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Optimizing Operational Factors for Maximizing Phenolic Compounds Adsorption under Continuous Flow Conditions using Central Composite Design and Response Surface Methodology

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Abstract

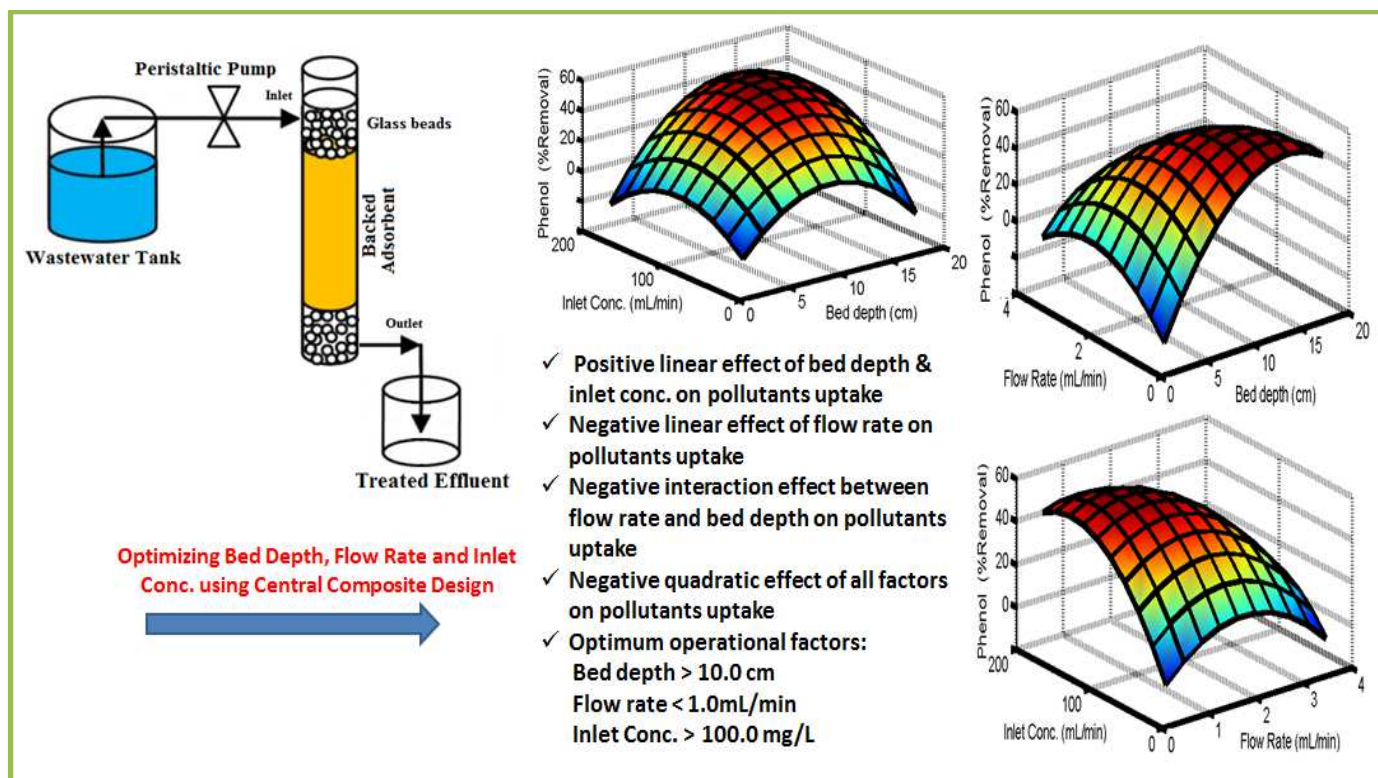
The factors controlling adsorption of phenol and 2-Nitrophenol under continuous-flow conditions using compost were optimized using central-composite-design and response-surface-methodology CCD-RSM. Compost-based adsorbent (C/N 21.0) manifested high affinity for capturing toxic phenols. Using CCD-RSM, adsorption process of phenols was presented in a second-order mathematical equation that accounts for the linear, interaction, and quadratic effects of bed depth, flow rate, and inlet concentration on the process. For linear effect, analysis indicated that increasing bed depth, increasing inlet concentration, and reducing flow rate had maximized pollutants uptake under flow conditions. For interaction effect, analysis indicated that bed depth and flow rate counteract each other's effect for pollutant uptake; that is, deeper bed depth and slower flow rate can maximize the uptake process. Examined factors have a significant negative quadratic effect on pollutant uptake, and this indicates that increasing factors increases pollutant uptake up to a point, then declines. Contour plots revealed that uptake process under flow conditions is maximized at a bed depth >10.0 cm, a flow rate <2.0 mL/min, and an inlet concentration >100.0 mg/L. Breakthrough curves were conducted at the optimum operational factors. Compost was effective under flow conditions with bed volumes 98.4 and 119.4 for phenol and 2-Nitrophenol, respectively. Maximum bed capacities were 29.5 and 43.4 mg/g and reported at bed depth 15-cm, flow rate 1.0-mL/min, and inlet concentration 150.0-mg/L for phenol and 2-Nitrophenol, respectively. The prepared compost was reusable under flow conditions for removing phenol and 2-Nitrophenol up to four cycles with a final efficiency of 70-65%.

Highlights

- Mature and stable compost was derived from organic solid wastes.
- Operational factors were optimized for maximizing uptake of pollutants under flow conditions.
- 3D and 2D plots generated by CCD-RSM were useful to locate the optimum operational factors.
- The bioadsorbent was reusable up to four cycles under flow conditions.

Keywords Factors Optimization • Phenolic Wastewater • Compost • Fixed Bed Adsorption • Central Composite Design

Graphical Abstract



1 Introduction

Phenolic compounds are commonly used in the chemical, pharmaceutical, textile, and petrochemical industries, and they are toxic pollutants that frequently detected in the wastewater generated by the earlier activities [1]. Phenolic compounds can cause unpleasant odors and tastes even at a concentration down to 5.0 $\mu\text{g/L}$. In addition, phenolic compounds are highly toxic at levels higher than 2.0 mg/L [1]. Due to its high toxicity, environmental protection agencies have enlisted phenol as a priority pollutant, which led to the creation of firm concentration limits to ensure human health and guard the ecosystem's balance [2]. For example, the United States Environmental Protection Agency has established 1.0 $\mu\text{g/L}$ as the safe limit concentration of phenol in surface water [2]. In this frame, searching for effective treatment technologies has been extended. Conventional technologies, including chemical oxidation, biological degradation, and physical adsorption, have been tested for removing compounds from wastewater [3]. However, the earlier technologies suffer from limitations, including incomplete elimination of the pollutants, high running costs, and the production of toxic byproducts [3]. In this regard, biological treatment, while effective for some pollutants, is insufficient for removing phenolic compounds due to their toxicity to microbial populations [3]. On the other hand, chemical oxidation like advanced oxidation processes can degrade phenolic compounds but are energy-consuming and produce harmful byproducts [3]. Physical adsorption using activated carbons derived from diverse feedstocks was an

active treatment method for phenolic compounds; however, application of this technology is limited by the high regeneration and production costs [3]. To overcome the earlier challenges, there is urgent need to look for alternative adsorbents and technologies for removing phenolic compounds from wastewater. In this regard, many synthetic and non-conventional adsorbents exhibited a high adsorption of phenolic pollutants including zeolite [4], cocos nucifera shell-clay composite [5], metal-organic frameworks [5], zirconium-ferrite nanoparticles [7], silica/calcium alginate nanocomposite [8]. Recent studies indicated that composts, which derived from diverse organic wastes, were effective for removing pollutants from wastewater [9,10]. Although compost showed a high performance for removing heavy metals, organic dyes, and organic pollutants, its application for removing phenolic pollutants has not yet been reported [11]. The effectiveness of the adsorbent for eliminating phenolic compounds is often examined under batch conditions and very infrequently fixed bed conditions [12]. In fact, adsorption under batch conditions is limited to small-scale treatment units and less convenient for the industrial scale units. On the other hand, much attention has been given to adsorption under fixed-bed conditions for removing contaminants from wastewater. Compared to batch adsorption, adsorption under flow conditions is helpful due its scalability for industrial applications, and can treat large volumes of wastewater in a short time [13]. To assess the performance of an adsorbent under fixed-bed conditions, many operational factors, including bed depth and flow rate, should be optimized to maximize the uptake capacity. In many cases, univariate factorial design is adopted for factor optimization. In univariate design, the response (adsorption capacity) is measured while varying one variable (like pH) but keeping other variables (like mass and contact time) at fixed levels [8]. Recently, advanced designs of experiments were adopted in the adsorption field [12]. Following a suitable DoE, all factors (like mass, flow rate, inlet concentration) that affect the measured response (like adsorption capacity) were varied in a balanced way to determine the best combination of variables leading to maximizing the target response [14-16]. Among designs, central composite design, full factorial design, fractional factorial design, and Plackett-Burman were the most applied [16-18]. In fact, CCD-RSM is a powerful statistical methodology that often used to optimize adsorption processes [12]. With aid of CCD-RSM, the significant factors that affect adsorption process can be easily identified and optimized. Initially, CCD use to build a set of experiments with conditions. The effect of the examined variables on the target response is mathematically visualized using RSM [15,19].

In continuation of our research for developing new bioadsorbents, compost was derived from organic wastes and further tested for removing phenol and 2-Nitrophenol under fixed bed conditions. Prior to application as a bioadsorbent, the maturity, stability and bioavailability of heavy metals were assessed for prepared compost. The optimization of the experimental variables that

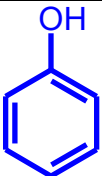
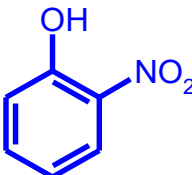
affect phenol and 2-Nitrophenol adsorption under flow conditions was performed using three-variable central composite design. The linear, interaction, and quadratic effects of the examined variables on pollutant uptake under flow conditions were analyzed using response surface methodology. Under the optimum combinations of flow rate, bed depth, and inlet concentration, uptake of pollutants using fixed-bed adsorption was examined. The overall performance of compost for removing pollutants under flow conditions was critically assessed by estimating bed capacity, bed volume, and adsorbent exhaustion rate. Recycling of the exhausted compost under flow conditions was also examined.

2 Methodology

2.1 Chemicals, solutions and instruments

Phenol and 2-Nitrophenol were purchased from SIGMA-ALDRICH® (USA) with a high purity (>99.9%). Other reagents were purchased from DBH® (UK). Doubly distilled water was used throughout the experimental work. Structural formulae of the phenolic compounds along with some important physiochemical parameters are presented in Table 1.

Table 1 The structural formulae with important physiochemical parameters of the examined pollutants ^a

IUPAC	Structural formula	Acidity constant pK_a	Solubility in water g/L (20 °C)	Partition coefficient Log P	$\lambda_{\max}$ (nm)
Phenol		9.89	93.0	1.48	270.0
2-Nitrophenol		7.17	2.0	1.79	345.0

^a Physiochemical parameters were collected from [20,21]

For each adsorbate, a stock solution of 1000 mg/L was prepared by dissolving 1.0 (± 0.001) g in 10.0 mL distilled water. The earlier solution was transferred to a 1000 mL volumetric flask and diluted with distilled water. All working solutions were prepared from the stock ones after appropriate dilution. The Thermo Scientific UV-Vis spectrometer was used to perform spectral analysis of phenolic compounds (Thermo Scientific, USA). A peristaltic pump (Smith and Nephew Watson-Marlow, England) was used to pump solutions through the particles of compost that were packed in a glass column.

2.2 Compost preparation and characterization

Composting is a promising procedure for converting organic wastes into useful growing medium and bioadsorbents. However, the proper selection and conditioning of feedstocks and proper composting are necessary to end up with a stable and mature product. In addition, physiochemical characterization of the produced compost is also essential for safe applications, especially as a bioadsorbent. In this work, vegetable scraps and fruit peels were chosen as nitrogen-rich ingredients, while dried leaves and straw were chosen as carbon-rich ingredients. Initially, the plants were cut into small pieces using an electric chopper. For effective composting, 2.0 parts of carbon-rich ingredients were mixed with 1.0 part of nitrogen-rich ingredients (by volume) in a five-liter composting bin. The generated pile was well mixed and aerated every week to speed up biological decomposition of organic wastes. During composting, the moisture content was maintained around 40-50%, and the temperature should not exceed 60-65°C. Mixing was terminated when a homogeneous product with a brownish color and a scent similar to soil was obtained. The composting process was lasted for three months, and the final weight of the compost was 500-550 gram. Before application as a biosorbent, compost was washed with distilled water, dried at 105.0°C for 24 hr, and sieved to a particle size less than 1.0 mm. Many physiochemical characterization tests were performed to check out the maturity of compost and suitability as bioadsorbent, including pH, electrical conductivity, C/N, bulk density, and FTIR. pH and electrical conductivity were measured by preparing a 1:5 *vol/vol* mixture of compost and distilled water. The mixture was agitated for 1.0 hr, and the particles of compost were removed by simple filtration. pH and conductance were measured for the clean filtrate using a calibrated pH meter and conductometer, respectively. Total organic carbon was estimated by heating a 2.0 g sample to 550°C for 3.0 hr. Bulk density of compost was measured using a pycnometer. Total nitrogen in compost was measured using the common Kjeldahl method. 10.0 grams of dried compost were extracted with 100.0 mL of 6% KCl for 2.0 hr. The mixture was filtered, and the content of NH_4^+ was measured colorimetrically after reaction with Nessler's reagent. Porosity was measured using water absorption methodology and at room temperature. Surface spectral analysis was carried out

with the aid of an FTIR spectrometer. Total content of toxic heavy metals in compost was measured as follows: 1.0 g of the dried sample was digested with 16.0 M HCl at 180°C for 1.0 hr and further extracted with 74% HClO₄ at 200°C for elution of heavy metals. The content of heavy metals in the clean filtrate was measured using atomic absorption spectrometry. The toxicity characteristics leaching procedure (TCLP) was used to assess the bioavailability of toxic metals in compost as follows: To a 10.0 g dried compost sample, 200 g of 0.1 M acetic acid at pH 2.88 was added, and the mixture was agitated for 18.0 hr. The content of heavy metals in the clean filtrate was measured using atomic absorption spectrometry. The main results of the characterization tests are provided in Table 2.

Table 2 Physiochemical characterization of prepared compost for safe application as bioadsorbent

Test		Value	Recommended or safe limit
pH ^a		7.13	6.0-8.5
Electrical conductivity ^a , dS/m		1.82	<3.0
Total organic matter ^b , %		86.2	>20
Bulk density ^c , kg/m ³		389.0	<400
NH ₄ ⁺ -N ^d , mg/kg		278.6	<500
C/N		21.0	>20
Porosity ^c , %		89.4	>85
Specific surface area ^e , m ² /g		28.4	> 1.0
Infrared spectroscopy, cm ⁻¹		1500–1600 -COOH, -RCON 2870 and 2930 - R-C-H 3200–3400 - COOH 3500 - NH ₂ 1780 -C=O	
Total Heavy Metal (mg/kg) ^f	Cu	167.4	230
	Zn	77.8	500
	Pb	123.7	140
	Ni	68.5	100
	Cd	10.5	1.5
	Hg	ND	1.5
	Cr	ND	0.5
TCLP (mg/L) ^g	Hg	ND	0.5
	Cd	0.33	1.0
	Cr	ND	5.0
	Pb	0.12	5.0
	As	ND	5.0

- pH was measured as outlined in the literature [22]
- Total organic matter was measured as outlined in the literature [23]
- Bulk density and porosity for compost were measured as outlined in the literature [24]
- NH₄⁺ content was measured as outlined in the literature [25]
- Surface area was measured as outlined in the literature [14]
- Total content of heavy metals were measured as outlined in the literature [26]
- TCLP test was carried out as outlined in the literature [27]

2.3 Continuous flow operation

Removal of phenolic compounds under continuous flow conditions was typically performed as follows: A glass column (1.0 cm diameter × 50 cm length) was loaded with a fixed amount of 4.0–10.0 g of compost at particle size 1.0 mm. To achieve a uniform inlet flow, a layer of glass beads (3.0 mm) was loaded on top of packed adsorbent. The column test was made by pumping the solution through the column in downward flow type with the aid of a peristaltic pump. Based on the preliminary screening tests, removal of phenol and 2-Nitrophenol was favorable at acidic medium and at room temperature. Accordingly, column tests were performed at pH 2.0 and 25.0 °C. The optimum operational factors including flow rate (1.0–3.0 mL/min), bed-depth (5.0–15.0 cm), and influent concentration (50–150 mg L⁻¹) on phenol and 2-Nitrophenol adsorption were ascertained using central composite design and response surface methodology as explained next.

2.4 Central composite design CCD and response surface methodology RSM

In fact, CCD-RSM is a statistical technique and a powerful optimization tool to optimize systems and processes. CCD is an experimental design that generates a set of tests to investigate the effect of multiple factors like flow rate, bed depth, and inlet concentration on a response variable like phenol adsorption. On the other hand, RSM is a statistical technique that is often used to analyze data from CCD tests and generate a mathematical model that describes the relationships among factors and response variables. In this regard, CCD-RSM can effectively reduce the number of experiments, provide better insights into the relationships among experimental factors and response variables, and optimize the adsorption process, which leads to improved performance and efficiency [28 (Brereton 2003)]. When applying CCD-RSM to model phenol uptake under flow conditions, the following mathematical equation that connects experimental factors, or x , with the response variable, or y , is generated [15]:

$$y = \beta_0 + \beta_1x_1 + \beta_2x_2 + \beta_3x_3 + \beta_{12}x_1x_2 + \beta_{13}x_1x_3 + \beta_{23}x_2x_3 + \beta_{11}x_1^2 + \beta_{22}x_2^2 + \beta_{33}x_3^2 \quad (1)$$

The response variable y represents the percentage removal of pollutants under flow conditions. In the above equation, x_1 , x_2 , and x_3 are presenting the input experimental factors or variables, namely bed depth, flow rate, and inlet phenol concentration, respectively. Basically, the above equation is composed of three terms: a) linear terms (x_1 , x_2 , and x_3), which represent the main or linear effect of each variable on phenol adsorption; b) interaction terms (x_1x_2 , x_1x_3 , x_2x_3), which represent the effect of interaction between variables on phenol adsorption; and c) quadratic terms (x_{11} , x_{22} , and x_{33}), and these terms represent the non-linear effect of each variable on phenol adsorption. The estimated coefficients of the above equation were crucial to understanding the overall effects of the experimental factors on phenol adsorption. The intercept (β_0) indicated the predicted phenols uptake

under flow conditions when bed depth, flow rate, and inlet concentration are at their middle levels in CCD design. The sign and magnitude of the coefficients ($\beta_1, \beta_2, \beta_3$) indicated the direction and strength of the linear effect of factors on phenols uptake [15]. A positive sign indicates that increasing the factor ends up with increasing pollutant uptake or vice versa. On the other hand, the larger magnitude of the coefficient indicates a higher effect of the factor or vice versa. The sign and magnitude of the coefficients ($\beta_{12}, \beta_{13}, \beta_{23}$) reflected the direction and strength of the interaction effect of factors on phenols adsorption. A positive sign indicated that the factors work synergistically, i.e., enhancing each other's effect, for phenol uptake. However, the negative sign indicated that the factors work antagonistically, i.e., counteract each other's effect, for phenols uptake. The larger magnitude of the coefficient indicated their stronger interaction effect on phenol uptake. The sign and magnitude of the coefficients ($\beta_{11}, \beta_{22}, \beta_{33}$) indicated the direction and degree of curvature of the response surface. A positive sign indicated a convex curvature, i.e., phenols uptake increases then decreases or vice versa. However, negative sign indicated a concave curvature, i.e. phenols uptake increases then decreases or vice versa. The higher magnitude of the quadratic coefficient indicated a stronger curvature effect or vice versa. In CCD, the total number of experiments (N) is often estimated as [28]:

$$N = 2^k + 2k + C \quad (2)$$

The variables 2^k , $2k$, and C represent the number of factorial points, the number of axial points, and the number of central points, respectively. For small to medium-sized designs ($k < 5$), C are typically set at 3-5 points. For the three factors and at the pre-selected ranges, a central composite design was provided for optimizing phenol and 2-Nitrophenol adsorption by compost under continuous flow conditions, as summarized in Table 3. The original and coded values for the examined factors are also provided in Table 3.

Table 3 Three-level and three-factor central composite design used for optimization of phenol and 2-Nitrophenol uptake by compost under flow conditions

Code	Factor (unit)	Levels				
		-1	0	1		
x_1	Bed Depth (cm)	5.0	10.0	15.0		
x_2	Flow Rate(mL/min)	1.0	2.0	3.0		
x_3	Inlet Conc. (mg/L)	50.0	100	150.0		
Column Test	Independent variable or experimental factor			Dependent variable y (Bed Capacity q_{bed} mg/g)		
	x_1	x_2	x_3	Phenol	2-Nitrophenol	
1	-1.0 (5.0)	-1.0 (1.0)	-1.0 (50.0)	14.3	20.1	
2	-1.0 (5.0)	-1.0 (1.0)	1.0 (100)	22.0	29.3	
3	-1.0 (5.0)	1.0 (3.0)	-1.0 (50.0)	10.4	17.4	
4	-1.0 (5.0)	1.0 (3.0)	1.0 (150.0)	15.5	23.3	
5	1.0 (15.0)	-1.0 (1.0)	-1.0 (50.0)	33.4	40.6	
6	1.0 (15.0)	-1.0 (1.0)	1.0 (100.0)	74.3	84.7	
7	1.0 (15.0)	1.0 (3.0)	-1.0 (50.0)	11.2	19.4	

8	1.0 (15.0)	1.0 (3.0)	1.0 (150.0)	19.1	26.3
9	-1.7 (1.6)	0.0 (2.0)	0.0 (100.0)	4.2	11.2
10	1.7 (18.4)	0.0 (2.0)	0.0 (100.0)	22.7	30.5
11	0.0 (10.0)	-1.7 (0.32)	0.0 (100.0)	33.6	40.7
12	0.0 (10.0)	1.7 (3.8)	0.0 (100.0)	18.5	25.3
13	0.0 (10.0)	0.0 (2.0)	-1.7 (15.9)	14.5	21.4
14	0.0 (10.0)	0.0 (2.0)	1.7 (184.0)	33.2	40.4
15	0.0 (10.0)	0.0 (2.0)	0.0 (100.0)	50.4	58.5
16	0.0 (10.0)	0.0 (2.0)	0.0 (100.0)	50.5	58.3
17	0.0 (10.0)	0.0 (2.0)	0.0 (100.0)	50.3	58.1

As shown in Table 3, 17 adsorption tests were needed for maximizing adsorption of pollutants by compost. Generation of CCD design and RSM analysis were performed using Chemoface® program which operated under the Matlab® environment [29]. For both pollutants, fixed bed adsorption and measuring breakthrough curves were performed as following. A solution of known inlet concentration C_{inlet} was pumped inside backed column at a pre-selected flow rate. The level of pollutants in the effluent solution $C_{effluent}$ was periodically measured with the aid of UV-Vis spectrometer at 270 nm. Breakthrough and exhaustion points were reported when the ratio $C_{effluent}/C_{inlet}$ equal to 0.1 and 0.9, respectively. Percentage removal of phenol or 2-Nitrophenol is estimated:

$$y(\%) = \frac{C_{inlet} - C_{effluent}}{C_{inlet}} \times 100 \quad (3)$$

In Eq. 3, percentage removal of adsorbate was estimated at breakthrough point. Removal of both pollutants under flow conditions will be performed under the optimum operational factors which will be predicted by CCD-RSM.

2.5 Regeneration and reusability of compost under continuous flow conditions

Regeneration of compost was carried out under flow conditions as follows: 200.0 mL of the eluent reagent was pumped inside the exhausted adsorbent at variable flow rates of 0.2-0.8 mL/min. The reagents, including HCl, CH₃CH₂OH, and NaOH, were tested for elution purposes. The effect of NaOH on phenolic compound elution was studied at different concentrations (0.01-0.2 M). The concentration of desorbed pollutant was measured using a spectrophotometer at 270 nm. The extent of desorption of the pollutants from the exhausted adsorbent was determined as follows:

$$\%Desorption = \frac{Conc.of\ desorb\ pollutant\ (\frac{mg}{L}) \times volume\ of\ collected\ solution\ (L)}{q_{bed} (\frac{mg}{g}) \times mas\ of\ packed\ bed\ (g)} \times 100 \quad (4)$$

After surface regeneration, the column was washed with distilled water at a flow rate of 1.0 mL/min in preparation for another adsorption-desorption cycle. The reusability of compost was examined for phenol and 2-Nitrophenol.

3 Results and Discussion

3.1 Characterization of compost as a safe adsorbent for phenolic compound

Unlike other biosorbents, compost is generated from complex biological degradation processes, and hence its characterization is rather necessary. Before application as a bioadsorbent for phenolic compounds, the maturity, stability, and bioavailability of heavy metals of compost should be evaluated [11]. As summarized in Table 2, many physiochemical characterization tests were carried out to check the maturity of the prepared compost. As shown in Table 2, both pH and electrical conductivity indicated the maturity of the prepared compost with values of 6.0-8.5 and <3.0 ds/m, respectively [22]. The prepared compost was also rich with organic matter, which also evidenced the presence of humic and fulvic acids, and these characteristics will improve its affinity for removing organic pollutants. The measured bulk density of the prepared compost, 389.0 kg/m³, also indicated its maturity, which should not be more than 400 kg/m³ [24]. Another important index that indicates the maturity and stability of the compost is C/N ratio, which is recommended to be higher than 20 [11]. The estimated porosity and specific surface area were high and comparable to other compost prepared from variable organic feedstocks [11]. The measured FITR of the prepared compost indicated the presence of many surface functional groups, including –COOH, - OH, - RCON, -R-C-H, - NH₂, and –C=O. The earlier functional groups can be involved in attracting phenolic compounds through different chemical forces. In addition to the earlier tests, analysis indicated that the total contents of toxic heavy metals were within the safe limits for safe application as recommended by the standard regulations [11,26]. In fact, compost toxicology is also dependent on the bioavailable or leachable forms of heavy metals rather than the total concentration. Many researchers have reported the reduction of bioavailable and leachable forms of heavy metals after composting [30]. According to the toxicity characteristics leaching procedure, the amount of the bioavailable heavy metals was much lower than the safe limits, as shown in Table 2. Thus, the prepared compost with a negligible concentration of bioavailable forms of heavy metals is safe for adsorption purposes.

3.2 Optimization of phenolic compounds adsorption under flow conditions using CCD-RSM

Based on CCD-RSM analysis, the combined effect of experimental factors on phenol adsorption under flow conditions is presented in the following quadratic equation (Eq. 5) and the significance of coefficients of all terms are also provided Table 4:

$$y = 49.4 + 7.8x_1 - 8.3x_2 + 6.9x_3 - 8.3x_1x_2 - 11.7x_1^2 - 7.3x_2^2 - 8.0x_3^2 \quad (5)$$

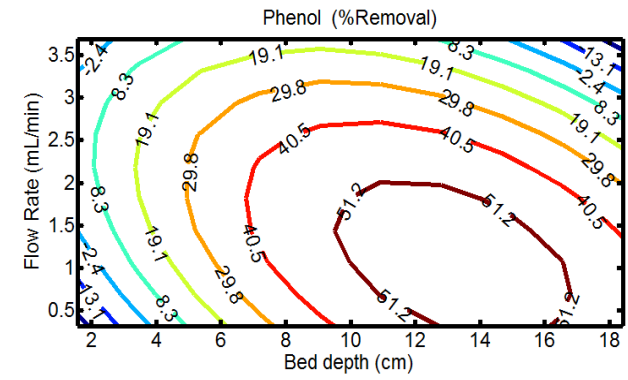
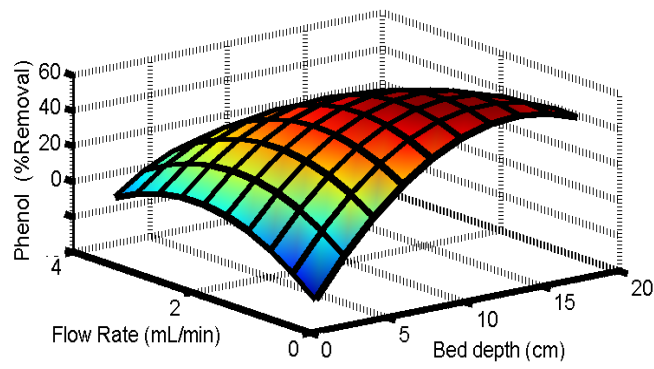
Table 4 Estimated regression coefficients and their significance for modeling phenol adsorption under flow conditions

Term	B	Standard Error	<i>t</i>	<i>p</i>	Significant ^a
β_0 ($x=1$)	49.4	6.004	8.232	0.000174	Yes
Bed Depth (cm)(x_1)	7.8	2.305	3.376	0.014925	Yes
Flow Rate (mL/min)(x_2)	-8.3	2.304	3.597	0.011404	Yes
Inlet Conc. (mg/L)(x_3)	6.9	2.304	2.984	0.024478	Yes
x_1*x_2	-8.3	3.011	2.739	0.03375	Yes
x_1*x_3	4.5	3.011	1.494	0.185699	No
x_2*x_3	-4.5	3.011	1.494	0.185699	No
x_1^2	-11.7	2.798	4.186	0.00577	Yes
x_2^2	-7.3	2.798	2.607	0.040259	Yes
x_3^2	-8.0	2.798	2.860	0.028797	Yes

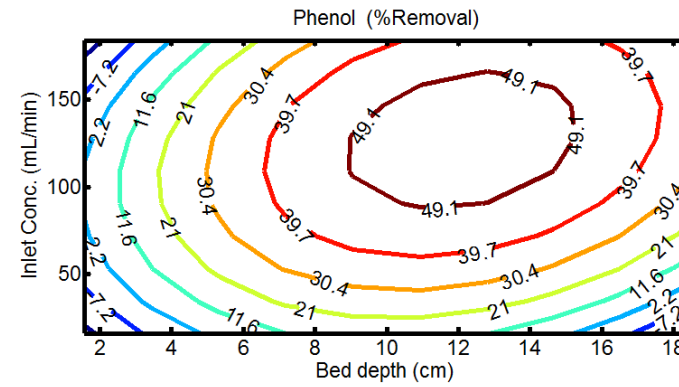
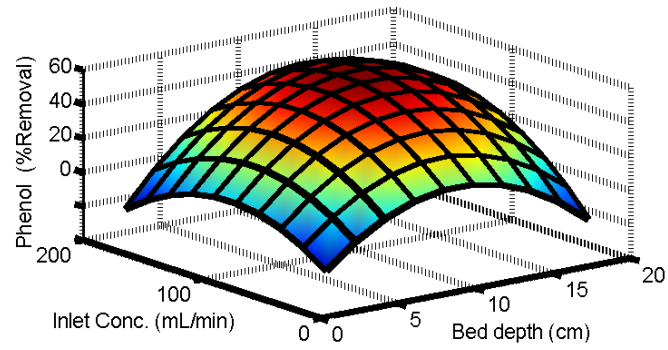
^aP<0.05

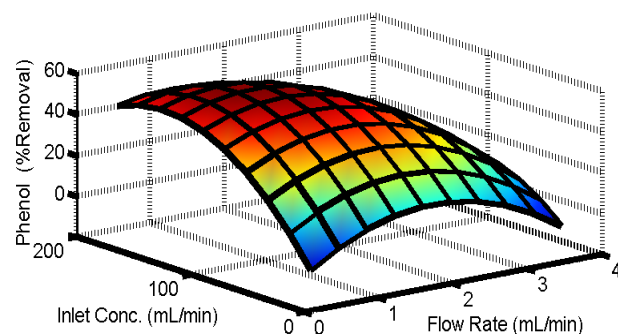
Initially, statistical analysis of the significance coefficients, as summarized in Table 4, indicated that interaction effects of bed depth and flow rate with inlet concentration were not significant for modeling phenol adsorption and hence excluded from Eq. 5. Based on the magnitude of the intercept, the predicted removal of phenol at the middle levels of factors (10.0 cm, 1.0 mL/min, and 100 mg/L) is 49.5%, as shown in Eq. 5. Moreover, the linear coefficient indicated a high strength but with different directions of the variables on phenol adsorption under flow conditions. For instance, bed depth (x_1) and inlet concentration (x_3) have positive linear effects on phenol adsorption with coefficient values of +7.8 and +6.9, respectively. However, flow rate (x_2) has a high negative effect on phenol uptake under flow conditions with a coefficient value of -8.3, as indicated in Eq. 5. Accordingly, CCD analysis suggested that higher removal of phenol is achievable at deeper bed depth and higher inlet concentration and slow flow rate. According to CCD analysis, bed depth and flow rate counteract each other's effect for phenol adsorption. As shown in Eq. 5, the negative interaction effect (-8.3) indicates that the effect of bed depth on phenol adsorption is reduced at a faster flow rate or vice versa. For higher adsorption of phenol under flow conditions, a deeper bed depth is recommended while maintaining a low flow rate during the process. Interestingly, CCD analysis indicated that all factors have a quadratic or nonlinear effect with negative coefficients of -7.3, -8.0, and -11.7, as shown in Eq. 5. The strongest effect was reported for bed depth, or x_1 , with a negative coefficient of -11.7, which indicates a concave curvature, meaning that phenol adsorption increases with bed depth up to a point, then declines. The same discussion is holding true for the other factors. Discussing the mutual influence of the operational factors on phenol uptake under flow conditions is necessary for full utilization of the adsorbent. The interaction effects of the examined experimental variables on phenol uptake are provided in Figure 1.

A



B





C

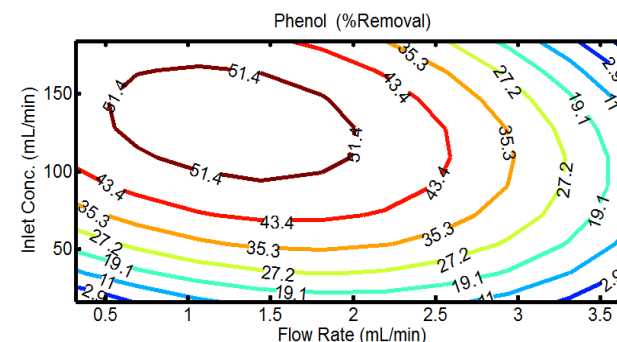


Figure 1 Surface and contour plots for the mutual effect of bed depth and flow rate on phenol adsorption. Plots were obtained at 100 mg/L (A) Surface and contour plots mutual effect of bed depth and concentration on phenol adsorption. Plots were obtained at 2.0 mL/min (B) Surface and contour plots for the mutual effect of concentration and flow rate on phenol adsorption. Plots were obtained at bed depth 10 cm (C)

The mutual interaction of bed depth and flow rate is presented as 2D and 3D plots in Figure 1A. As shown in Figure 1, the 3D plot indicated a concave curvature where phenol uptake increased with bed depth and flow rate up to a point and then reduced. Analysis of a 2D or contour plot is necessary to locate the optimal values for bed depth and flow rate that maximize phenol uptake under flow conditions. As shown in Figure 1A, a high retention of phenol is predicted while keeping bed depth higher than 10.0 cm and flow rate less than 2.0 mL/min. The interactive effect between bed depth and flow rate had a significant impact on the adsorption capacity of phenol under flow conditions. Generally, pollutant uptake under flow conditions increased at deeper bed depths and slow flow rates [31]. At slow flow rates and deeper depths, the adsorbate molecules have longer time to adsorb on adsorbent surface, while the increased bed depth offers more active site for adsorption, leading to higher adsorption capacities [31]. In fact, lower flow rate and longer bed depths offer longer residence time and mass transfer for pollutant adsorption. The mutual interaction between bed depth and inlet concentration is provided in Figure 1B. As shown, the 3D plot exhibited a high concave curvature as phenol adsorption increased with bed depth and inlet concentration up to a point and then highly reduced. To maximize phenol uptake under flow conditions, 2D plots indicate that bed depth should be higher than 10.0 cm and inlet concentration should be within 100–150 mg/L. The high adsorption of pollutants at deeper bed depth and high inlet

concentration is often reported [32]. At deeper bed depths, the adsorbates have more residence time to interact with the adsorbent surface, and the increased bed depth also provides more active sites, leading to higher adsorption. Higher inlet concentration also increases the driving force for adsorption under flow conditions [32]. The interaction effect between inlet phenol concentration and flow rate is provided in Figure 1C. As shown, the 3D plot exhibited a concave curvature as phenol uptake increased with flow rate and inlet concentration up to a point and then declined. To increase phenol uptake under flow conditions, the corresponding 2D plots predicted that this factor should be within 0.5–2.0 mL/min, while the inlet concentration should be higher than 100 mg/L. In fixed-bed adsorption, pollutant uptake is often improved at lower flow rate and higher inlet concentration. At lower flow rate, pollutant molecules have more time to contact with the surface of adsorbent, while, higher inlet concentration increases the driving force for adsorption [31]. Based on CCD-RSM calculations, the influence of experimental factors on 2-Nitrophenol adsorption under flow conditions is provided in equation 6 while the significance of coefficients is summarized Table 5.

$$y = 57.4 + 8.2x_1 - 8.3x_2 + 7.2x_3 - 8.8x_1x_2 - 11.8x_1^2 - 7.6x_2^2 - 8.0x_3^2 \quad (6)$$

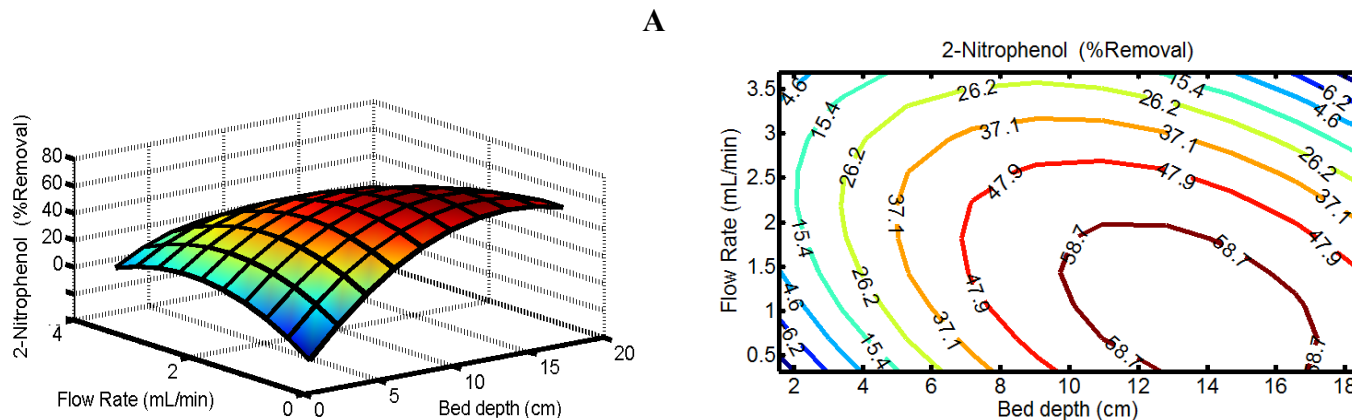
Table 5 Estimated regression coefficients and their significance for modeling 2-Nitrophenol adsorption under flow conditions

Term	B	Standard Error	<i>t</i>	<i>P</i>	Significant ^a
β_0 ($x=1$)	57.4	6.407	8.958	0.00010808	yes
Bed Depth (cm)(x_1)	8.2	2.459	3.333	0.015738647	yes
Flow Rate (mL/min)(x_2)	-8.3	2.459	3.371	0.015016936	yes
Inlet Conc. (mg/L)(x_3)	7.2	2.459	2.917	0.026748776	yes
$x_1 * x_2$	-8.7	3.213	2.723	0.034494141	yes
$x_1 * x_3$	4.5	3.213	1.401	0.210892688	no
$x_2 * x_3$	-5.0	3.213	1.556	0.170678709	no
x_1^2	-11.8	2.986	3.953	0.007511259	yes
x_2^2	-7.6	2.986	2.532	0.044551296	yes
x_3^2	-8.3	2.986	2.769	0.032467358	yes

^aP<0.05

As provided in Eq. 6, linear, interaction, and quadratic terms were included for modeling 2-Nitrophenol adsorption on compost under fixed-bed conditions. In general, the effect of experimental factors on the adsorption behavior of 2-Nitrophenol was similar to that reported for phenol, except that the former pollutant showed a higher interaction with compost under flow conditions. The slope of Eq. 6 indicated that percentage of 2-Nitrophenol removal was 57.4% at the middle levels of the experimental variable, 10.0 cm, 1.0 mL/min, and 100.0 mg/L for bed depth, flow rate, and inlet concentration, respectively. Based on the magnitude of the slopes, 2-Nitrophenol has a higher adsorption on compost compared to phenol with values of 57.4 and

49.4%, respectively. According to the signs and magnitudes of linear coefficients, increasing bed depth and inlet concentration ends up with higher 2-Nitrophenol adsorption with values of +8.2 and +7.2, respectively. In contrast, increasing flow rate results in lowering solute uptake with a negative coefficient of -8.3, as shown in Eq. 6. As was the case for phenol, coefficients of interaction terms of bed depth and flow rate with inlet concentration were statistically insignificant (Table 5), and hence both terms were removed from Eq. 6. On the other hand, the variables bed depth and flow rate counteract each other's effect on solute adsorption as inferred from the negative coefficient (-8.8) and as shown in Eq. 6. Accordingly, the effect of flow rate on 2-Nitrophenol adsorption is reduced with a shorter bed depth. As indicated in Eq. 6, flow rate x_2 , inlet concentration x_3 , and bed depth x_1 have a negative quadratic effect on 2-Nitrophenol adsorption with values of -7.6, -8.0, and -11.8, respectively. The negative sign of quadratic coefficients indicated that 2-Nitrophenol adsorption increases by increasing all variables up to a point, then reduces by reducing variables, i.e., a concave curvature in the response surface. To further understand the interaction effects among factors, it is required to examine the 2 and 3D plots produced by CCD-RSM for 2-nitrophenol adsorption under flow circumstances. Furthermore, contour plots make it simple to locate the ideal set of factors that maximize 2-Nitrophenol adsorption. The obtained 2 and 3D plots for 2-Nitrophenol adsorption are provided in Figure 2.



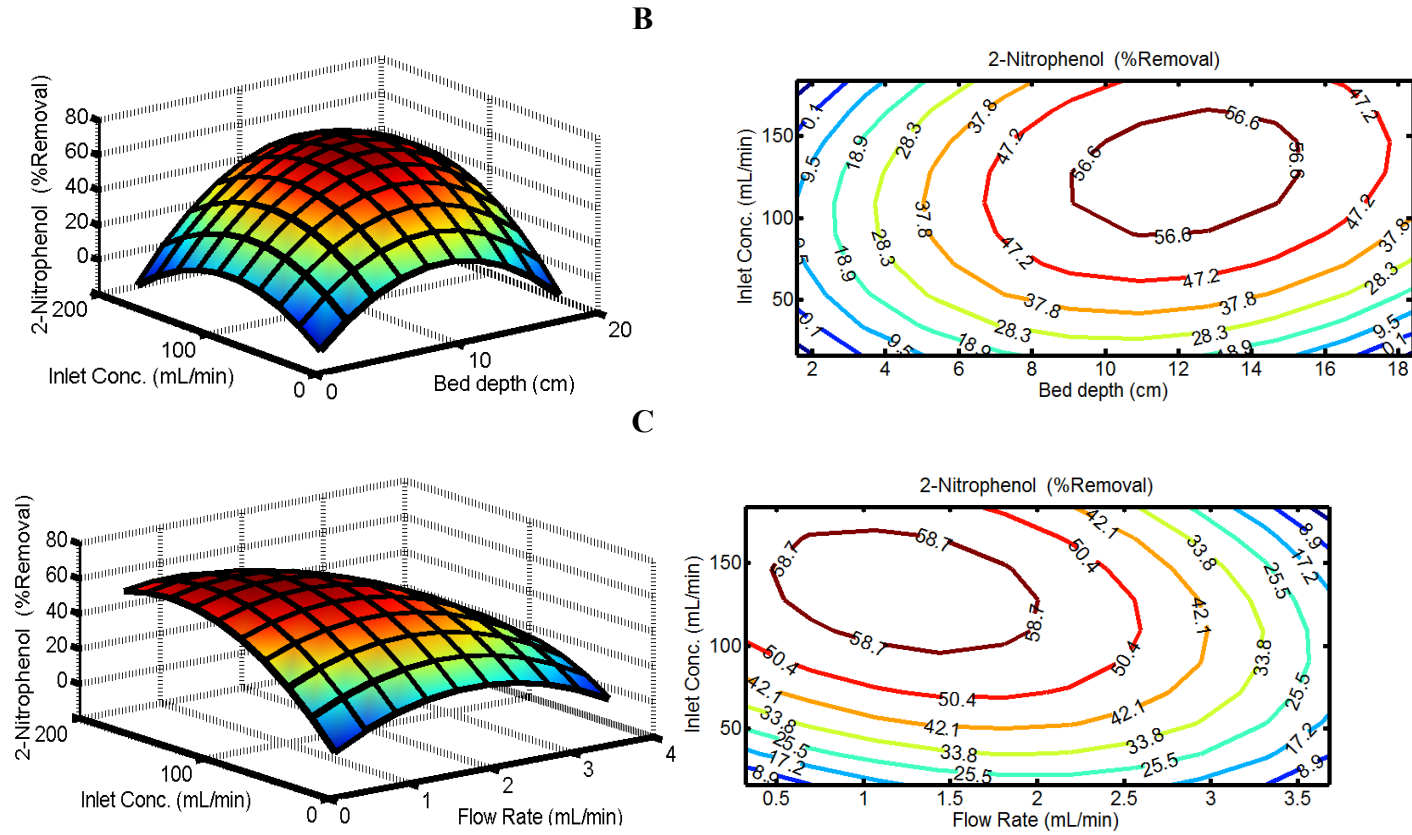


Figure 2 Surface and contour plots for the mutual effect of bed depth and flow rate on 2-Nitrophenol adsorption. Plots were obtained at 100 mg/L (A) Surface and contour plots for the interaction effect of bed depth and inlet concentration on 2-Nitrophenol adsorption. Plots were obtained at 2.0 mL/min (B) Surface and contour plots generated for the interaction effect of inlet concentration and flow rate on phenol adsorption. Plots were obtained at bed depth 10 cm (C)

The mutual interaction between bed depth and flow rate on 2-Nitrophenol adsorption are presented in Figure 2A. As shown, the negative curvature in the surface plot indicated that increasing bed depth and flow rate leading to high solute uptake up to a point, then decline. The related contour plot evidenced that adsorption of 2-Nitrophenol is notably increased at deeper bed depth (>10.0 cm) while maintaining low flow rate (<1.5 mL/min). The preferential uptake of 2-Nitrophenol at deeper bed depth and slow flow rate is attributed to the availability of large number of active site and the long contact time. The interaction effect between inlet concentration and bed depth on 2-Nitrophenol uptake showed a high negative curvature in the surface response as shown in Figure 3B. The ideal combination of the earlier variables to maximize 2-Nitrophenol retention under flow conditions is: bed depth 10-15 cm and inlet concentration 100-150 mg/L as shown in Figure. The high retention of 2-Nitrophenol at deeper bed depths and higher inlet concentration is often attributed to the presence of a large number of active sites and the higher driving force for adsorption. The interaction effect between inlet concentration and flow rate showed a lower negative curvature as shown in Figure 2C. The corresponding 2D plot indicated that the ideal combination of the earlier variables to ensure a high retention of the pollutant is: flow rate 0.5-1.5 mL/min and inlet concentration 100-150 mg/L. The preferential uptake of 2-Nitrophenol at lower flow rate and higher pollutant concentration is attributed to the longer interaction time and higher driving force for adsorption process. According to the previous discussions, the following circumstances can optimize phenol and 2-Nitrophenol adsorption under flow conditions: inlet concentration should be higher than 100 mg/L, bed depth should be deeper than 10.0 cm, and flow rate should be less than 2.0 mL/min. In conclusion, using CCD-RSM to forecast the ideal operational conditions for maximizing phenol adsorption by compost and under fixed-bed conditions proved to be very practicable.

3.3 Removal of phenolic compounds under optimized operational conditions

After determining the optimum ranges of operational factors that maximized uptake process, removal of both pollutants was performed under flow conditions for better assessment of compost as an adsorbent. The measured breakthrough curves at different operational factors of phenol 2-Nitrophenol are provided in Figure 3 and 4, respectively.

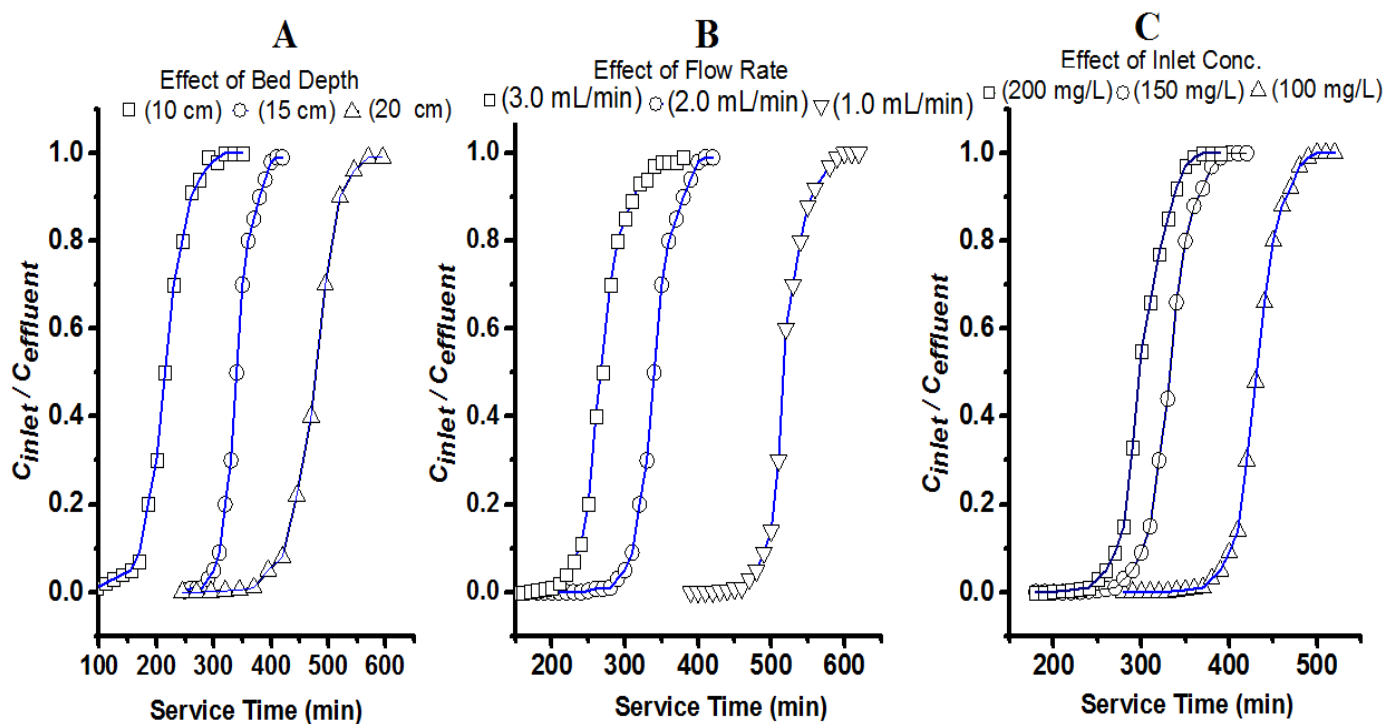


Figure 3 Effect of operational factors on phenol adsorption. Effect of bed depth was examined at 2.0 mL/min and 150.0 mg/L (A). Effect of flow rate was examined at 15.0 cm and 150.0 mg/L (B). Effect of inlet concentration was examined at 15.0 cm and 2.0 mL/min (C). Lines present Thomas modeling.

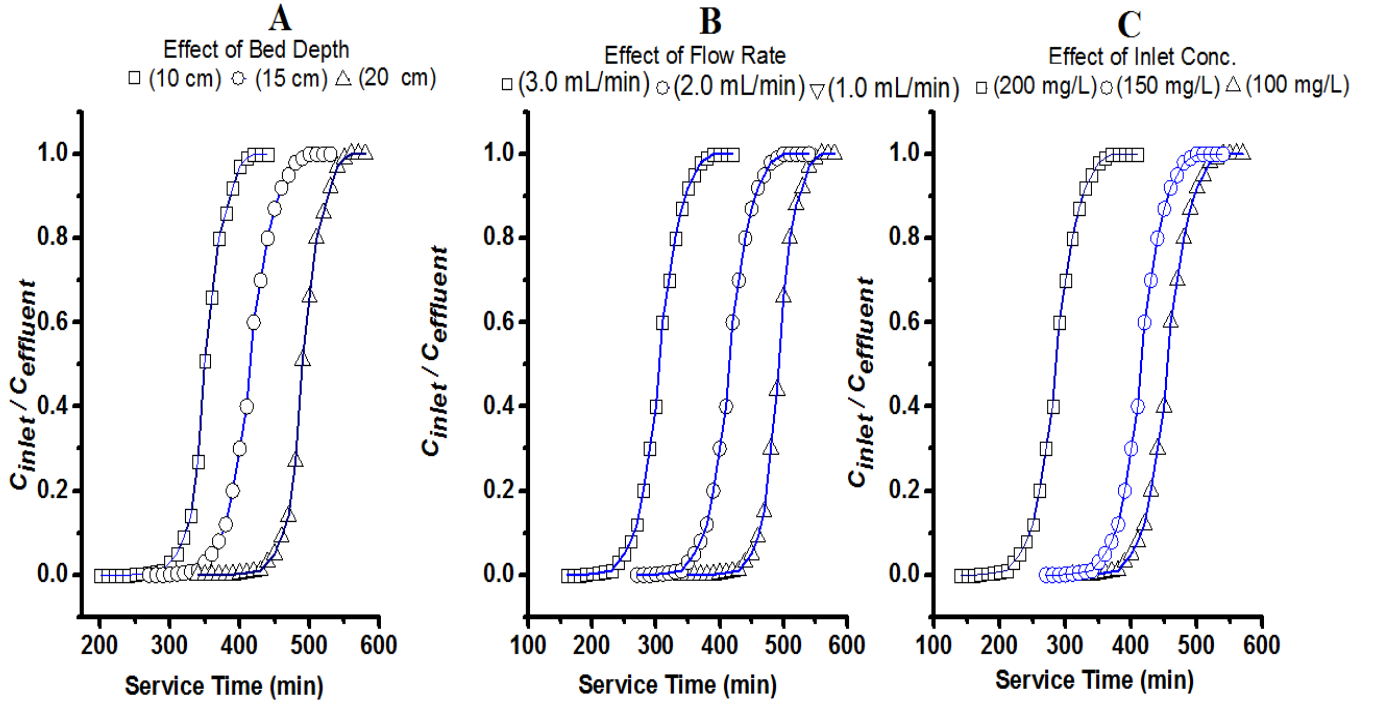


Figure 4 Effect of operational factors on 2-Nitrophenol adsorption. Effect of bed depth was examined at 2.0 mL/min and 150.0 mg/L (A). Effect of flow rate was examined at 15.0 cm and 150.0 mg/L (B). Effect of inlet concentration was examined at 15.0 cm and 2.0 mL/min (C). Lines present Thomas modeling.

As shown in Figures 3 and 4, typical or S-shaped breakthrough curves for phenol and 2-Nitrophenol were reported. At the initial stage, the curve indicates a zero level of pollutants as the compost effectively captures the pollutants. At the breakthrough point, $C_{effluent}/C_{inlet} = 0.1$, the concentration of adsorbates starts to rise in the treated solution, and this indicates that the compost is close to saturation. In the steepness region, which is known as the mass transfer zone, the level of pollutants increases sharply, and the steepness of this zone often depends on operational factors, including bed depth and flow rate [13]. In the final or exhaustion stage, the curve plateaus where the ratio $C_{effluent}/C_{inlet}$ is close to 1.0. For better assessment of the treatment process under flow conditions, the breakthrough curves were presented using common Thomas model. Among mathematical models, this model was the most applied for modeling pollutants uptake under flow conditions and this model based on the following assumptions [13,33]: (a) adsorption and desorption follow Langmuir theory; (b) negligible axial and radial dispersion; (c) adsorption rate of the solute follows pseudo-second order kinetics; (d) negligible intra-particle diffusion and negligible external resistance. Thomas model is presented in equation 7 [13,33]:

$$\frac{C_{effluent}}{C_{inlet}} = \frac{1}{1 + e^{\left(\frac{k_{Th} q_{Th}^m}{u} - k_{Th} C_{inlet} t\right)}} \quad (7)$$

Where $C_{effluent}$ (mg L^{-1}), C_{inlet} (mg L^{-1}), k_{Th} ($\text{L mg}^{-1} \text{ min}^{-1}$), u (ml min^{-1}), q_{Th} (mg g^{-1}), m (g), and t (min) are effluent adsorbate concentration, inlet adsorbate concentration, Thomas rate constant flow rate, saturation capacity at exhaustion point, mass of the compost and service time of the column, respectively. The parameters of Thomas model including q_{Th} and k_{Th} are rather important as they determine bed saturation capacity and adsorption rate as a function with operational factors [13]. The parameters of Thomas model were estimated by non-linear regression analysis using Origin Pro2016® software. The average relative error ARE was used to evaluate the degree of closeness between experimental and model-predicted results as following [34]:

$$ARE = \frac{100}{n} \sum_{i=1}^p \left| \frac{(C_{effluent}/C_{inlet})_{pred} - (C_{effluent}/C_{inlet})_{exp}}{(C_{effluent}/C_{inlet})_{exp}} \right| \quad (8)$$

Where $(C_{effluent}/C_{inlet})_{pred}$, $(C_{effluent}/C_{inlet})_{exp}$ and n are the predicted concentration ratio, the experimental concentration ratio, and number of experimental points in the curve, respectively. The estimated parameters of Thomas model for removing phenol and 2-Nitropheol under flow conditions are provided in Table 6.

Table 6 Parameters of Thomas model and performance indicators of compost for removing phenolic compounds under flow conditions

Factor	Level	Parameters of Thomas Model and Performance Indicators						
		Phenol						
		K_{Th}	q_{Th} (mg/g)	ARE ^d	AER ^e (g/L)	BV ^f	Breakthrough time (hr) ^g	Exhaustion time (hr) ^g
Bed-depth (cm) ^a	10.0	0.00052	17.4	4.4	9.4	43.5	3.3	4.4
	15.0	0.00044	22.3	6.5	8.5	77.3	5.2	6.3
	20.0	0.00037	27.2	8.3	7.8	87.0	7.2	8.7
Flow rate (mL/min) ^b	1.0	0.00032	29.5	9.3	3.4	98.4	8.4	9.4
	2.0	0.00044	22.3	6.5	8.5	77.3	5.2	6.3
	3.0	0.00063	18.3	7.5	11.6	40.3	4.1	5.5
Concentration (mg/L) ^c	100.0	0.00057	19.4	6.5	6.7	110.2	6.6	7.8
	150.0	0.00044	22.3	5.8	8.5	77.3	5.2	6.3
	200.0	0.00032	25.2	3.4	11.7	41.3	4.5	5.9
2-Nitrophenol								
Bed-depth (cm) ^a	10.0	0.00081	29.5	8.0	10.6	63.1	5.5	6.4
	15.0	0.00076	35.7	7.2	9.6	94.6	6.3	7.9
	20.0	0.00063	42.8	5.6	7.7	142.0	7.8	8.5
Flow rate (mL/min) ^b	1.0	0.00055	43.4	2.5	6.7	119.4	7.9	8.9
	2.0	0.00076	35.7	7.2	9.6	94.6	6.3	7.9
	3.0	0.00081	29.5	5.0	12.3	59.4	4.5	5.8
Concentration (mg/L) ^c	100.0	0.00084	31.8	2.9	7.4	108.4	7.0	8.3
	150.0	0.00076	35.7	7.2	9.6	94.6	6.3	7.9
	200.0	0.00055	39.7	3.7	11.7	62.7	4.1	5.7

^a Flow rate 2.0 mL/min; initial concentration 150 mg/L.^b Bed-depth 15 cm; Initial concentration 150 mg/L .^c Bed-depth 15 cm; flow rate 2.0 mL/min.^d Average relative error^e Adsorbent exhaustion rate estimate at breakthrough time^f Processed bed volume estimate at breakthrough time^g Breakthrough and exhaustion times as estimated from breakthrough curve.

3.3.1 Effect of bed depth on the performance of compost for removing pollutants

As indicated in Table 6, adsorption behavior of both pollutants using different bed depths was adequately presented by Thomas model a low prediction error 4.4-8.3. For both pollutants, increasing bed from 10.0 to 20.0 cm has notably increased column service time, as shown in Figures 3A and 4A. As summarized in Table 6, doubling bed depth led to an increase in service time from 3.3 to 7.24 and from 5.5 to 7.9 hr for removing phenol and 2-Nitrophenol, respectively. Analysis of breakthrough curves that were measured at different bed depths using the Thomas model showed that bed capacity q_{Th} increased with bed depth, while uptake rate k_{Th} reduced. For instance, increasing bed depth up to 20.0 cm had increased bed capacity from 17.4 to 27.2 and from 29.5 to 42.8 mg/g for phenol and 2-Nitrophenol, respectively. On the other hand, adsorption rate under flow conditions was reduced from 0.00052 to 0.00037 and from 0.00081 to 0.00076 L mg⁻¹ min⁻¹ with increasing bed depth for phenol and 2-Nitrophenol, respectively as summarized in Table 6. At deeper bed depths, more surface area, more active sites for adsorption, and longer contact with inlet solution and this ended up with higher bed capacity. In contrast, at deeper depths large numbers of active sites are available for adsorption and this lead to reduce reaction rate of k_{Th} . The results indicated that 2-Nitrophenol manifested higher adsorption on compost compared to phenol. The higher affinity of 2-Nitrophenol for compost is attributed to the following reasons; (a) As shown in Table 1, the solubility of phenol in water is 47 times higher than that of 2-Nitrophenol. Accordingly, the high solubility of phenol will reduce its affinity toward compost compared to the other pollutant. In addition, 2-Nitrophenol has a higher partition coefficient compared to phenol and this may increase its potential to adsorb on organic-rich compost adsorbent; (b) the presence of nitro groups shall increase the interaction of 2-Nitrophenol with the surface via H-bonding forces and π - π with aromatic system in compost. Similar

observations were reported in the literature on the high adsorption of nitro-phenol compared to phenol [7,35-37]. In addition to Thomas model, the following parameters including bed volume and adsorbent exhaustion rate are often used to measure the performance of the adsorbent for removing pollutants under flow conditions [31,38]. Bed volume BV essentially measures the volume of the solution that can undergo treatment per unit volume of adsorbent bed, hence high BV indicates high performance [39]. BV is estimated as [38]:

$$\text{Bed Volume } BV = \frac{\text{Treated Volume at Breakthrough Point (L)}}{\text{Bed Volume (L)}} \quad (9)$$

In addition, the rate of adsorbent exhaustion AER is often used to establish the regularity at which the adsorbent needs to be replaced. AER is estimated as [40]:

$$\text{Adsorbent Exhaustion Rate } AER \left(\frac{g}{L} \right) = \frac{\text{Mass of Adsorbent (g)}}{\text{Treated Volume (L)}} \quad (10)$$

In general, the bed performance is considered effective at lower and higher values of AER and BV, respectively. As indicated in Table 6, increasing bed depth up to 20.0 cm has been increasing BV while reducing AER. For instance, doubling bed depth increased BV from 43.5 to 87.0 and from 63.1 to 142.0 for phenol and 2-Nitrophenol, respectively. In contrast, AER was notably reduced from 9.4 to 7.8 and from 10.6 to 7.7 g/L for phenol and 2-Nitrophenol, respectively. Similar observations were reported in the literature [38,40]. Accordingly, running fixed-bed adsorption at 20.0 cm depth was the most effective; as a large treated volume was obtained with a low consumption rate of the compost. Despite the high performance of compost for treating pollutants at bed depth 20.0 cm, however, this led to an increase in the column service time, which makes the use of 15 cm depth better as one ensure high adsorption in a reasonable service time.

3.3.2 Effect of flow rate on the performance of compost for removing pollutants

Adsorption behavior of pollutants under flow conditions and at variable flow rates was adequately presented by Thomas model with a prediction error 2.5-9.3, as summarized in Table 6. Moreover, increasing flow rate from 1.0 to 3.0 mL/min had reduced treatment time for both pollutants, as shown in Figures 3B and 4B. As shown in Table 6, increasing flow rate resulted in a significant decrease in breakthrough time from 8.4 to 4.1 and from 7.9 to 4.5 for phenol and 2-nitrophenol, respectively. Among operational factors, the longest treatment time was reported at flow rate 1.0 mL/min, as shown in Table 6. Modeling experimental breakthrough curves by Thomas model at variable flow rates indicated that bed capacity notably increased by reducing flow rate from 3.0 to 1.0 mL/min. In contrast, adsorption rate of pollutants increased at flow rates. As summarized in Table 6, reducing flow rate down to 1.0 mL/min has resulted in a high uptake capacity with values

of 29.5 and 43.4 mg/g for phenol and 2-Nitrophenol, respectively. In addition, rate of adsorption increased from 0.00032 to 0.00063 and from 0.00055 to 0.00081 L mg⁻¹ min⁻¹ for phenol and 2-Nitrophenol with flow rate, respectively, as shown in Table 6. The high adsorption capacity at lower flow rate could be attributed to the increased residence that offers for the pollutants more time to interact with composts. Moreover, at slower flow rate allow for more effective mass transfer between inlet solution and the adsorbent. In this line, slower flow rates can minimize channeling leading to better adsorbent utilization. In contrast, higher flow rates enhance mass transfer for the pollutants leading to a higher adsorption rate. Although a high adsorption capacity was reported at slow flow rate, however, this ended up with a long breakthrough time for phenol and 2-Nitrophenol with values of 8.4 and 7.9 hr, respectively, as shown in Table 6. On the other side, running fixed adsorption slower rates has resulted in a higher BV and lower exhaustion of the compost. For instance, reducing flow rate down to 1.0 mL/min has improved the performance of column for removing phenol with BV and AER values of 98.4 and 3.4 g/L as shown in Table 6. A similar trend was reported for 2-Nitrophenol which is common in the reported literature [31,38-40].

3.3.3 Effect of inlet concentration on the performance of compost for removing pollutants

As indicated in Table 6, Thomas model was adequate for presenting breakthrough curves of phenol and 2-nitrophenol at different inlet concentrations with a prediction error of 2.9-7.2. As shown in Figure 3C and Figure 4C, increasing inlet concentration leads to a faster breakthrough of the pollutants. In addition, increasing inlet concentration resulted in higher bed capacity for both pollutants. As summarized in Table 6, increasing inlet concentration from 100.0 to 200.0 mg/L has increased adsorption capacity of phenol and 2-Nitrophenol from 19.4 to 25.2 and from 31.8 to 39.7 mg/g, respectively. The higher adsorption of the pollutants under flow conditions is attributed to the increased driving forces for mass transfer at higher inlet concentrations. In contrast, increasing inlet concentration has reduced adsorption rate from 0.00057 to 0.00032 and from 0.00084 to 0.00055 L mg⁻¹ min⁻¹ for phenol and 2-Nitrophenol, respectively. The lower adsorption rate at higher inlet concentration is often attributed to the higher competition among molecules for active sites. As summarized in Table 6, increasing inlet concentration resulted in a fast saturation of the compost with breakthrough times of 4.5 and 4.1 hr for phenol and 2-Nitrophenol, respectively. In addition, increasing inlet concentration has affected the performance of the adsorber by reducing the volume of treated solution before breakthrough. For instance, upon increasing inlet concentration, the BV volume was highly reduced from 110.2 to 41.3 and from 108.4 to 62.7 for phenol and 2-Nitrophenol, respectively, as summarized in Table 6. In addition, a higher exhaustion rate of compost was also reported at high inlet concentration and for both pollutants. At high concentration,

the bed is saturated more quickly, leading to a higher or faster exhaustion of the adsorbent [38-40]. For effective utilization of compost for removing pollutants under follow conditions, the flow rate should be maintained at 1.0 L/min while bed depth at 20.0 cm and this in agreement with CCD-RSM analysis.

3.4 Reusability of compost and mechanism of interaction with phenolic compounds

Disposal of exhausted compost would result in environmental damage, and it must be disposed of according to applicable environmental legislation. The negative impact on the environment can be avoided by implementing sustainable recycling procedures [41,42]. In this work, regeneration of compost was carried out under flow conditions, i.e., desorption of pollutants was performed inside the adsorber. The main results of compost reusability are provided in Figure 5.

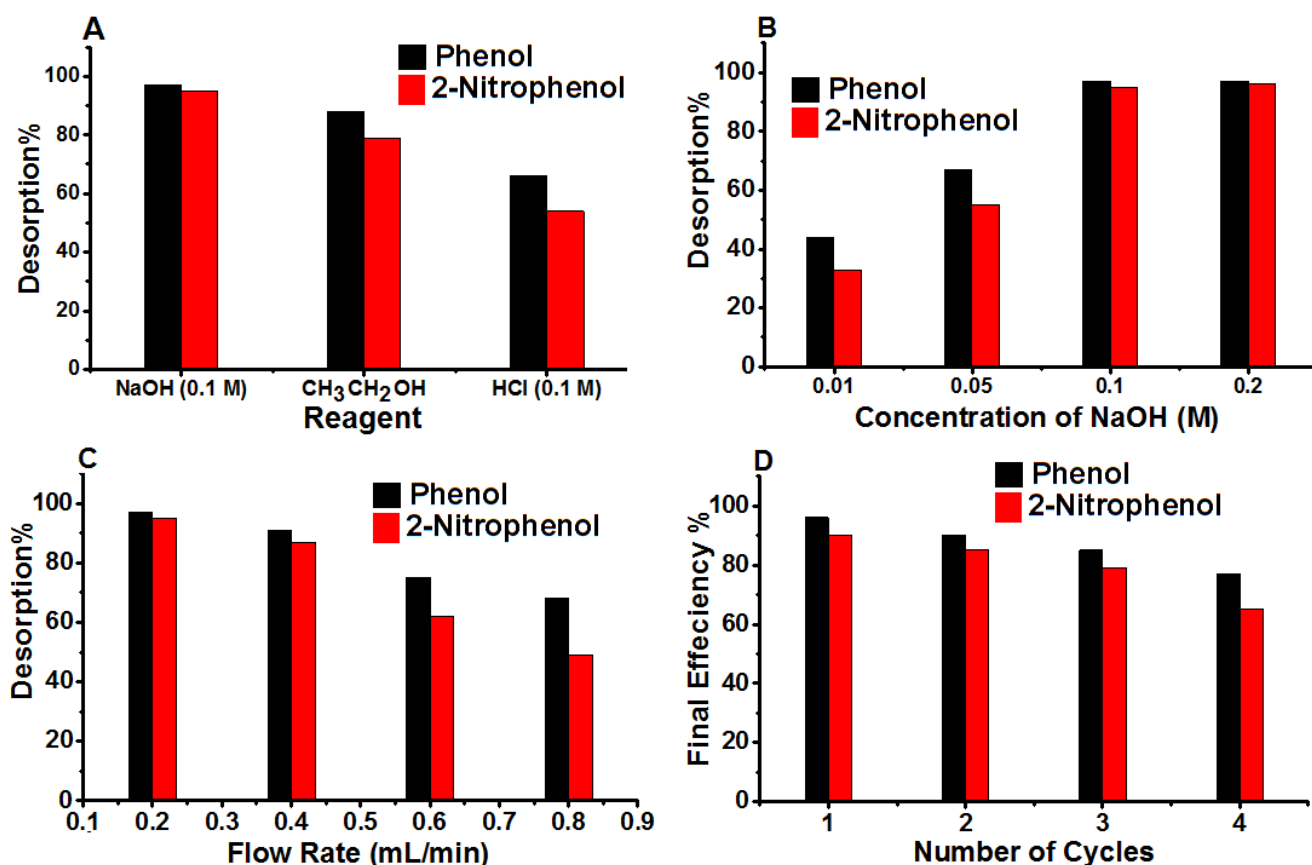


Figure 5 Regeneration solvents (A) Effect of NaOH concentration on elution (B) Effect of flow rate on elution by 0.1 M NaOH (C), Number of adsorption cycles using 0.1 M NaOH (D).

As shown in Figure 5A, the reagents including NaOH and CH₃CH₂OH were effective for eluting both pollutants with desorption efficiency higher than 80%. The desorption efficiency of HCl not high compared with other reagents. Among reagents, 0.1 M NaOH was most effective for surface regeneration with desorption efficiency higher than 95% for both pollutants. The high affinity of NaOH and CH₃CH₂OH for surface regeneration was reported in the literature [12,43-46]. As shown

in Figure 10, phenol showed a high desorption from the surface compared to 2-Nitrophenol and this would be attributed to the higher interaction affinity of earlier the pollutant with the compost. As shown in Figure 5B, concentration of NaOH has a high effect on the amount of eluted amount of adsorbed pollutants. The recovery of adsorbed pollutants increases with the concentration of eluent up to 0.1 M and remains constant after this level. The high desorption efficiency of NaOH for eluting phenol and 2-Nitrophenol is attributed to the followings [43,47]: a) hydrolysis of chemical forces, including H-bonding between phenol ($-OH$) and surface oxygen groups available in compost; and b) the higher solubility of phenol under basic environments. On the other hand, the high desorption efficiency of ethanol was attributed to the strong hydrophobic-hydrophobic forces of phenol with organic solvents [47]. As previously mentioned, the process of recovering adsorbed pollutants from compost was carried out under flow conditions, and therefore the flow rate was studied from this side. As shown in Figure 5C, lowering the flow rate from 0.8 to 0.4 mL/min resulted in higher pollutant recovery, and recovery was not highly increased after 0.4 mL/min. The higher recovery that was achieved at a lower flow rate was attributed to the increase in the reaction time between eluent and adsorbed pollutants. As shown in Figure 5D, compost was reusable for up to four cycles for removing phenol and 2-Nitrophenol with final capacities of 70 and 65%, respectively. The reduction in compost capacity after surface regeneration is often attributed to [48,49]: (a) high damage or attenuation of the surface active sites during the regeneration with NaOH, which reduces the adsorption affinity in the next cycles; (b) incomplete elution of the adsorbed phenolic compounds, which may reduce the capacity of compost; and (c) blockage of the porous channels of compost.

4 Performance of compost for removing phenol against other bioadsorbents

As a new bioadsorbent, the performance of the prepared compost for removing phenolic compounds under flow conditions should be compared with other materials. Table 7 summarizes retention capacity and optimum operational conditions of different adsorbents for removing phenol flow conditions.

Table 7 Performance of compost against other adsorbents for removing phenol under continuous flow conditions

Adsorbent	Operational Factors			Bed capacity (mg/g) ^a	Reusability	Service time (hr)	Prod. Cost ^c	Reference
	Bed Depth (cm)	Flow Rate (mL/min)	Inlet Conc. (mg/L)					
Pb-magnetic-AC	5.0	20.0	300.0	114.0	4 cycle (80%)	10.0	High	50
Commercial-AC	5.0	20.0	300.0	102.6	NR ^b	6.7	High	50
Commercial-AC	11.0	2.0	180.0	94.5	NR	30.0	High	51
Corn cobs-AC	12.0 g	18.3	500.0	68.0	NR	3.3	Moderate	52
Rice husk-AC	8.5	10.0	200.0	45.8	3 cycle (75%)	3.0	Moderate	53
Garlic peel	15.0	15.0	150.0	33.5	NR	5.8	Low	54
Commercial-AC	25.0	NR	55.0	32.4	NR	150.0	High	55
Calcium alginate- citrus peel	3.5	10.0	100.0	20.9	NR	6.7	Moderate	56
Rice husk-AC	9.0	10.0	100.0	14.6	NR	20.0	Moderate	57
Rice straw-biochar	6.0	2.0	300.0	9.1	8 cycle (90%)	20.0	Moderate	12
Corn cob-AC	9.0	10.0	100.0	8.6	NR	20.0	Moderate	58
Silica aerogel	1.6 g	2.0	50.0	6.6	3 cycle (50%)	2.3	Low	59
Areca nut waste-AC	NR	0.5	7.89	2.7	NR	17.0	Moderate	60
Compost	15.0	1.0	150.0	30.0	4 cycle (80%)	9.0	Low	This work

a. Measured at the exhaustion point.

b. Not reported

c. The production cost of adsorbents was tentatively provided as reported in the recent literature [49,61].

The performance of the adsorbents for removing phenol under flow conditions can be assessed through different aspects, including bed capacity, service time, reusability, and production cost. As summarized in Table 7, compost with an uptake capacity of 30.0 mg/g outperformed many

nonconventional and synthetic adsorbents, including calcium alginate-citrus peel composite (20.9 mg/g), activated carbon derived from rice husk (14.6 mg/g), and biochar derived from rice straw (9.1 mg/g). In the meantime, the prepared compost in this work showed a comparable adsorption capacity for phenol to those reported for commercial activated carbon (32.4 mg/g) and garlic peel (33.5 mg/g). Interestingly, few adsorbents, including activated carbon derived from corn cobs, silica aerogel, and activated carbon derived from areca nut waste, exhibited poor uptake capacity for phenol under flow conditions with values of 8.6, 6.6, and 2.7 mg/g, respectively. In fact, the earlier adsorbents may not be suitable for treating real wastewater. Among adsorbents, Pb-magnetic-AC composite and two commercial activated carbons were the most effective for capturing phenol under flow conditions with uptake capacity of 94-114.0 mg/g; however, their high production cost would retard their application on a large scale. Another important criterion to be considered while assessing adsorbents is their reusability for phenol adsorption. In this regard, activated carbon derived from rice husk is a good candidate for removing phenol under flow conditions with an uptake capacity of 45.8 mg/g, moderate production cost, and use for three adsorption-desorption cycles with a final efficiency of 75%. Although some commercial activated carbons have a high capacity for removing phenol under flow conditions, 32.4-94.5 mg/g, their long service time, 30.0-150.0 hr, may retard their utilization for real applications. The prepared compost is a suitable candidate for capturing phenol under flow conditions with a bed capacity of 30.0 mg/g, moderate service time, and low production cost and is used for four adsorption-desorption cycles with a final efficiency of 80%.

5 Conclusion

Composting was effectively used for preparing active bioadsorbent for removing phenol and 2-Nitrophenol under fixed-bed conditions. Characterization of the prepared compost indicated its stability, maturity, and safe application as a bioadsorbent. With a few experiments, central composite design and response surface methodology were able to define the optimum combination of experimental factors to maximize phenolic compound adsorption under flow conditions. Analysis indicates that bed depth and inlet concentration have high linear effects on pollutant uptake under flow conditions, while flow rate has a linear negative effect on the process. A high and negative interaction effect between bed depth and flow rate on pollutant uptake was predicted; that is, bed depth should be high while flow rate should be low during the treatment process. Based on CCD-RSM analysis, the removal process of both pollutants by compost can be maximized at a bed depth higher than 10.0 cm, a flow rate lower than 1.0 mL/min, and an inlet concentration higher than 100.0 mg/L. At the optimum combination of operational factors, the maximum adsorption capacity

of phenol and 2-nitrophenol as predicted by the Thomas model was 29.5 and 43.4 mg/g, respectively. Interestingly, the prepared bioadsorbent was reusable for removing both pollutants up to four adsorption-desorption cycles using cheap eluent.

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Data Availability

The data that support the findings of this research is available upon request

Declarations

Competing Interests

The authors declare no competing interests.

Ethical approval

Not applicable

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