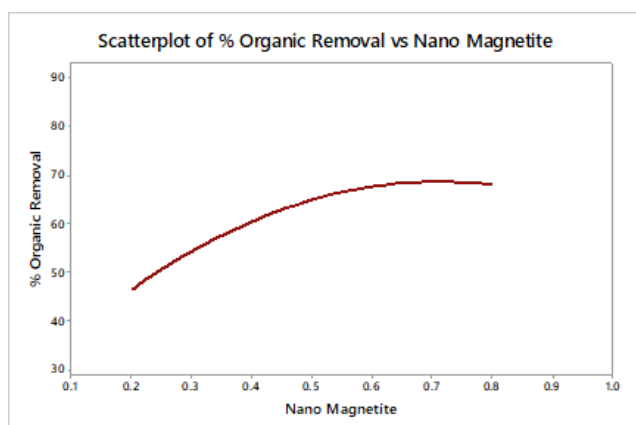


Figure 3. The effect of Nano Magnetite on the organic removal (pH =6, time 120 min).

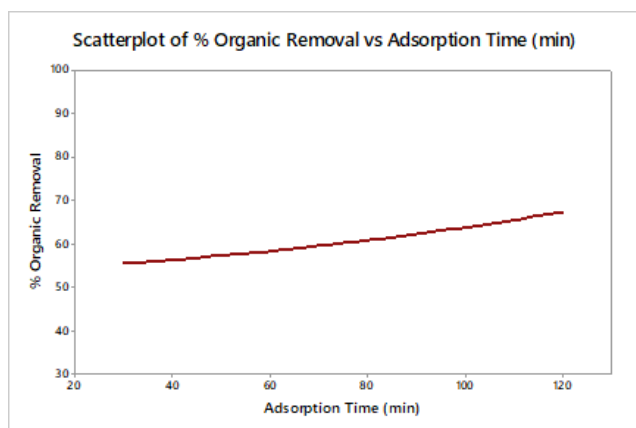


Figure 4. The effect of adsorption time on the organic removal (Nano magnetite dose = 0.7 g, pH = 6).

3.4. Adsorption time effect

To develop adsorption studies, it is important to investigate the effect of adsorption duration upon on adsorption treatment. (Jafer *et al.*, 2019). The trials are organic that adsorption time differs from the first thirty minutes to the end of 120 minutes at a various amount Nanomagnetite dose between (0.2) and (0.8) g. Adsorption treatments persist, so the adsorption equilibrium can be achieved within 90 minutes, as shown in Figure 4. Extra increases in the adsorption period do not recover the adsorption

activity. The singularity might remain temporarily, first, all vigorous positions above the surface of the adsorbent remain free and the concentration of organic remains rising. Later that duration, insufficient surface vigorous locations remain available, so only a sharp low increase in organic uptake remains experiential (Hassan *et al.*, 2020). Consequently, the finest adsorption time for organic content in produced water was specified of two hours preparing for all the cases.

3.5. pH solution effect

The pH of the PW in which the adsorption process was a significant limit in the treatment. The degree of electrostatic customers associated with ionized contaminants and the functional groups above the surface of nanomagnetite stay chiefly an important role of final pH (Ofomaja *et al.*, 2009). For pH interval 3–9, sorption of both divalent organic content was considerably little, while the initial value of pH increased, organic content uptake increased as shown in Figure 5. It is clear that the best organic removal happened at the interval pH between (5) and (7). To avoid probable hydroxide deposition, high levels of (pH) are being used. (El-Shafey, 2010). These points can be further clarified by the fact that the surface charge of the biomass stays positive at lower levels of pH, which is unfavorable for cationic bio_sorption. Furthermore, at the active sites, hydrogen ions interact intensely with organic content, as well as the conflicting effect of hydrogen ions, decreases as pH rises (Feng *et al.*, 2009).

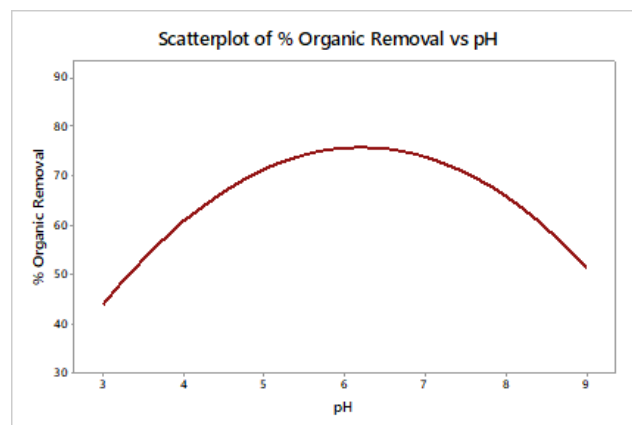


Figure 5. The pH effect on the organic removal (Nano magnetite dose = 0.7, time = 120 min).

3.6. Enhancing the employed variables

From the previous studies, it is obvious that can explore the optimum conditions by selecting the minimum and maximum values. Minitab-17 was also used to obtain premium values for the variables used, such as adsorption period, Nanomagnetite dosage, and pH. The quantitative consequences of the D-optimization are labeled in Figure 6. The highest organic content elimination abilities were greater than 89 percent.

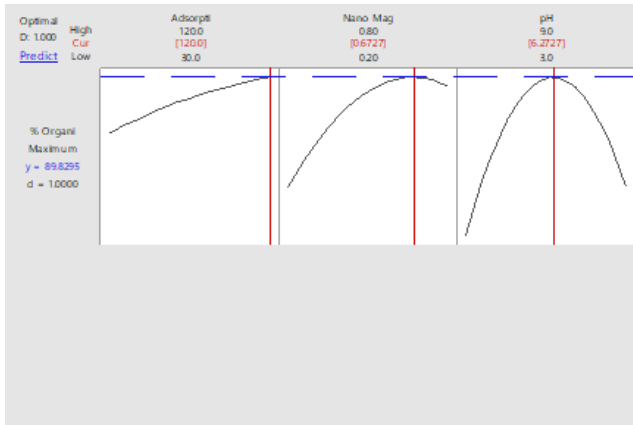


Figure 6. Optimum operating variables for maximum organic removal.

Two confirmation experiments has been conducted at the optimum operating conditions as shown in Table 4:

Table 4. Confirmation experiments

Run	Time	pH	Nano magnetite	Removal%
1	120	6	0.7	86
2	120	6	0.7	92

3.7. Adsorption Isotherms

As a result of the adsorption process, there are some implications that were agreed to equations of Langmuir, Freundlich and Brunauer-Emmett-Teller (BET) (Figures 7–9) contrasting mathematical isotherm equations for Nanomagnetite adsorbent. Isothermal limitations were computed from the modification of each adsorption isotherm in order to choose the best model shadowed by untested bio-sorption test results. (Dakhil, 2020) and (Naser *et al.*, 2021). The working conditions must be carefully considered for the contrast of the data, as the setup isothermal experimental effects are dependent on them. (Rangabhashiyam and Selvaraju, 2015). The Langmuir model and BET appear to best fit the experimental results over the experimental range with reasonable correlation values ($R^2 > 0.99$). This attitude is similar to the interpretations given in the (Pereira *et al.*, 2010; Mukhtar *et al.*, 2020; Dakhil and Ali, 2021). The equations of Langmuir, Freundlich and Brunauer-Emmett-Teller can be written respectively as:

$$1/q_e = 1/q_m + (1/K_L q_m)(1/C_e) \tag{5}$$

Where: (q_e) acts adsorption capacity ($mg\ g^{-1}$), (C_e) indicates to the equilibrium concentration ($mg\ L^{-1}$), (K_L) acts Langmuir constant ($L\ mg^{-1}$), so (q_m) acts the maximum capacity ($mg\ g^{-1}$) (Xu *et al.*, 2013).

$$\log(q_e) = \log K_f + (1/n)\log(C_e) \tag{6}$$

Where K_f : acts the Freundlich constant isotherm model and n : represents linearity adsorption constant (Ramachandran *et al.*, 2011).

$$C_e/(C^o - C_e)q_m = [(K_B-1)/K_B q_m] [C_e/C^o]+1/K_B q_m \tag{7}$$

Where q_m : symbolize maximum ability ($mg\ g^{-1}$), C_o : acts the adsorbate initial concentration ($mg\ L^{-1}$) and K_B : acts the BET constant isotherm model (Mousa and Taha, 2015).

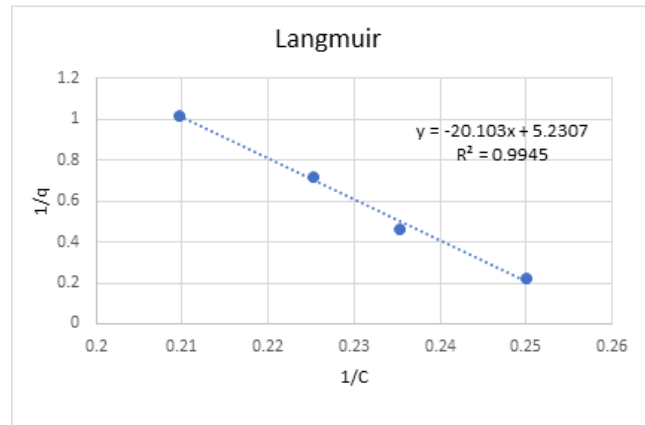


Figure 7. Langmuir isotherm plot for adsorption of organic content on PW.

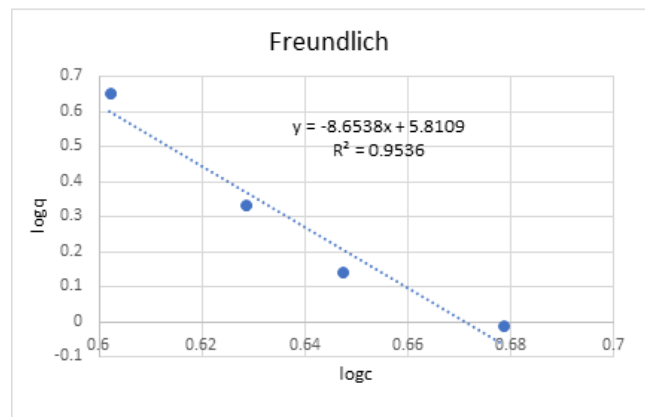


Figure 8. Freundlich isotherm plot for adsorption of organic content on PW.

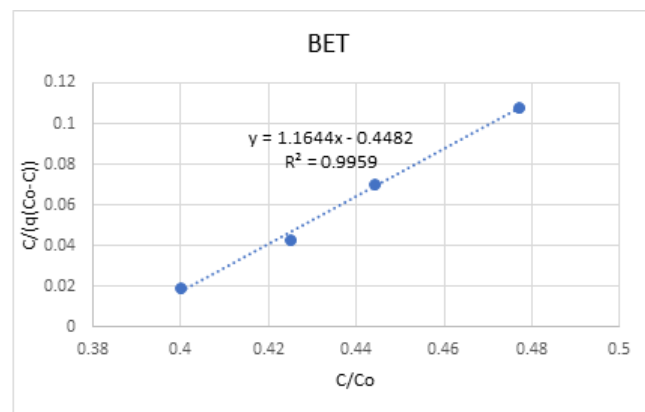


Figure 9. BET isotherm plot for adsorption of organic content on PW.

4. Conclusions

The ability of Nano magnetite as an active and low-cost bio sorbent to remove organic materials from PW was discussed in this study. Adsorption limits computed from the Langmuir, Freundlich, and BET isotherms are useful for understanding the mechanisms of the adsorption process and the equilibrium results were found to fit with the Langmuir and BET model. Response surface methodology (RSM) was used to construct the tests, and the data were then regression investigated and modified to find the best

operating parameter. It was discovered that the maximum removal efficiency more than 89% at pH = 6.0 and 0.7 g of Nanomagnetite dose in two hours .

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