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Response Surface Methodology: A Review on Optimization of Adsorption Studies

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Authors' contributions

This work was carried out in collaboration among all authors. Authors SB, MK, JN, IK, and CTA designed the study, performed the statistical analysis, wrote the protocol, and wrote the first draft of the manuscript. Authors HKN, IK and MM managed the analyses of the study. Authors JS and RAN managed the literature searches. All authors read and approved the final manuscript.

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ABSTRACT

Herein, we reviewed response surface methodology (RSM), a powerful statistical tool widely used in optimizing adsorption processes to remove synthetic dyes, toxic heavy metals, and phenols from wastewater. The widely used RSM models during optimization are the central composite design and Box-Behnken, which are second-order polynomial models. The models give a predictive insight into the number of experimental runs to be carried out. Furthermore, an in-depth overview of RSM and its application in optimizing various adsorption parameters, such as adsorbent dosage, initial pollutant concentration, contact time, and pH is discussed. In addition, RSM enables researchers to efficiently determine the optimal conditions for maximum pollutant removal. Lastly, the findings of this research highlight the potential of RSM as a valuable tool for optimizing adsorption processes and contributing to sustainable water treatment technologies.

Keywords: Response surface methodology; phenols; toxic heavy metals; wastewater; adsorption.

1. INTRODUCTION

Currently to get the most benefits from a process and achieve maximum output, optimization of the process conditions is required. Optimization has been defined as the choosing of factors from a collection of potential independent variables (Usman et al. 2021). We need to understand the product and process conditions and parameters required to develop a good model design. The design needs a good understanding of the entire process to give a better prediction. The design choice might be determined by considering the factorial designs based on the dependent and independent variables. Upon analysis, we can determine the variables that give the maximum response for a given set of data. The commonly used optimization tool is response surface methodology (RSM). RSM is a mathematical and statistical which uses a second-degree polynomial model through the application of the univariate and multivariate models to study the relationship between two or more response variables and several independent variables (Alzorqi et al. 2016, Vivek et al. 2016). The multivariate approach is widely used since it compares interactions between different factors, unlike the univariate which does not compare interactions and is time-consuming (Reji 2022). Based on the input variables the optimum conditions can be determined through the maximum and minimum response within the region of interest. The choice of RSM approach to be used for a given design experiment depends on the experiment treatments for a given data set (Yolmeh et al. 2017). Regression analysis during optimization predicts the response but also optimizes the process parameters (Kaur and Kaur 2013). It is also observed that RSM provides better process output and reproducibility of results. RSM is a tool used for the design, analysis, and process optimization of experimental data (Lamidi et al. 2022).

A second-order polynomial regression is used to predict the response based on the input parameters as shown in Equation 1.

$$S = a_o + \sum_{i=1}^{k} a_i x_i + \sum_{i=1}^{k} a_{ii} x^2 + \sum_{ij=1\ j \neq i} a_{ij} x_i x_j \qquad (1)$$

Where S is a response, a_o is the average of responses, a_i , a_{ii} and a_{ij} are response coefficients. The linear, high-order and interaction effects are described by the second, third, and fourth terms respectively.

Additionally, it is observed that the second-order polynomial is flexible and simple to approximate but also predicts the quadratic surfaces such as the ridge, maximum, minimum, stationary, and saddle point (Lamidi et al. 2022). The response can be determined graphically using either two-dimensional contour plots, which display the response values, or three-dimensional space, which describes the independent variable interaction effects (Jensen 2017).

RSM has found wide application in water treatment for the optimization of parameters during the removal of a wide range of pollutants such as textile dyes, pharmaceuticals, phenols, toxic heavy metals and pollutants of emerging concern (Alardhi et ql. 2024, Zahmatkesh et al. 2024, Birniwa et al. 2024, Bashir et al. 2012, Karume et al. 2023). This study is intended to look at the optimization of some of the synthetic dyes, toxic heavy metals, and phenols.

2. MAIN MODELS IN RESPONSE SURFACE METHODOLOGY

This is a technique that relates the process parameters and the output to identify the design variables capable of enabling further experimental investigation. The Design of experiments (DOE) is divided into first and second order designs with the former comprising 2^k factorial, Plackett-Burman and simplex while the later is divided into 3^k, Box Behnken and central composite design (CCD).

The 2^k factorial design which is a first order design which evaluates variables at two levels that correlate at the low and high parameters and programmed at values of -1 or 1. It is observed that this design is employed when the interactions and particular effects vary linearly in the direction of interest.

Plackett-Burman another first-order design also depends on two levels but with fewer experimental runs corresponding with particular k variables which are characteristic of its size.

It is assumed to be a saturated model design as the number of approximated variables is equal to the number of design points.

The last first-order model called the simplex is described by a k-dimensional figure whose design points at the vertices have two points formed at an angle and a cos =$^-$1/k with sum points of n k+1 as the design center.

In addition, the CCD which is the widely applied second-order model has a center point which is known as the design space, axial points which are coordinate and symmetrically arranged systems with respect to the central point and the factorial points illustrated as -1, +1.

Another second-order model is the 3^k factorial design which consists of three levels embeds the permutations of control in the k variables. It is observed that in this design the number of trial runs is 3^k which is considerably large for any given k value. It is a cost-efficient model as the cost of the trial runs can be reduced using the 3^kfactorial design.

The last second-order model is a 3^k factorial design having a particular subset of factorial combinations defined by three levels of each factor. During optimization, different parameters can be carried out simultaneously while the other parameters are not varied and the first factors analyzed. Furthermore, the Box Behnken consists of 1,0,1 configuration which necessitates the use of only three levels of each element and as such has found application in the industrial sector as it is a of low cost.

3. COMPONENTS OF RESPONSE SURFACE METHODOLOGY

The model through the design of experiments (DOE) describes the probable points where the response to be determined is to be found.

It also shows the experimental runs that should be performed for a particular study (Myers et al. 2023).

RSM also helps to interpret the relationship between the dependent (responses) and the independent variables (factors) for a given experiment (Bezerra et al. 2008).

It further gives the factorial values for which an experiment is to be carried out but also the lower and upper limits of the independent variables for a given experimental domain (Bezerra et al. 2008, Belmir et al. 2021, Ranade 2017).

In addition, the experimental design and space have ranges of values for variation in the factors and the specific experiments to be carried out are defined by a matrix containing different level combinations (Cox 2007).

RSM also contains the response surface which illustrates the mean response, residual which correlates the observed and calculated results, interaction which describes the cumulative effects of two or more variables, DOE matrix which is a collection of combination process variables whose effect on the output is paramount (Reji 2022, Belmir et al. 2021, Tetteh and Rathilal 2021, Ghaedi et al 2019).

Lastly, RSM introduces the center point which shows the curvature of the response, controlled experiments where responses are observed on experimental units after treatment and levels of variables that express the different variables at which the experimental runs are to be carried out (Reji 2022, Belmir et al. 2021, Dolatabadi et al. 2022).

4. OPTIMIZATION OF ADSORPTION PROCESS PARAMETERS FOR WASTEWATER TREATMENT USING RESPONSE SURFACE METHODOLOGY

Several research has been carried out on the optimization of adsorption process parameters using RSM during wastewater treatment. Researchers have used RSM in several pollutants such as toxic heavy metals, synthetic dyes, pharmaceuticals, phenols, etc. The optimization is instrumental in the reduction of the experimental runs, is time efficient, and reduces errors during analysis.

Bayuo et al. 2022 investigated the optimization of mercury by adsorption on granular activated carbon that was synthesized from maize plant residues. The CCD and analysis of variance (ANOVA) were studied to understand the influence of the independent factors on the removal efficiency of Hg (II). The factors studied were contact time, adsorbent dosage, and initial concentration through 20 run optimizations. The experimental data showed that the optimal reaction conditions gave a removal percentage of 96.7 %. The quadratic models were designed for the prediction of the response as a function of the independent factors.

Muntean et al. 2023 used the RSM specifically the Box-Behnken factorial design to determine the effect of three variables on the removal of Copper, Lead, and Zinc. Using the polynomial models a highly significant value of ($p > 0.001$) was achieved with correlation coefficients of 0.96 for Zn (II) and 0.99 for both Cu (II) and Pb (II). It was observed that the theoretical and experimental results correlated well which supports the applicability of the method.

Saensook and Sirisuk 2022 investigated the optimization of $NaBH_4$ on TiO_2 using a 2 × 2 × 3 factorial experimental design with two replicates having factors of calcination time, temperature, and molar ratio. The model showed that the titanium dioxide optimized at 500 °C at 10 hours and a molar ratio of 1:1 of TiO_2: $NaBH_4$. The observed removal percentage of UV and visible light for methylene blue was 82.17%.

Kweinor Tetteh et al. 2021 used the Box-Behnken model adopted from response surface methodology to optimize AC-TiO_2 to determine the removal efficiency for turbidity and color using three input variables that is reaction time (15-45 minutes), catalyst load (2-4 g) and pH (6-9). BBD gave a 17-run experiment that showed the interaction effects of the three independent variables. Using a quadratic model the theoretical and experimental data were in good agreement. The analysis of variance showed that turbidity and color had a high significance with correlation coefficients of 0.988 and 0.972 respectively. It was also further observed that the efficiency was positively impacted due to the interaction of pH and time.

Bayuo et al. 2020 discussed the optimization using the central composite design for Cr (VI) adsorption using Arachis hypogea husk based on three independent variables (pH, concentration, and contact time). Through the use of ANOVA, the optimization showed a high significance with a P-value (2.2×10^{-16}), F-value (1832), and correlation coefficient of 0.9985 which was all through the reduced full second-order polynomial model. RSM theoretical data was in agreement with the experimental results and the removal percentage was 90.2%.

Jaafari and Yaghmaeian 2019 investigated the optimization of three parameters including time, concentration, and adsorbent dose on the removal of toxic heavy metals by Chroococcus dispersus algae and used the Box-Behnken design of RSM. The second-order polynomial was used and it confirmed the validity of the model through the use of the regression equation coefficients. The ANOVA described a high coefficient of determination values (R^2) in the range of 0.994-0.995.

Van Thuan et al. 2017 determined the effect of three variables including pH, adsorbent dosage, and toxic heavy metal concentration on adsorption capacity as the response by applying the RSM through the CCD. The maximum adsorption capacity obtained was in the order Cu^{2+} (14.3 mg/g) < Ni^{2+} (27.4 mg/g) < Pb^{2+} (34.5 mg/g) which was well in agreement with the optimization approach which shows the application and reliability of CCD.

Alipour et al. 2020 investigated the optimization of four parameters that is concentration, contact time, pH, and adsorbent dosage on the removal of Lead (Pb) ions by TZFNC as an adsorbent through the application of RSM. The experimental data showed that the efficiency of Pb(II) removal is 99.99 % under the optimum conditions of experimental factors of contact time

(14.5 min), adsorbent dose (40 mg), concentration (60 mg/L), and pH (6.5).

Lebbihi et al. 2024 discussed the optimization of three independent parameters that is time, adsorbent mass, and pH on the removal of a dye through the use of the Box-Behnken model of RSM. The ANOVA was used to study the significance of the model and this resulted in an adsorbent mass of 0.09942 g, pH of 3.4, and removal dye percentage of 90.16%.

Malakootian et al., 2019 used the CCD of RSM to optimize three operational parameters ie pH, adsorbent dose, and MNZ initial concentration. The quadratic model was used it showed a high degree of fit for the obtained data. The ANOVA showed that the predicted optimal adsorption capacity (qe) was 36.897 mg/g and the operational parameters were contact (46.25 min), adsorbent dose (450mg/L), and pH (5.02).

Teixeira et al. 2019 investigated the removal of Metronidazole and Sulfamethoxazole by activated carbon from walnut shell and optimized the three independent variables with a three-level Box-Behnken experimental design of RSM. The second-order polynomial model was used to describe the relationship between the independent variables (sorption capacity) and independent variables (pH, initial concentration, and temperature). Using the quadratic models, it was observed that pH had the highest significance on the removal of both Metronidazole and Sulfamethoxazole.

Norouzi et al. 2020 investigated the statistical optimization of three factors i.e. initial phenol concentration, pH and photocatalyst concentration using a CCD of RSM. The quadratic model was a better fit for relating the three factors on the photocatalytic degradation of phenol. Using the ANOVA a high significance was observed due to the strong interaction between the three factors and the photocatalytic degradation of phenol. From the data, it was observed that photocatalyst concentration influenced the degradation with a percentage of 92.91%.

Mutar and Saleh 2022 discussed the optimization for the removal of Arsenic from water and a CCD was used for this study which formed three levels with three central points. The three optimized parameters were contact time, pH, and bentonite dosage. The quadratic models were also developed and they gave better fits with a correlation factor (R^2) of 0.9998 and a removal percentage of 97.09%.

Dolatabadi et al. 2022 discussed the optimization of operating parameters using response surface methodology under the subcategory of CCD. The ANOVA investigated the interactions, significance of variables, and the quadratic effects. The value of determination coefficient (R^2), Adjusted R^2 (Adj.R^2), and predicted R^2 (Pred.R^2) were 0.9855, 0.9791, and 0.9743, respectively; also, a p-value of $P < 0.0001$, and F-value of 65.91 were obtained. Using the experimental data the removal efficiency was 99.3%.

Enyoh et al. 2022 investigated the optimization of four parameters temperature, concentration, contact time, and pH) on the response (removal efficiency) using a CCD under RSM. The model shows that the optimum reaction conditions for the removal of phenol from aqueous solutions are a concentration of 50 mg/L, pH of 6, temperature of 298 K, and contact time of 77 minutes respectively.

Ghaedi et al. 2019 studied the effect of a adsorbent dosage, pH, time, and concentration on the adsorptive removal of malachite green using a five-level six-factor CCD model through RSM. The quadratic models suggested a good fit between the experimental and predicted data and the removal percentage observed was about 94.26 %. The coefficient of determination (R^2) of 0.9976 and F-value of 2048.92 showed a good significance for the data.

Tetteh and Rathilal 2021 through RSM optimized three parameters (pH, dosage, and magnetic time) on removal of turbidity and color. The adsorbent dosage was the most influential parameter during the adsorption process. The ANOVA tests showed that the quadratic models were significant with P-vales less than 0.05 and a correlation coefficient of 0.95. The model showed the experimental and theoretical data were in agreement as the color and turbidity removal percentages were between 80-95%.

5. CONCLUSION

Response Surface Methodology (RSM) has proven to be a powerful statistical tool for optimizing adsorption processes for the removal of synthetic dyes, toxic heavy metals, and phenols. By systematically varying experimental factors and analyzing the resulting response variables, RSM enables the identification of optimal conditions that maximize adsorption efficiency. This approach offers several advantages over traditional one-factor-at-a-time methods, including reduced experimental effort, increased precision, and a comprehensive understanding of the interaction effects between variables. Through the application of RSM, researchers can gain valuable insights into the underlying mechanisms of adsorption processes, leading to the development of more efficient and cost-effective adsorbent materials. Furthermore, RSM can aid in the scale-up of laboratory-scale experiments to industrial-scale applications, ensuring optimal performance and environmental sustainability. In conclusion, RSM is an indispensable tool for optimizing adsorption studies and has the potential to revolutionize the field of wastewater treatment and environmental remediation. By embracing this methodology, researchers can contribute to the development of innovative solutions for addressing pressing environmental challenges.

DISCLAIMER (ARTIFICIAL INTELLIGENCE)

Author(s) hereby declare that NO generative AI technologies such as Large Language Models (ChatGPT, COPILOT, etc.) and text-to-image generators have been used during the writing or editing of this manuscript.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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