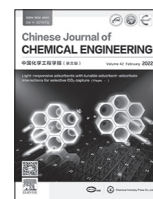




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Evaluation of phenanthrene removal from soil washing effluent by activated carbon adsorption using response surface methodology

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ABSTRACT

Soil washing is a promising technology for the remediation of polycyclic aromatic hydrocarbons (PAH)-contaminated sites, but surfactant needs to be recovered to reduce remediation cost and avoid secondary pollution. In this study, activated carbon (AC), prepared from waste walnut shells, was applied to the adsorptive removal of phenanthrene (PHE) from synthetic soil washing effluent with Tween 80 as a model surfactant. Box-Behnken statistical experiment design (BBD) and response surface methodology (RSM) were used to investigate the influence of AC dosage, Tween 80 concentration and adsorption time, and their potential interaction effect on PHE removal. A response surface model was established based on the BBD experimental results. The goodness of fit of the model was confirmed by determination coefficient, coefficient of variation (CV) and residuals analysis. The RSM model indicates that AC dosage or adsorption time had positive effect on PHE removal while Tween 80 concentration had negative effect. The interaction effect between AC dosage and Tween 80 concentration was significant but the other two interaction effects were not. The 3D response surface plots were developed based on the RSM equation. The RSM model was validated by an additional experiment and the obtained result of PHE removal was very close to the model prediction, indicating the RSM model can effectively predict the PHE removal from soil washing effluent with activated carbon adsorption.

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1. Introduction

Polycyclic aromatic hydrocarbons (PAHs) are widespread in the natural environment. With continuous developments of urbanization and industrialization in China, more and more PAHs are released mainly from coal combustion, coking coal, oil burning and natural gas combustion [1]. In China, it was reported that 16 kinds of PAHs emission increased from 80,000 tons in 2000 to 104,360 tons in 2012 [1,2]. PAHs have the low solubility in water, low volatility and high octanol–water partition coefficients (K_{ow}) [3], which results in their strong adsorption and long residence in soil. Identified as mutagenic, carcinogenic and genotoxic [4] to human beings and other living things in the ecosystem, PAHs are listed as priority pollutants in 1979 by the US Environmental Protection Agency (USEPA) that must be under regulation [5]. The ubiquitous PAHs cause great harm to human beings and ecosystem

by food chains, which make the remediation of PAH-contaminated soil very urgent.

Soil washing is a promising technology to remediate PAH-contaminated sites by making use of surfactant solution to enhance solubility of PAHs and form oil/water (O/W) emulsions [6–9]. The surfactants widely used in soil washing are Tween 80, Triton X-100 and sodium lauryl sulfonate (SDS), etc. [10]. The extraction of PAHs from soils results in the generation of soil washing effluents containing surfactant and PAHs. Direct discharge of the effluents will cause contamination to environment. In the meantime, surfactant consumption is a major part of total operating expense [11]. Therefore, it is necessary to recycle surfactant to reduce soil remediation cost. Selective adsorption has proved to be an excellent method to deal with soil washing effluents [12–16]. Activated carbon (AC) is emerging as environmentally-friendly and efficient adsorbent due to its high surface area and relatively lower cost [17]. However, compared with its extensive application in wastewater treatment, there are fewer reports on the AC selectively adsorptive removal of PAHs from soil washing effluent. In addition, previous work mainly focused on the treatment of soil

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washing effluent containing phenanthrene (PHE) and surfactant such as TX-100 or SDS [18–21]. The removal of PHE from soil washing solution with Tween 80 as an extraction agent was scarcely investigated, and only 40.5% of PHE was adsorbed by Aldrich charcoal-based AC [14]. More importantly, traditional one-factor-at-a-time approach was usually employed to investigate the effect of operating parameters on the AC selective adsorption process. It is well known that this approach is time-consuming and cannot evaluate the interactions between the operating parameters [22,23]. As a result, high efficient experimental studies, which are based on experimental design and quantitative assessment methods, are proposed [24]. Response surface methodology (RSM), an experimental design technique which mathematically fits the experimental domain studied in the theoretical design through a response function [25–28], can overcome the drawbacks of one-factor-at-a-time approach. Box-Behnken design (BBD), known as a modified central composite experimental design, is a widely used RSM analysis method for three-level factorial design [29,30]. It is an independent quadratic design which does not contain an embedded factorial, and consequently, the parameter combinations are at the center and at the midpoints of edges of the process space. Therefore, the number of experiments is considerably reduced [31]. The BBD requires three levels of each factor, which possesses a limited capability for orthogonal blocking in comparison with central composite designs (CCD) [31]. To the best of our knowledge, there are few reports on the adsorptive removal of PAHs from soil washing effluent using BBD analysis. Therefore, in this study, PHE was selected as a typical PAH while Tween 80 as a model nonionic surfactant to simulate soil washing effluent. Meanwhile, BBD was applied to investigate the influence of AC dosage, Tween 80 concentration and adsorption time, as well as their potential interaction effect on PHE removal from synthetic soil washing effluent.

2. Materials and Methods

2.1. Materials

Phenanthrene was supplied by Sun Chemical Technology (Shanghai) Co. Ltd and Tween 80, phosphoric acid (H_3PO_4), hydrochloric acid (HCl) and acetonitrile (CH_3CN) were supplied by Sinopharm Chemical Reagent Co. Ltd. (Shanghai, China). All chemicals were used without further purification. AC was prepared with walnut shells according to our previous study [13]. The morphology of the fabricated AC was observed using a Zeiss SIGMA field-emission scanning electron microscopy (FESEM) and it is irregular with a clear pore structure (Figure S1, Supplementary Material). The specific surface area of prepared AC was determined by nitrogen adsorption at 77 K according to the Brunauer-Emmett-Teller (BET) method on an ASAP 2020 analyzer. The specific surface area was $410.84 \text{ m}^2 \cdot \text{g}^{-1}$, pore volume was $0.61 \text{ cm}^3 \cdot \text{g}^{-1}$ and average pore diameter was 5.92 nm.

2.2. Batch sorption experiments

A synthetic soil washing effluent was prepared by dissolving PHE and Tween 80 in deionized water. The 100 mL solution containing PHE ($20 \text{ mg} \cdot \text{L}^{-1}$) and Tween 80 (1.0, 10.5 and $20.0 \text{ g} \cdot \text{L}^{-1}$) was put into a 250 mL conical flask. After adding the required amount of AC ($0.1, 0.3$ and $0.5 \text{ g} \cdot \text{L}^{-1}$), adsorption experiments were performed in a series of conical flask placed on an orbital shaker ($200 \text{ r} \cdot \text{min}^{-1}$) at room temperature of $(25 \pm 1)^\circ\text{C}$. At certain time, samples were taken with a syringe and filtered through a $0.45 \mu\text{m}$ nylon filter for the analysis of PHE. The concentrations of PHE were

determined with a Shimadzu LC-20AB high-performance liquid chromatography (HPLC) fitted with a diode array detector (SPD-M20A) and a Shimadzu Shim-pack VP-ODS column ($4.6 \text{ mm} \times 150 \text{ mm}$, $5 \mu\text{m}$) using acetonitrile/water (v/v, 70/30) as the mobile phase at a flow rate of $1 \text{ mL} \cdot \text{min}^{-1}$ at a wavelength of 254 nm.

The removal percentage of PHE was calculated from Eq. (1).

$$\text{PHE removal} = \frac{[\text{PHE}]_0 - [\text{PHE}]_t}{[\text{PHE}]_0} \times 100\% \quad (1)$$

where $[\text{PHE}]_0$ ($\text{mg} \cdot \text{L}^{-1}$) is the initial concentration of PHE and $[\text{PHE}]_t$ ($\text{mg} \cdot \text{L}^{-1}$) is the concentration of PHE at time t .

2.3. Design of experiments

RSM was performed using BBD method to investigate the effects of three independent variables on response factor. The three variables considered in this study were AC dosage (A), Tween 80 concentration (B) and adsorption time (C). The low, central and high levels of each variable are designed as $-1, 0$ and $+1$, respectively, as shown in Table 1. PHE removal (Y or y) was chosen as the response parameter. The operational parameters for each run are presented in Table 2, and three runs were conducted at the center point of each variable to check the reproducibility of the results.

By using Design Expert software, the experimental data from BBD were fitted to a second-order polynomial model using Eq. (2) [32].

$$Y = \beta_0 + \sum_{i=1}^k \beta_i X_i + \sum_{i=1}^k \beta_{ii} X_i^2 + \sum_{i=1}^{k-1} \sum_{j=i+1}^k \beta_{ij} X_i X_j \quad (2)$$

where Y is the response, X_i and X_j are variables, β_0 is a constant coefficient, β_i , β_{ii} and β_{ij} are interaction coefficients of linear, quadratic and the second order terms, respectively, and k is the number of studied variables.

The relationship between coded and actual values can be expressed by Eq. (3) [33].

$$X_i = \frac{x_i - x_0}{\Delta x_i} \quad (3)$$

where X_i is dimensionless coded value of the i th independent variable, x_i is the uncoded value of the i th independent variable, x_0 is the actual i th independent variable at the center point, and Δx_i is the step change value between low level (-1) and high level ($+1$).

3. Results and Discussion

3.1. Main variables

Experimental design and the results of response parameter are listed in Table 2. Based on the results, the main effects plot for PHE removal was obtained and illustrated in Fig. 1. As can be seen, AC dosage and adsorption time had positive effects on PHE removal while Tween 80 concentration had negative effect. In addition, the slope of the plot was indicative of importance of the variable on PHE removal and it was obvious that the effect of AC dosage and Tween 80 concentration were more important than that of adsorption time [34]. It should be noted that Fig. 1 only expressed the main effects of three variables on response factor and the straight lines did not mean linear relationships between the variable change and PHE removal.

3.2. Interaction effects

Fig. 2 shows the interaction effects between the variables. As can be seen, the influence of variable A on PHE removal at high

Table 1
Independent variables of the three-level Box-Behnken design

Variables	Symbol	Level		
		−1	0	+1
A: AC dosage/g·L ^{−1}	X ₁	0.1	0.3	0.5
B: Tween 80 concentration/g·L ^{−1}	X ₂	1.0	10.5	20.0
C: adsorption time/h	X ₃	0.5	2.0	3.5

Table 2
Design matrix in actual and coded units and the experimental responses

Run No.	Independent variables (coded)			Responses Y (PHE removal)/%
	X ₁ (AC dosage)/g·L ^{−1}	X ₂ (Tween 80 concentration)/g·L ^{−1}	X ₃ (adsorption time)/h	
1	0.1 (−1)	1.0 (−1)	2.0 (0)	83.79
2	0.5 (+1)	1.0 (−1)	2.0 (0)	98.53
3	0.1 (−1)	20.0 (+1)	2.0 (0)	21.35
4	0.5 (+1)	20.0 (+1)	2.0 (0)	80.83
5	0.1 (−1)	10.5 (0)	0.5 (−1)	25.20
6	0.5 (+1)	10.5 (0)	0.5 (−1)	73.43
7	0.1 (−1)	10.5 (0)	3.5 (+1)	37.48
8	0.5 (+1)	10.5 (0)	3.5 (+1)	91.43
9	0.3 (0)	1.0 (−1)	0.5 (−1)	88.87
10	0.3 (0)	20.0 (+1)	0.5 (−1)	45.31
11	0.3 (0)	1.0 (−1)	3.5 (+1)	97.12
12	0.3 (0)	20.0 (+1)	3.5 (+1)	66.71
13	0.3 (0)	10.5 (0)	2.0 (0)	74.05
14	0.3 (0)	10.5 (0)	2.0 (0)	72.59
15	0.3 (0)	10.5 (0)	2.0 (0)	72.90

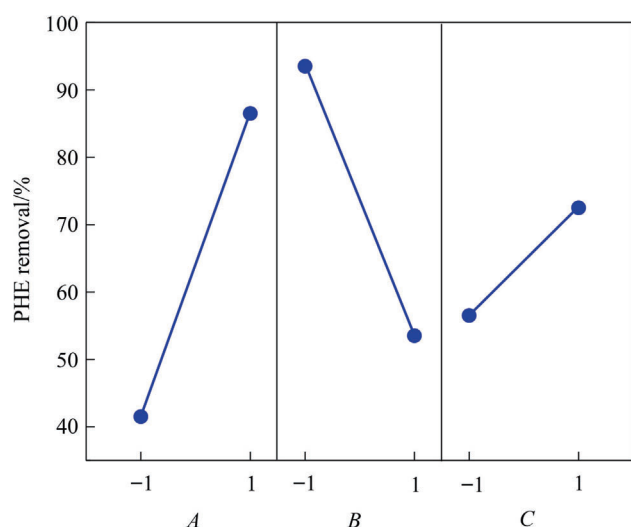


Fig. 1. Main effects plot for PHE removal (A: AC dosage; B: Tween 80 concentration; C: adsorption time).

level of variable B (B⁺) is different from that at low level of variable B (B[−]). In other word, the plots are tending to across, which means the interaction between variable A (AC dosage) and variable B (Tween 80 concentration) is significant [29,35–37]. However, the effect of variable A on PHE removal is almost independent of variable C, and the plots are nearly parallel as illustrated in Fig. 2. It indicates there is insignificant interaction between variable A (AC dosage) and variable C (adsorption time) [37,38]. The similar result was observed between variable B (Tween 80 concentration) and variable C (adsorption time). This could be justified by the higher probability values (0.56 for X₁X₃ and 0.21 for X₂X₃) based on ANOVA analysis. Since the both values are higher than 0.05, the two interaction items (X₁X₃ and X₂X₃) would be removed from the RSM model.

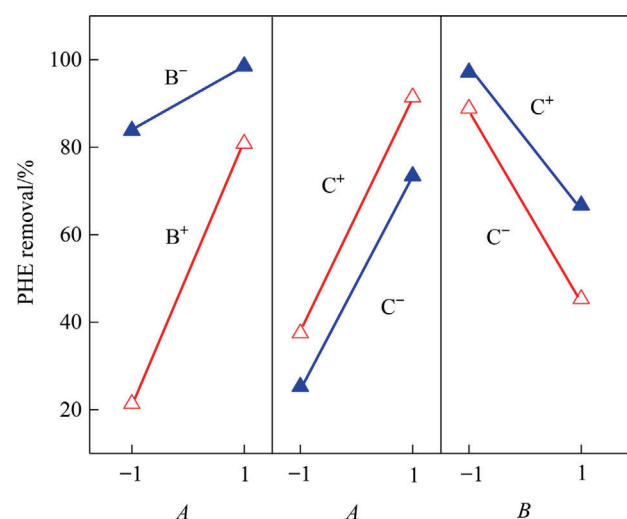


Fig. 2. Interaction plots for PHE removal (A: AC dosage; B: Tween 80 concentration; C: adsorption time).

3.3. RSM model

The RSM model excluding the insignificant variables (X₁X₃ and X₂X₃) was determined in terms of coded variables and shown in Eq. (4).

$$Y = 73.18 + 22.05X_1 - 19.26X_2 + 7.49X_3 + 11.19X_1X_2 - 9.84X_1^2 + 7.78X_2^2 - 6.46X_3^2 \quad (4)$$

Accordingly, the RSM model equation in terms of actual (uncoded) variables was given in Eq. (5).

$$y = 45.84 + 195.98x_1 - 5.60x_2 + 16.48x_3 + 5.89x_1x_2 - 245.91x_1^2 + 0.09x_2^2 - 2.87x_3^2 \quad (5)$$

The significance of the RSM model was justified via ANOVA analysis. As shown in Table S1 (Supplementary Material), the F -value of the model was 55.34 and the (Prob > F) value was less than 0.0001, indicating the model was significant. According to the RSM equation, the order of importance on PHE removal of each influencing variables is $X_1 > X_2 > X_3$ [34]. The fitness of the model was also checked by the determination coefficient (R^2), which was 0.9823 in this study, meaning only 1.77% of the variability for PHE removal could not be explained by the model. Besides, the value of adjusted determination coefficient ($R^2_{adj} = 0.9645$) was close to R^2 , further confirming a high significance of the model [23,39]. In addition, the value of predicted determination coefficient (R^2_{pred}) was 0.8589, which was in reasonable agreement with R^2_{adj} . The difference between R^2_{pred} and R^2_{adj} was less than 0.2, which was also a support for a high significance of the model.

The signal to noise ratio was obtained to be 24.04, which was greater than 4, indicating an adequate precision signal. So the model could be used to navigate the design space [29,40]. The coefficient of variation (CV) was 6.86%, which was within the acceptable range (0.5%–13.5%), giving a great reproducibility of this model [33,41].

3.4. Adequacy of the model

The normality of the data could be checked by constructing a normal probability plot of the residuals as shown in Fig. S2 (Supplementary Material). As can be seen, the points on the plot followed a straight line indicates that the residuals were normally distributed [22], and the normality assumption was confirmed. Fig. 3 shows the relationship between the externally studentized residuals and the predicted values of PHE removal, and the residuals appeared to be a random scatter. It suggested that the equality of variance did not seem to be violated [42,43]. The plot of externally studentized residuals versus run number (Fig. 4) was observed to fall in the range of -3.0 to $+3.0$, which supports that the model presented a minimal deviation and the difference had normal distribution [44]. Fig. S3 shows Box-Cox plot for power transformations and it was concluded that the data did not require any transformation [45]. The natural logarithm ($\ln$) of the residuals sum of square (SS) against λ was found to be 1. The best λ was found to be 1.36 and the minimum and maximum confidence interval values were found to be 0.31 and 2.82, respectively.

To calibrate the RSM model, the comparison between the predicted and measured PHE removal was made and illustrated in Fig. 5. The result confirms that the predicted values are in agreement with the observed ones. It indicates there was insignificant violation of the model, and the RSM model was satisfactory and accurate. To furthermore validate the RSM model, an additional separate experiment was performed under the conditions randomly selected as $0.5 \text{ g} \cdot \text{L}^{-1}$ AC dosage, $5 \text{ g} \cdot \text{L}^{-1}$ Tween 80 concentration and 3 h adsorption time. A 94.8% of PHE removal was achieved after the experiment, which was very close to the predicted value of 94.2% calculated by the model equation (Eq. (5)) based on the same conditions. Then the RSM model was verified accordingly.

3.5. Response surface plots

The response surface plots were developed based on Eq. (5) and the variation of PHE removal with three variables (AC dosage, Tween 80 concentration and adsorption time) is shown in Fig. 6. As can be seen from Fig. 6(a) and (b), increasing AC dosage could significantly improve the PHE removal rate in the effluents. This is because higher AC dosage meant more adsorption sites for PHE removal. However, increasing Tween 80 concentration would

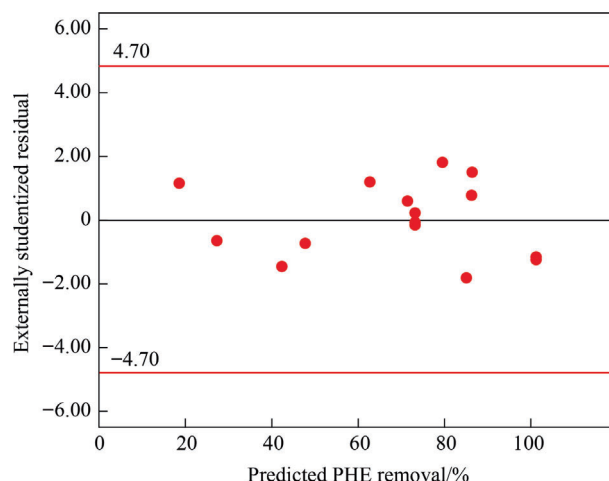


Fig. 3. Residuals versus predicted plot for PHE removal.

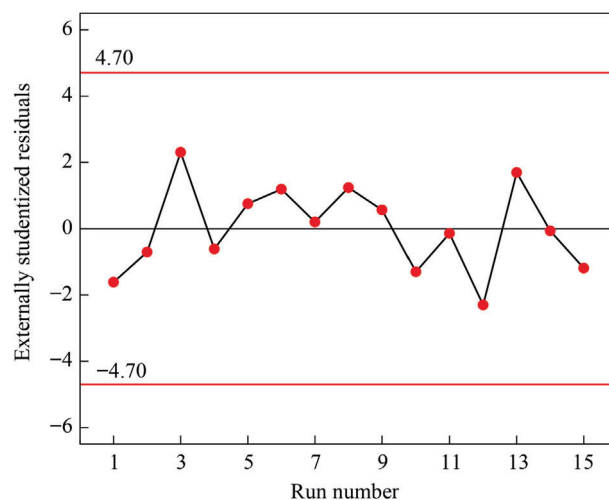


Fig. 4. Plot of externally studentized residuals versus run number.

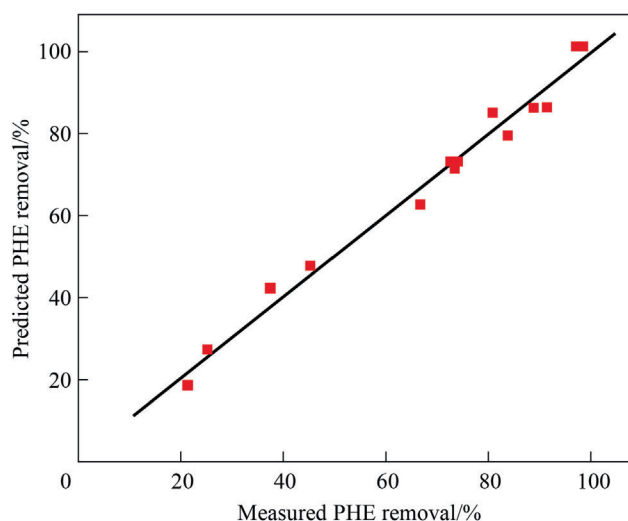


Fig. 5. Predicted versus measured values of PHE removal.

decrease PHE removal (Fig. 6(a) and (c)), since higher concentration of Tween 80 will form more micelles, which can increase the capacity of PHE solubilization and obstruct the removal of PHE

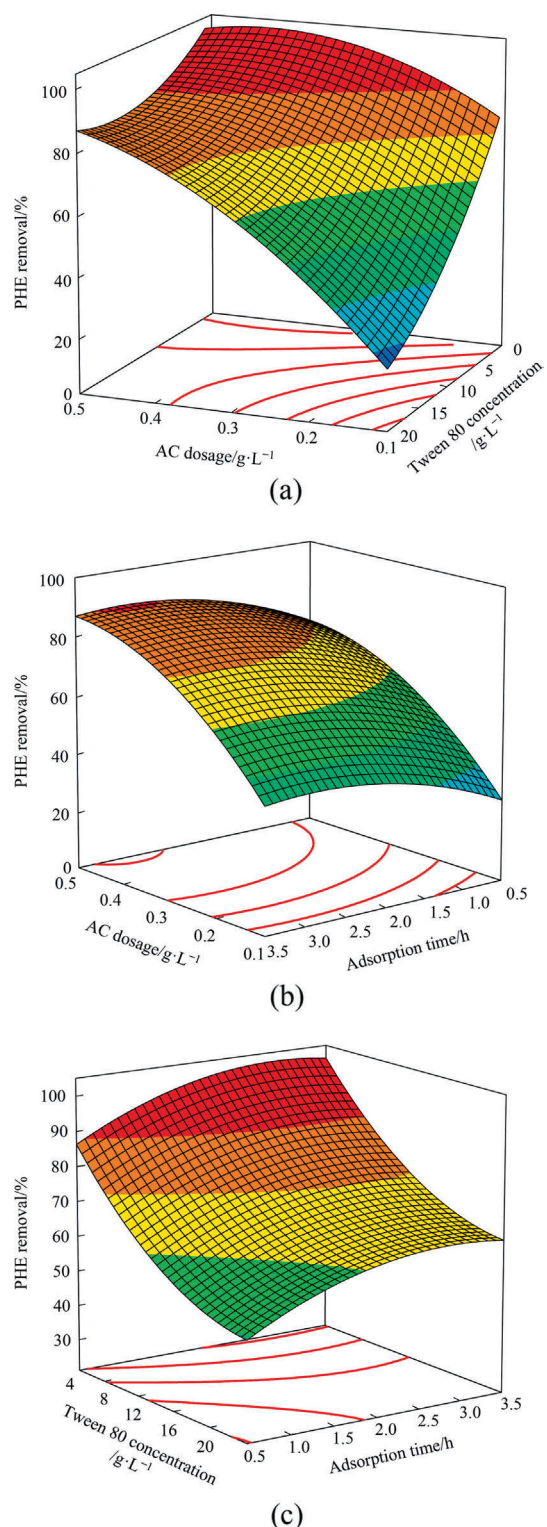


Fig. 6. Response surface showing PHE removal as a function of two independent variables (the fixed values of other variables are center level: AC dosage: $0.5 \text{ g}\cdot\text{L}^{-1}$, adsorption time: $10.5 \text{ g}\cdot\text{L}^{-1}$, adsorption time: 2 h). (a) AC dosage and Tween 80 concentration, (b) AC dosage and adsorption time, (c) Tween 80 concentration and adsorption time.

[13]. Increasing adsorption time will also benefit for PHE removal, as shown in Fig. 6(b) and (c). This is because longer time means that the micelles can have longer contact time with AC, which can allow more sufficient adsorption for PHE removal.

Table 3

Comparison of PHE removal in different processes

Process	Operating parameters	PHE removal efficiency	Reference
Adsorption	$[\text{PHE}]_0 = 14.48 \text{ mg}\cdot\text{L}^{-1}$, $[\text{Tween } 80] = 1.884 \text{ g}\cdot\text{L}^{-1}$, $[\text{AC}] = 1.0 \text{ g}\cdot\text{L}^{-1}$, 48 h	40.2%	[14]
Adsorption	$[\text{PHE}]_0 = 20 \text{ mg}\cdot\text{L}^{-1}$, $[\text{TX-100}] = 4 \text{ g}\cdot\text{L}^{-1}$, $[\text{bentonite}] = 1 \text{ g}\cdot\text{L}^{-1}$, 24 h	96.7%	[15]
Adsorption	$[\text{PHE}]_0 = 20 \text{ mg}\cdot\text{L}^{-1}$, $[\text{SDS}] = 5 \text{ g}\cdot\text{L}^{-1}$, $[\text{resin SP850}] = 1 \text{ g}\cdot\text{L}^{-1}$, 2 h	98.6%	[16]
Biodegradation	$[\text{PHE}]_0 = 8.26 \text{ mg}\cdot\text{L}^{-1}$, $[\text{Tween } 80] = 2.5 \text{ g}\cdot\text{L}^{-1}$, $[\text{K}_2\text{HPO}_4] = 6.3 \text{ g}\cdot\text{L}^{-1}$, $[\text{KH}_2\text{PO}_4] = 1.8 \text{ g}\cdot\text{L}^{-1}$, $[\text{NH}_4\text{NO}_3] = 1 \text{ g}\cdot\text{L}^{-1}$, bacterial consortium ($\text{OD}_{600\text{nm}} = 1$), 7 days	~100%	[3]
Electro-Fenton	$[\text{PHE}]_0 = 18 \text{ mg}\cdot\text{L}^{-1}$, $[\text{HPCD}] = 4 \text{ g}\cdot\text{L}^{-1}$, $[\text{Fe}^{2+}] = 0.05 \text{ mmol}\cdot\text{L}^{-1}$, $I = 2000 \text{ mA}$, $[\text{Na}_2\text{SO}_4] = 0.150 \text{ mol}\cdot\text{L}^{-1}$, Pt anode, carbon-felt cathode, pH_0 3.0, 4 h	100%	[46]
UV/PDS	$[\text{PHE}]_0 = 10 \text{ mg}\cdot\text{L}^{-1}$, $[\text{SDS}] = 10 \text{ g}\cdot\text{L}^{-1}$, $[\text{PDS}] = 2 \text{ mmol}\cdot\text{L}^{-1}$, pH_0 8.6, UV power = 6 W, 8 min	98.7%	[47]
Adsorption	$[\text{PHE}]_0 = 20 \text{ mg}\cdot\text{L}^{-1}$, $[\text{Tween } 80] = 5 \text{ g}\cdot\text{L}^{-1}$, $[\text{AC}] = 0.5 \text{ g}\cdot\text{L}^{-1}$, 3 h	94.8%	This work

Abbreviation: TX-100—Triton X-100; HPCD—hydroxypropyl-beta-cyclodextrin; PDS—persulfate.

3.6. Comparison of PHE removal from soil washing effluent by different processes

The comparison of this work with the adsorption using AC as well as other adsorbents was made firstly. As can be seen in Table 3 the removal of PHE achieved 94.8% in a 3-h adsorption in this study, while only 40.5% of PHE could be adsorbed from soil washing solution containing the same surfactant (Tween 80) by Aldrich charcoal-based AC [14]. Although a slightly higher removal efficiency of PHE was obtained using bentonite (96.7%) or resin SP850 (98.6%) as a adsorbent, a longer adsorption time and/or a doubled dosage of adsorbent were needed [15,16]. This indicates the AC fabricated from walnut wall has a greater adsorption capacity than bentonite and resin SP850. This study was also compared with other processes such as biodegradation and advanced oxidation processes (AOPs). Table 3 indicates that biodegradation could remove PHE, but it took 7 days to achieve a nearly complete removal of PHE [3]. AOPs such as electro-Fenton and UV/PDS processes are superior to AC adsorption [46,47]. It is because in these AOPs, the additional energy was applied and the introduction of oxidants gave rise to the generation of reactive radicals with greater oxidization ability.

4. Conclusions

Box-Behnken statistical experiment design was proven to be a suitable response surface methodology to determine the effects of operative variables (AC dosage, Tween 80 concentration and adsorption time) and their interactions on PHE removal from soil washing effluent. The order of three variables on PHE removal was AC dosage > Tween 80 concentration > adsorption time. The analysis of variance (ANOVA) indicated the interaction effect on PHE removal between AC dosage and Tween 80 concentration

was statistically significant compared with that between AC dosage and adsorption time or between Tween 80 concentration and adsorption time. The significance and adequacy of the obtained RSM model was confirmed by the various determination coefficients, diagnostics with normal probability plot of residuals, residuals versus predicted plot, predicted versus actual values plot, plot of externally studentized residuals versus run number and Box-Cox plot for power transformations. Moreover, a 94.8% of PHE removal was observed at 0.5 g·L⁻¹ of AC dosage and 5 g·L⁻¹ of Tween 80 in a 3-h adsorption, which is similar to the predicted value (94.2%) obtained via model equation, and further verified the accuracy of the RSM model. The 3D response surface plots illustrate the PHE removal boosted with the increase of AC dosage and adsorption time, while declined with the increase of Tween 80 concentration.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary Material

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.cjche.2021.02.027>.

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