AUIQ Complementary Biological System
ISSN: 3007-973X
Journal homepage:
https://acbs.alayen.edu.iq

Volume 1 | Issue 1 Article 10

Activated Carbon Derived from Rice Husk Biomass using Pyrolysis-assisted Tartaric Acid Activation for Removal of Reactive Orange 16 Dye: Adsorption Modeling using Response Surface Methodology

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Recommended Citation

Abdulhameed, Ahmed Saud; Wu, Ruihong; Reghioua, Abdallah; Mbuvi, Harun M.; Yaseen, Zaher Mundher; and Garba, Zaharaddeen N. (2024), Activated Carbon Derived from Rice Husk Biomass using Pyrolysis-assisted Tartaric Acid Activation for Removal of Reactive Orange 16 Dye: Adsorption Modeling using Response Surface Methodology, *AUIQ Complementary Biological System*: Vol. 1: Iss. 1, 89-95.
DOI: https://doi.org/10.70176/3007-973X.1009
Available at: https://acbs.alayen.edu.iq/journal/vol1/iss1/10

AUIQ COMPLEMENTARY BIOLOGICAL SYSTEM 1 (2024) 89–95

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ORIGINAL STUDY

Activated Carbon Derived from Rice Husk Biomass using Pyrolysis-assisted Tartaric Acid Activation for Removal of Reactive Orange 16 Dye: Adsorption Modeling using Response Surface Methodology

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ABSTRACT

In the current work, rice husk (RH) biomass was pyrolyzed using tartaric acid as an activating agent to produce activated carbon (AC). For 60 min, the procedure was run at 700 °C and 5 L/min of nitrogen (N_2) flow rate. The study used response surface methodology (RSM) and a 3-level Box-Behnken design (BBD) to optimize the extraction efficiency of rice husk activated carbon (RHAC) in eliminating reactive orange 16 (RO16) dye from an aqueous solution. Different process parameters, such as pH (2–12), RHAC dose (0.04–0.14 g), and time (60–240 min), were chosen as part of the optimization method. When the solution's pH was 2, the adsorbent dosage was 0.09 g, and the contact time was 240 min, the maximum amount of dye removal (53%) was achieved. This work demonstrates the ability to use abundant husk biomass to create activated carbon, which can be used as an adsorbent to effectively remove organic dyes.

Keywords: Activated carbon, BBD-RSM, Dye removal, Response surface methodology, Rice husk biomass, Tartaric acid

1. Introduction

Nowadays, dye manufacturing is widely used in many industries, including plastic, textile, beauty products, paper, leather, and pharmaceuticals [1]. The release of these dyes into water and wastewater poses significant dangers to aquatic life by reducing sunlight penetration, which in turn affects photosynthetic activity [2]. In addition, dyes may pose health risks to humans, including conditions such as jaundice, tumors, allergies, and skin irritation [3]. Reactive Orange 16 (RO16) is a reactive azo dye that interacts with wool's cellulose hydroxyl or amino acid moieties as well as comparable artificial fabrics. The ecology, agricultural productivity, and water quality are all negatively impacted by this organic

Received 13 May 2024; accepted 10 July 2024.
Available online 2 August 2024

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https://doi.org/10.70176/3007-973X.1009

pollutant [4]. Hence, it is essential to eliminate dyes from various types of wastewaters to maintain the natural ecosystem and human well-being.

Various techniques have been used to eliminate dyes, including adsorption [5, 6], membrane filtering [7], and photocatalytic destruction [8]. Several of these technologies include drawbacks such as reduced efficiency, elevated operational expenses, production of hazardous compounds, and time-consuming processes [9]. Adsorption is considered one of the most efficient techniques for removing colors since it is ecologically safe, easy to design, and cost-effective [10]. Moreover, the production of noxious chemicals during the adsorption process is reduced in comparison to other approaches.

Activated carbon (AC) is the predominant adsorbent that is extensively used for the purpose of eliminating colours from wastewater [11–13]. Nevertheless, the process of preparing and regenerating activated carbon incurs significant expenses. Consequently, the scientists turned their attention to natural biopolymers, biomass, and waste materials as cost-effective and eco-friendly substances for adsorption [14, 15]. Biomass and agricultural wastes are increasingly being utilized to produce carbonaceous adsorbents due to their numerous advantages [16]. These materials are renewable, ensuring a sustainable supply that does not deplete finite natural resources. Additionally, they are low-cost, providing an economical alternative to synthetic adsorbents. The utilization of biomass and agricultural wastes also offers significant environmental benefits. By converting these wastes into useful adsorbents, we reduce landfill waste and minimize the environmental impact associated with their disposal. Furthermore, carbonaceous adsorbents derived from biomass possess excellent properties such as high surface area, porous structure, and a variety of functional groups [17]. These characteristics enhance their adsorption capabilities, making them effective in removing pollutants from water [18–21]. Chemical activation of biomass is a highly used approach for creating carbonaceous adsorbents that are oxygenated and hydrophilic. These adsorbents include various functional groups such as carboxyl (–COOH) and hydroxyl (–OH) groups.

In the present study, activated carbon (AC) was produced by pyrolyzing rice husks (RH) with tartaric acid as a chemical activator. The selection of tartaric acid as the activating agent for biomass in the present study is ascribed to its non-toxic nature, thus it offers a distinct advantage over some traditional activating agents, which can pose environmental issues. Response surface methodology (RSM) with Box-Behnken design (BBD) was employed to enhance the adsorption factors for the removal of the reactive orange 16 (RO16) dye from an aqueous system, utilizing activated carbon derived from rice husk (RHAC). A statistical and graphical analysis of the BBD model was conducted to determine the optimal levels of the most vital elements. RSM-BBD was used in the studies to enhance the percentage of adsorption. RSM is a set of statistical tools and mathematical approaches that may be used to examine the effects of different elements, how parameters interact, and how to optimize the process for all variables.

2. Materials and methods

2.1. Materials

The source of the Reactive Orange (RO16) dye was ACROS, Organics. The chemical formula for this substance is $C_{20}H_{17}N_3Na_2O_{11}S_3$, while its molecular weight is 617.54 g/mol and its maximum absorption wavelength (λ_{max}) is 493 nm. The supplier of tartaric acid ($C_4H_6O_6$) was R&M Chemicals. All supplementary reagents utilized in this inquiry belonged to the analytical grade. The experiments performed in this research employed ultrapure water.

2.2. Preparation of RHAC

The RH was thoroughly cleaned with distilled water many times to eliminate any impurities. The specimen was subjected to a drying process in an oven set at a temperature of 80 °C for a duration of 24 hours. Next, the RH was subjected to treatment with tartaric acid in a 1:1 ratio, which served as an activating agent. Subsequently, a quantity of 50 g of RH was introduced into the pyrolysis reactor and subjected to a temperature of 700 °C for a duration of 60 min, while maintaining a nitrogen flow rate of 5 litres per minute. Following the pyrolysis procedure, the reactor was extracted from the furnace, yielding the resulting product known as rice husk activated carbon. Preparation of RHAC and its application in the removal of RO16 dye are given in Fig. 1.

2.3. RHAC characterization

A scanning electron microscope (SEM, Zeiss Supra 40 VP, Germany) was employed to examine the surface morphology of RHAC both prior to and subsequent to the adsorption of RO16. By employing Fourier Transform Infrared (FTIR) Spectroscopy with a Perkin-Elmer Spectrum RX I instrument, the functional groups present in the samples were discriminated.

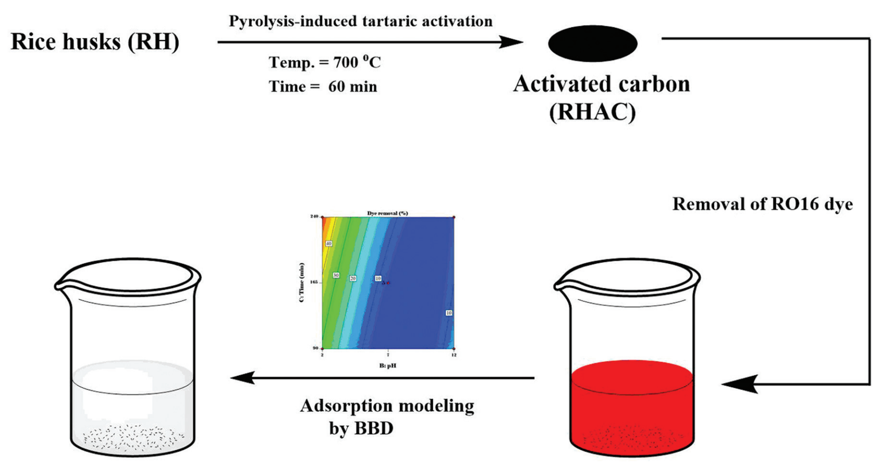

Fig. 1. Preparation of RHAC and its application in the removal of RO16 dye.

2.4. Design of experiments

The primary focus of this investigation is a three-level of BBD implemented in RSM. The objective is to determine the most effective adsorbent quantity, solution pH, and process duration. For the purpose of determining the significance of each variable in the optimization procedure, statistical analysis software [Design Expert Software version 11.0 Stat Easy,] was utilized. Three distinct levels were used to manipulate each component: −1, 0, and +1, which corresponded to low, medium, and high values, respectively. The configuration of the factorial design is illustrated in Table 1. A comprehensive set of seventeen experiments was undertaken in this research endeavour to evaluate the influence of the four input factors on the efficacy of RO16 removal. The experimental data were fitted with a second-order polynomial using a nonlinear regression technique to identify the significant model components. Equation (1) illustrates the quadratic response model, which comprises linear terms, square terms, and linear by linear interaction items:

$$Y = \beta_0 + \sum \beta_i X_i + \sum \beta_{ii} X_i^2 + \sum \sum \beta_{ij} X_i X_j \tag{1}$$

Y is the response variable that is being optimised. The coefficients are represented as follows: β_0 represents the constant, β_i represents the linear, β_{ii} represents the quadratic, and β_{ij} represents the interaction. X_i and X_j are encoded values that denote the independent components. For the purpose of optimizing the designated input variables, a series of seventeen experimental trials were performed. 125 mL Erlenmeyer flasks containing 50 mL of a solution containing 50 mg/L of RO16 were supplemented with an unspecified amount of adsorbent. After securing the containers, they were subjected to a continuous swaying motion of 100 strokes per minute for a period of 60 minutes using a temperature-controlled water bath shaker. Preliminary testing was utilized to determine the range of the variables. The adsorbent was subsequently separated utilizing a syringe filter featuring a pore size of 0.45 μm, subsequent to the stirring operation. Slight fluctuations in RO16 concentration were subsequently quantified at a wavelength of 464 nm utilising a HACH DR 2800 Direct Reading Spectrophotometer. Equation (2) was employed to calculate the removal efficiency (RE,%) of the dye (RO16).

Table 1. Coded and actual variables and their levels.

Coded variables	Actual variables	Level 1 (−1)	Level 2 (0)	Level 3 (+1)
A	Adsorbent dose (g)	0.04	0.09	0.14
B	pH	2	7	12
C	Time (min)	90	150	240

$$RE\ \% = \frac{(C_o - C_e)}{C_o} \times 100 \tag{2}$$

The initial and final concentrations of the RO16 dye in a liquid solution are denoted by the symbols C_o (mg/L) and C_e (mg/L), respectively. Table 2 displays the present design of the experiment matrix for BBD.

3. Results and discussion

3.1. Response surface methodology

3.1.1. Box-Behnken design

A total of 17 experiments were planned to use the Box-Behnken design (BBD), as shown in Table 2. The study examined the independent process parameters and their individual and interacting impacts on the

Table 2. The 3-factors BBD matrix and experimental data for dye removal (%).

Run	A:Adsorbent dose (g)	B:pH	C:Time (min)	Dye removal (%)
1	0.09	2	90	23
2	0.14	2	165	44
3	0.04	12	165	7.2
4	0.09	12	240	14.36
5	0.09	7	165	6
6	0.09	2	240	53
7	0.09	7	165	6.47
8	0.14	12	165	7.42
9	0.04	7	240	7.37
10	0.09	12	90	11.53
11	0.09	7	165	7.43
12	0.14	7	240	7.79
13	0.14	7	90	9.12
14	0.04	7	90	6.08
15	0.04	2	165	35
16	0.09	7	165	7
17	0.09	7	165	9.79

effectiveness of RO16 removal. The investigation was conducted utilizing the BBD technique. A quadratic polynomial model was used to establish the mathematical correlation between the answer and the process components. The mathematical representation of the observed connections between the element being tested and the resulting response is expressed in Equation (3).

$$\text{RO16 removal } (\%) = -14.31B - 6.79BC - 4.22BC - 11.25AC + 16.97B^2 \quad (3)$$

3.1.2. Effect of input variables

Fig. 2 illustrates the use of the perturbation plot to examine the combined impact of three input factors on the efficiency of RO16 removal. There are four primary aspects that contribute to achieving the highest level of RO16 removal efficiency. A pronounced inflection in the pH (variable B) suggests that the RO16 removal was highly responsive to this variable. In general, a significant change in the rate of change over time (referred to as variable C) indicates that the effectiveness of RO16 removal was influenced by this variable. The relationship between the adsorbent dosage (variable A) and the response shows that the reaction is not affected by changes in the adsorbent dose levels.

3.1.3. Analysis of variance (ANOVA)

ANOVA was used to statistically analyze the experimental data on elimination efficiency, and the results are shown in Table 3. To determine important variables, ANOVA findings use *p*-values, sum of squares, and F-values. The RO16 dye model's F-value of 14.49

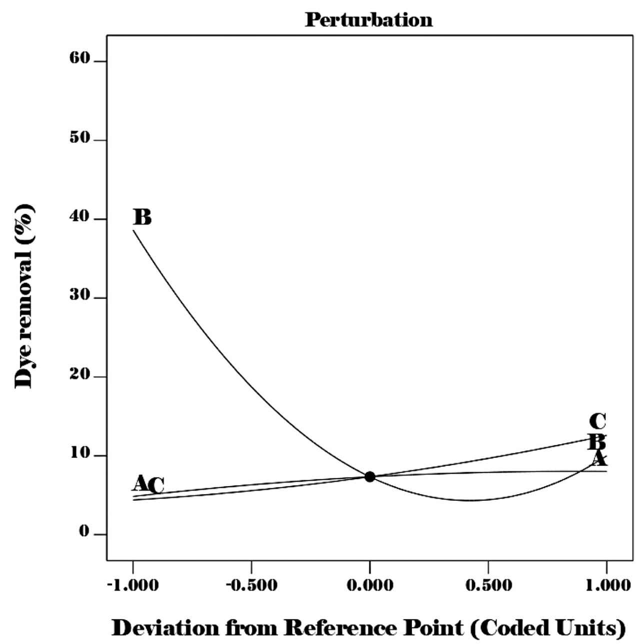

Fig. 2. Perturbation plots for the dye removal efficiency of MO. (A) adsorbent dose, (B) pH, and (C) time.

Table 3. Analysis of variance (ANOVA) of the response surface quadratic model for RO16 removal efficiency.

Source	Sum of squares	Df	Mean square	F-value	*p*-value
Model	3229.60	9	358.84	14.49	0.0010
A-Adsorbent dose	20.10	1	20.10	0.8116	0.3976
B-Ph	1638.50	1	1638.50	66.17	<0.0001
C-Time	134.40	1	134.40	5.43	0.0526
AB	19.27	1	19.27	0.7783	0.4069
AC	1.72	1	1.72	0.0693	0.7999
BC	184.55	1	184.55	7.45	0.0293
A^2	3.47	1	3.47	0.1401	0.7192
B^2	1213.23	1	1213.23	49.00	0.0002
C^2	5.66	1	5.66	0.2287	0.6471
Residual	173.33	7	24.76		
Cor Total	3402.94	16			

(*p*-value of 0.0010) indicates that the designed model is valuable [22]. The actual and projected values showed a significant correlation, as shown by the coefficient of determination (R^2) value of 0.95. The model terms with Prob > F < 0.0500 values, as shown in Table 3, suggest that the factors are significant under the selected conditions. Important elements in the model include the interaction between B and C, the quadratic term of B^2, and the linear terms of B. However, factors in the model with a *p*-value higher than 0.05 are regarded as insignificant. To increase the model's fit, these unimportant elements were thus eliminated. The model may be validated and the characteristics of the residual distribution of the model can be explained using the graphical approach. As

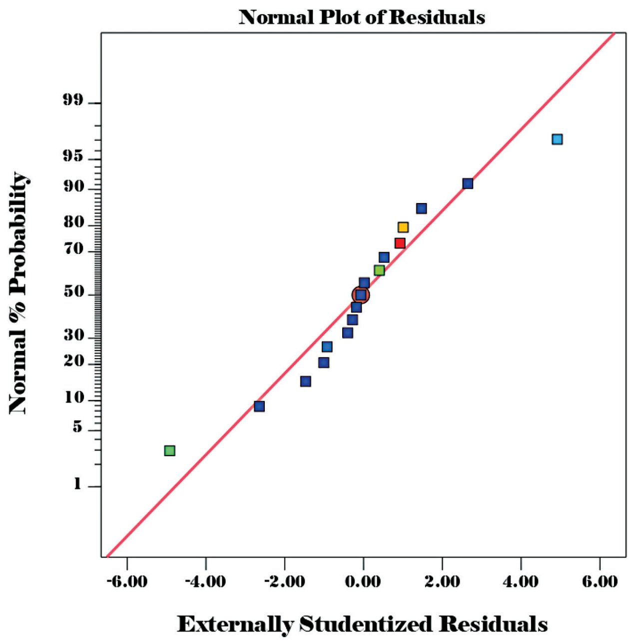

Fig. 3. Normal probability plot of residuals for dye removal efficiency.

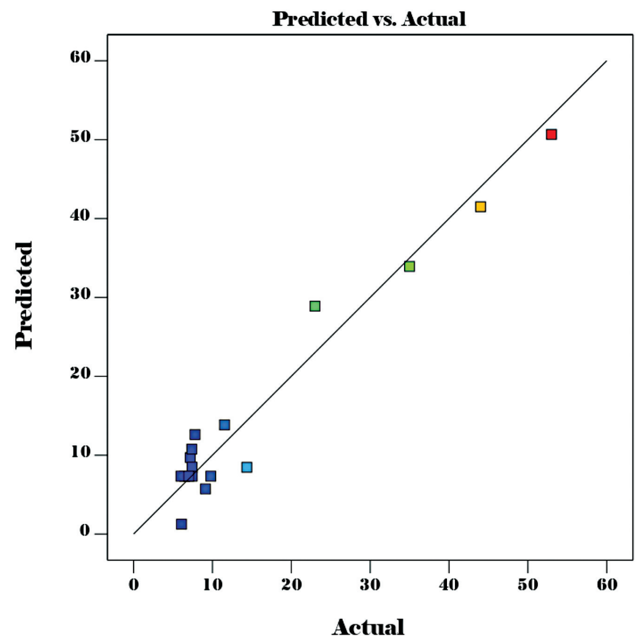

Fig. 4. Plot of the relationship between the predicted and actual values of RO16 removal (%).

shown in Fig. 3, the normal probability plot is often used to evaluate the residuals' distribution in the model [23]. There do not seem to be any departures from the underlying assumptions of the analysis, as shown by the plot of the normal probability of the residuals. On this map, there is a clear clustering of dots around a straight line. The residuals' perfect conformance to normal distributions provides evidence of the independence of the residuals and the validity of the assumptions. The association between the expected and actual values of the RO16 elimination percentage (%) of RO16 by RHAC is shown in Fig. 4. The model's statistical validity is confirmed by the great degree of closeness between the actual and anticipated values along a linear trajectory.

3.1.4. Impact of key interactions

The key interaction between every pair of input factors was investigated in the research (Table 3). With a p-value of 0.0293, the combined effects of pH (B) and time (C) significantly affected the efficacy of RO16 removal. While Fig. 5(b) depicts the two-dimensional contour plots, Fig. 5(a) presents the three-dimensional surfaces for the BC interaction. As the solution pH is lowered from 12 to 2, Fig. 5's measurements show that the removal efficacy of RO16 increases from 6% to 53%. The attraction between the negatively charged sulfonate group of RO16 and the positively charged surface of RHAC may help to explain this. The greatest removal of RO16 was clearly seen at pH 2, and the removal of both dyes gradually decreased as the pH value rose towards a basic environment. The adsorption of RO16 onto RHAC is primarily due to electrostatic forces, π-π, n-π, and H-bond interactions.

3.2. RHAC's characterization

3.2.1. FTIR analysis

The FTIR spectrum of RHAC before the RO16 dye was removed is shown in Fig. 6(a). The (v-O−H groups) and (v-C=O of RCOOH or RCOOR) are represented by the prominent peaks in Fig. 6(a), which are located at about 3400 cm^{-1} and 1700 cm^{-1}, respectively [24]. The vibrational modes of v-C=C in aromatic rings [25] and v-C–O–C and/or v-C–O in Ar-OH and ROH groups are represented by the bands seen at around 1610 cm^{-1} and 1200 cm^{-1}, respectively. Fig. 6(b) displays the FTIR spectrum of RHAC after the RO16 dye was removed. Certain bands in Fig. 6(b) shifted towards higher wavenumbers, indicating interactions between the RO16 dye molecules that were deposited on the surface of RHAC and the adsorption functional groups of RHAC.

3.2.2. SEM analysis

SEM examination was used to examine the surface morphology of the RHAC both before and after RO16 adsorption. Figs. 7(a) and 7(b) show the SEM images of RHAC and RHAC following RO16 removal, respectively, at a magnification level of 3000×. Fig. 7(a) displays the surface morphology of RHAC, which exhibits an uneven and diverse character with randomly dispersed visible voids. In contrast, the outer surface

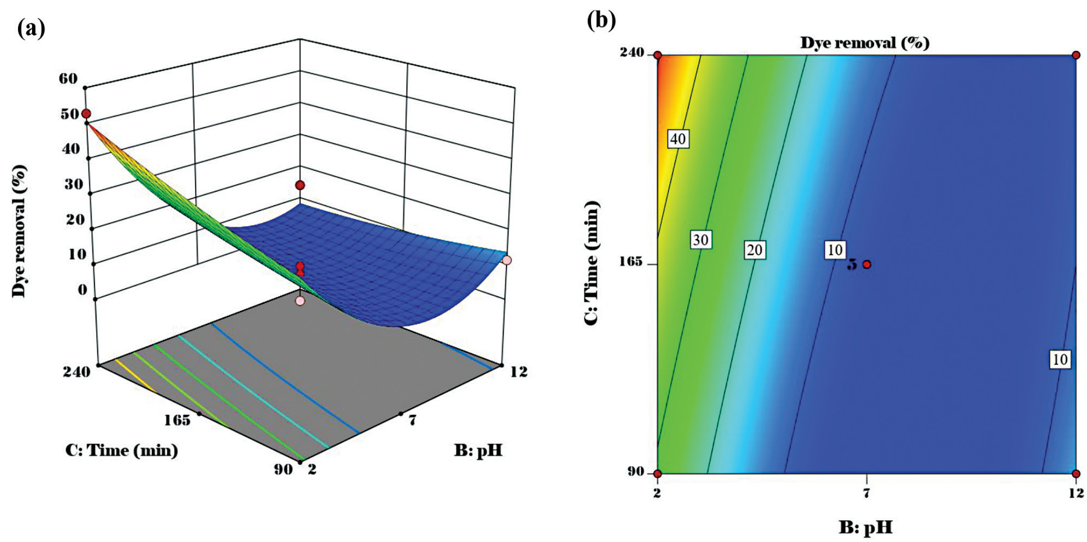

Fig. 5. (a) 3D response surface plot, and (b) contour plot (b) of dye removal efficiency showing interaction between pH (B) and time (C).

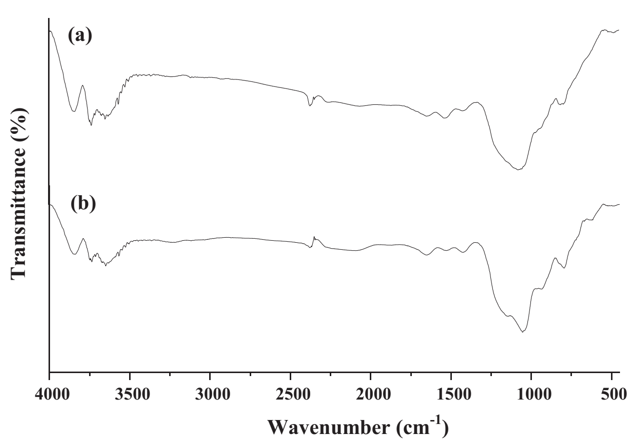

Fig. 6. FTIR spectra of (a) RHAC before dye removal, and (b) RHAC after dye removal.

of RHAC after removing the RO16 dye (as shown in Fig. 7(b)) exhibited a more condensed structure with less noticeable cavities. This shift may be ascribed to the presence of RO16 molecules that were absorbed onto the surface of the adsorbent.

4. Conclusion

To successfully remove RO16 dye from an aqueous solution, activated carbon (AC) was created using biomass derived from rice husks. Employing rice husk activated carbon (RHAC) and a 3-level BBD, RSM was used to increase the effectiveness of eliminating RO16 dye from an aqueous solution. A pH of 2, a contact time of 240 min, and an adsorbent dosage of 0.09 g were found to be the most advantageous conditions for the removal of RO16 dye. The combined interactions with BC at certain pH and time circumstances

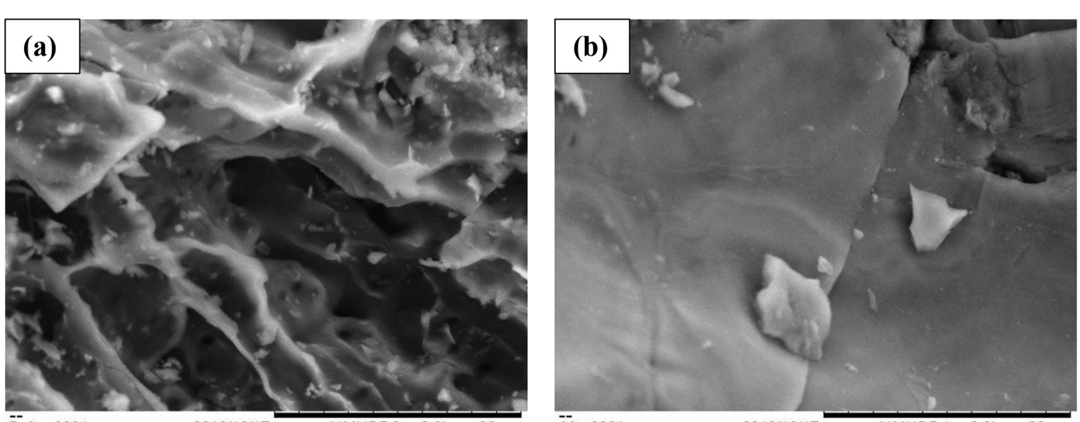

Fig. 7. SEM images (a) RHAC before dye removal, and (b) RHAC after dye removal.

resulted in the most efficient removal of RO16 dye. The capacity to produce activated carbon from rich husk biomass as an adsorbent for the removal of organic dyes is shown by this study.

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