



Chemical Regeneration of A Dye-Laden Activated Carbon: Optimization via The Box-Behnken Experimental Design

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Abstract

Activated carbon is widely used as an adsorbent to remove numerous pollutants from water and wastewater. The cost-effectiveness of an adsorbent depends upon its ability to be reused. This study focuses on regenerating *Milletia thonningii* seed pods' activated carbon (MAC) saturated with Methylene Blue (MB) using acetic acid as a regenerating solvent and exploring its potential to be reused. The effects of the variables such as, the concentration of the regenerating solvent, contact time, and volume of regenerating solvent on the regeneration process were ascertained using the Box-Behnken experimental design, which is a sub-set of Response Surface Methodology. The regeneration process was evaluated based on the desorption capacity of the active carbon. Scanning Electron Microscopy (SEM) and Fourier Transform Infrared spectroscopy (FTIR) was used to characterize the surface of the saturated active carbon before and after regeneration. Results revealed that the concentration of the regenerating solvent had the most significant synergistic effect on the regeneration process. The optimum conditions for the maximum regeneration of the spent activated carbon within the range of the variables studied were found to be: 8M acetic acid, 100 min, and 40 mL of acetic acid. The regenerated and pristine MAC when reused to adsorb fresh MB solutions (50 ml of 10mg/L MB: 0.2g adsorbent) had an adsorption capacity of 2.1912mg/g and 2.0977mg/g for MB respectively. Hence, the regenerated carbon outperformed the pristine active carbon. It could therefore be explored further as a recyclable adsorbent for wastewater treatment.

Keywords: active carbon, regeneration, desorption, reusability, experimental design

1. INTRODUCTION

Dye-laden active carbons obtained from wastewater treatment processes pose a significant threat when disposed directly into the environment due to the possibility of the dye leaching. A sustainable approach to mitigating the environmental impact of such secondary pollutant is to regenerate the spent active carbon [1]. Regeneration and reuse of spent adsorbents ensures that the entire remediation process is cost-effective [2]. The regeneration process basically shifts the adsorption equilibrium from adsorption to desorption. Several techniques have been used to regenerate spent active carbon, such as the thermal method [3][4], the chemical method [5], and the microwave-assisted regeneration method [6]. Chemical regeneration is a simple technique in which acids, alkalis, and organic solvents with

either oxidizing or solubilizing capacity are used to dissolve adsorbates and restore the adsorption ability of the active carbon. The chemical regeneration method has the main advantages of high regeneration efficiency, almost zero carbon loss, quick regeneration, and possible adsorbate recovery. It is useful, especially when adsorbates form strong bonds with the adsorbent's surface [7]. Larasati et al. [8] identified chemical regeneration as a viable alternative to the most commonly used off-site thermal regeneration because it can be performed both in-situ and on-site. Among other adsorbents, the chemical regeneration method has been successfully used in the regeneration of acid green 25 dye saturated waste biomass [9] and methylene blue-saturated bentonite [10]. Ghasemzadeh et al. [4] used hydrofluoric acid to regenerate dye-saturated active carbon and were able to achieve significant regeneration. To regenerate spent activated carbon, Nasiruddin et al. [11] used HCl and hot water. Marion-Peacock et al. [12] demonstrated that chemically regenerated active carbon outperformed thermally regenerated active carbon in a water cleaning system. The chemical regeneration of active carbon saturated with Methylene Blue (MB) dye is the basis for this research. It is cost-effective to regenerate spent active carbon for reuse during the sorptive removal of pollutants from wastewater.

The regeneration of methylene blue-saturated

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Table 1. Levels of factors in the BBD for the Regeneration of Spent MAC.

Factors	Codes	Box Behnken levels		
[Regeneration Solvent](M)	A	2	5	8
Contact time(min)	B	20	60	100
Solvent volume(mL)	C	10	25	40

Millettia thonningii seed pods active carbon (MB-MAC) is investigated in this paper using the Box-Behnken experimental design (BBD), a subset of the Response Surface Methodology (RSM). RSM is a multivariate statistical experimental approach to systematically solving problems. The primary objectives of using experimental design in analytical processes are to obtain optimal and statistically valid results with minimum effort, time, and resources [13][14]. The Box-Behnken experimental design ensures that the relationship between a response and the factors influencing it is understood. It also aids in determining the values of the operating factors or variables that will ensure response parameter optimization by conducting fewer experiments. It has been used to optimize a variety of analytical systems, including the production of activated carbon from agricultural residues [15]–[18], the adsorption of dyes, heavy metals, and antibiotics from aqueous solutions [19]–[21], chromatographic method optimization [22], and a variety of other physical and chemical processes. When compared to the univariate or one-factor-at-a-time (OFAT) experimental approach and other types of response surface methods, the BBD is usually very efficient in terms of the number of required experiments, making it less expensive to run. It is also preferred over other RSM designs because it does not contain combinations in which all of the factors are at their highest or lowest levels simultaneously thus, avoiding experiments performed under extreme conditions that may result in unsatisfactory results. Despite a large number of studies on chemical regeneration of spent activated carbon, it has been discovered from literature that little work has been done on the statistical optimization of process conditions for the chemical regeneration of dye-saturated activated carbons. The novelty of this study is the attempt to regenerate dye-saturated activated carbon using a 3-

factor-3-level Box-Behnken design (BBD). This study will use the BBD to evaluate the effects and interactions of the regenerating solvent concentration, volume of regenerating solvent, and contact time on the regeneration of Methylene blue-saturated *M. thonningii* seed pods activated carbon, as well as to determine the optimum conditions of these variables that will result in the maximum MB desorption capacity of the spent MAC. Scanning Electron Microscopy (SEM) and Fourier Transform Infrared spectroscopy (FTIR) were used to confirm the regeneration of the activated carbon. The suitability of regenerated carbon for reuse was also assessed based on its adsorption of methylene blue dye.

2. MATERIALS AND METHODS

2.1. Materials

The activated carbon used in this study was prepared according to the method described by Jasper et al. [23]. Methylene blue dye, acetic acid and tetraoxo-sulphate (vi) acid were used as reagents. All of the reagents used were of analytical quality. All solutions used in this study were prepared with de-ionized water.

2.2. Methods

2.2.1. Initial Adsorption Experiments

4 g of fresh *M. thonningii* seed pod activated carbon (MAC) was agitated for 180 minutes with 100 mL of 250 mg/L Methylene blue (MB) dye solution at pH 7. The spent MAC was filtered from the solution, washed with deionized water to remove any unadsorbed dye, and oven-dried for 1 hour at 100 °C.

2.2.2. Screening of suitable regenerating solvents

To find the best solvent for desorption of the

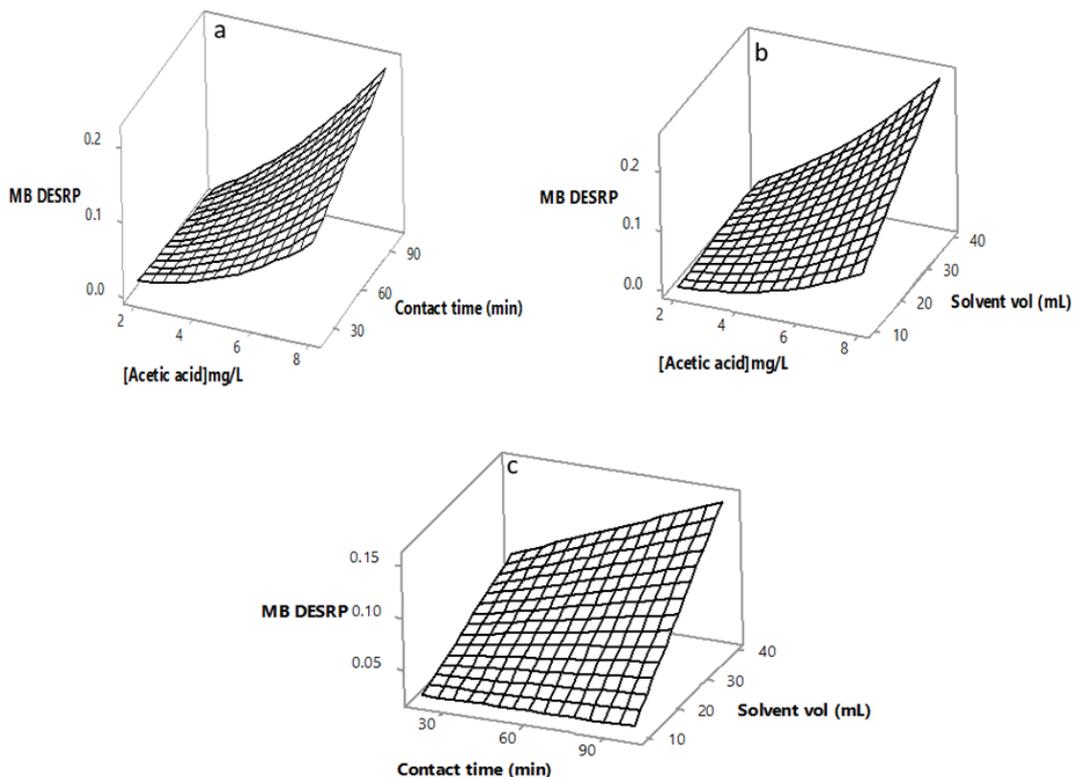


Figure 1. Effect of (a) contact time and acetic acid concentration on the amount of dye desorbed (b)acetic acid concentration and solvent volume and (c)contact time and solvent volume at holding values of 25 ml solvent volume, 60 min contact time and 5M acetic acid concentration

retained dye on MAC, 0.1 g of dye-saturated MAC was mixed separately in three different Erlenmeyer flasks with 50 mL of 0.1 M H₂SO₄, 0.1 M CH₃COOH, and deionized water . The resulting mixtures were agitated for 60 minutes before being separated and the supernatants concentration determined using UV-Vis spectrophotometry (664nm for MB). Equation 1 was used to compute the desorption efficiency [24].

$$\% \text{ desorption} = \frac{C_a - C_d}{C_a} \times 100 \tag{1}$$

where, where C_d is the concentration of dye desorbed (mg/L) and C_a is the dye adsorbed amount (mg/ L). For the following experiments, the solvent with the highest dye recovery was used.

Table 2. Percent MB desorbed by various solvents from saturated MAC.

Solvent	%MB Desorbed
CH ₃ COOH	1.68
H ₂ SO ₄	0.27
Deionized H ₂ O	0.24

2.2.3 Optimization of the Regeneration of Spent MTSPAC using the Box-Behnken Design

The BBD was used to investigate the optimization of factors that affect the regeneration of MB-saturated MAC. The parameters investigated were the regenerating solvent concentration (CH₃COOH), the volume of regenerating solvent, and the contact time. After screening for suitable solvents, acetic acid was chosen as a regenerating solvent because it provided the highest percent desorption. The levels of experimental parameters used for the optimization study are shown in Table 1.

The BBD created 15 randomized experimental runs with three replicated center points for the optimization experiments based on the equation (2).

$$N = n^2 + n + p \tag{2}$$

where *N* represents the total number of experiments, *n* represents the number of variables or factors, and *p* represents the number of center points. Centre points are experimental runs in which the factors are set at the medians of their high and low levels. To improve experiment precision, the

Table 3. BBD matrix for the optimization of the regeneration of MB-saturated MTSPAC.

Run	[CH ₃ COOH] (mol/L)	Contact time(min)	Solvent volume (mL)	MB Desorped (mg/g)
1	5	60	25	0.0665
2	8	20	25	0.1268
3	2	20	25	0.0144
4	5	100	40	0.1575
5	5	60	25	0.0634
6	2	100	25	0.0011
7	5	20	10	0.0260
8	8	60	40	0.2415
9	2	60	40	0.0216
10	8	100	25	0.2250
11	8	60	10	0.0758
12	5	60	25	0.0674
13	5	20	40	0.0766
14	2	60	10	0.0061
15	5	100	10	0.0270

center point is replicated. Table 3 shows the complete design matrix generated by Minitab Software, Version 17.0, based on the range of process variables (Minitab Inc., USA). In each run, different volumes of the desorbing solvent, CH₃COOH, were contacted with 0.2 g of MB-saturated MAC at contact times ranging from 20-100 min in 150 mL stoppered Erlenmeyer flasks at different initial concentrations as specified in Table 3. All samples were centrifuged at 500 rpm for 5 minutes after they were removed from the flasks. The concentrations of residual MB solutions were measured using UV-Vis spectrophotometry at 664 nm, and the amount of dye desorbed q_e (mg/g) was calculated using Equation (3).

$$q_{e \text{ desorption}} = V \left(\frac{C_f}{m} \right) \quad (3)$$

where V = volume of solvent (L); m = mass of adsorbent (g) and C_f = dye concentration in solvent (mg/L). The experimental data obtained was subjected to statistical treatment which included a multiple regression analysis and an analysis of variance (ANOVA) in order to derive suitable second-order polynomial regression model equations which could express each predicted response (Y) as a function of the independent preparation variables (factors) by fitting the

quadratic polynomial function (Eq. 4) to the data set.

$$Y = \alpha_0 + \alpha_1 A + \alpha_2 B + \alpha_3 C + \alpha_{11} A^2 + \alpha_{22} B^2 + \alpha_{33} C^2 + \alpha_{12} AB + \alpha_{13} AC + \alpha_{23} BC \quad (4)$$

where α_{0-23} , are regression coefficients, α_0 is a constant term; α_1 , α_2 , and α_3 are linear effect terms; α_{11} , α_{22} and α_{33} are the square effect terms and α_{12} , α_{13} , and α_{23} are cross-product or interactive effect terms. A, B, and C are the coded independent variables that represent the important factors affecting the process being carried out and Y is the particular response being evaluated. The data was analyzed with the Minitab 17.0 software, and the optimum conditions for regenerating the spent activated carbon, that is, the settings that could obtain the highest amount of MB desorbed, were obtained using the software's optimization functions. Experiments were carried out in triplicate at the optimal settings to validate the software's predictions.

2.2.4. Evaluation of the Adsorption capacity of MAC Regenerated at Optimum Conditions

Adsorption experiments were conducted in separate Erlenmeyer flasks by agitating 10 mg/L of MB with 0.2 g pristine MAC and regenerated MAC

per 50 mL of dye for 120 minutes, and their adsorption capacities for MB were determined using Eq. 5.

$$q_t = \frac{(C_o - C_t)V}{m} \tag{5}$$

Where C_o , C_t (mg/L) are the liquid phase concentrations of the dye at initial, and any time t , respectively, V is the volume of solution (L) and m is the mass of the dry adsorbent (g).

2.2.5. Surface and Morphological Characterization of the Regenerated MTSPAC

The morphology and surface functional groups of the active carbon regenerated under optimal conditions were compared to those of the MB-loaded MAC and the pristine MAC using Scanning Electron Microscopy (Phenom Pro X, Netherlands) and Fourier Transform Infrared Spectroscopy (FTIR-CARY 630, Agilent Technologies).

3. RESULTS AND DISCUSSIONS

3.1. Solvent Selection

The regeneration of spent activated carbon assumes paramount importance considering its economic reuse during the sorptive removal of pollutants from wastewater. Of the three solvents

tested for their suitability in the regeneration of spent carbon, CH_3COOH removed the most dye (Table 2) and was thus chosen as the regenerating solvent for subsequent experiments. Organic solutions with lower molecular weight than adsorbate have been reported to be effective regenerant solutions [25].

3.2. Experimental Design and Statistical Analysis

The regeneration of the spent *M. thonningii* seed pods' active carbon was evaluated as a function of its the methylene blue desorption capacity. The results of the desorption experiments are shown in Table 3.

The amount of MB desorbed per gram of spent MAC ranges from 0.0011 to 0.2415 mg/g, with the lowest value obtained at factor settings of 25 ml of 2 M acetic acid for a contact time of 100 minutes and the highest value obtained at factor settings of 40 ml of 8M acetic acid for a contact time of 60 minutes. The center points are replicate measurements taken on runs 1, 5, and 12 to determine the precision of the experiments.

3.3. Regression Analysis and ANOVA Interpretation

Multiple regression analysis was performed on the experimental data (Table 3) to obtain a polynomial equation that correlates the response

Table 4. ANOVA for final response surface quadratic model for the regeneration of MB-saturated MAC

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	7	0.080668	0.011524	198.40	<0.001
A: [Acetic acid] mol/L	1	0.048969	0.048969	843.06	<0.001
B: Contact time(min)	1	0.003478	0.003478	59.87	<0.001
C: Solvent vol (mL)	1	0.016408	0.016408	282.48	<0.001
A ²	1	0.001469	0.001469	25.29	0.002
AB	1	0.003108	0.003108	53.51	<0.001
AC	1	0.005640	0.005640	97.10	<0.001
BC	1	0.001596	0.001596	27.48	0.001
Error	7	0.000407	0.000058		
Lack-of-Fit	5	0.000398	0.000080	18.07	0.053
Pure Error	2	0.000009	0.000004		
Total	14	0.081074			
R-sq		99.50%			
R-sq(adj)		99.00%			
R-sq(pred):		96.72%			
S		0.0076213			

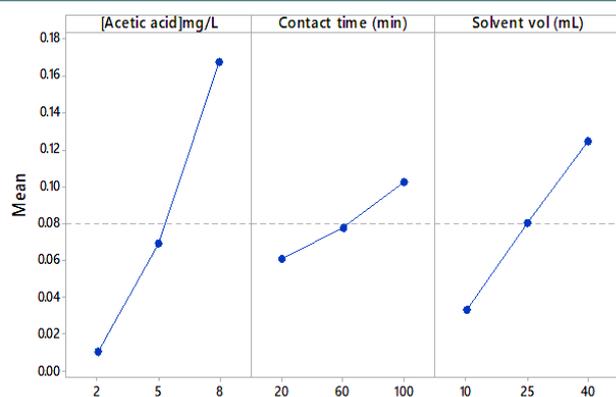


Figure 2. Main effects plots for the regeneration of MB-saturated MTSPAC.

variable, Y, that is, the amount of MB desorbed, to the independent variables (regenerating solvent concentration (CH_3COOH), volume of regenerating solvent, and contact time). Eq. 6 depicts the final model equation obtained after removing statistically insignificant terms.

$$Y(\text{MB desorbed}) = 0.06920 + 0.07824A + 0.02085B + 0.04529C + 0.0198A^2 + 0.02788AB + 0.03755AC + 0.01997BC \quad (6)$$

An analysis of variance (ANOVA) was used to determine the statistical significance of the model equation, which included a Fisher's F-test and its associated probability, p (F), at the 95 % confidence level. The large value of F indicates that the regression equation can explain the majority of the variation in the response, while the associated p-value estimates whether F is large enough to indicate statistical significance. The model is statistically significant if the p-value is less than 0.05. Table 4 showed that the model with an F-value of 198.40 and $p < 0.0001$ was statistically significant. The model had a high coefficient of determination, R^2 of 99.50, indicating that only 0.50 % of the variability in the response was not explained by the model. The adjusted R^2 value, R^2 (adj), which represents the proportion of the data that the model accounts for, was also high, at 99.00 %. The model's predictive ability was also statistically acceptable, with a high R^2 -pred value of 96.72 %. The R^2 (adj) and R^2 (pred) were in reasonable agreement, falling within about 20% of each other [25][26]. The ANOVA results also show a lack-of-fit (LoF), which was not statistically significant because the p-value was greater than

0.05. The hypothesis is that if the LoF is not statistically significant, the model is adequate; if the LoF is statistically significant, the model is unfit to represent the data [27]. The statistical findings support the model's good predictability and adequacy within the reasonable range of the variables studied.

The effect of the regenerating solvent (CH_3COOH) concentration (A), contact time (B), and solvent volume (C) on the amount of MB desorbed was also investigated. According to the ANOVA results (Table 4), all linear factors (A, B, and C) had a statistically significant influence on methylene blue desorption, as indicated by p-values of < 0.001 . With the highest F-value of 843.06, the concentration of the regeneration solvent, A, had the most significant effect on the amount of MB desorbed, followed by solvent volume ($F=282.48$) and contact time ($F=59.87$). The positive sign of the coefficients in the regression model Eq. 6 indicates that all linear factors had a synergistic effect on the response variable, which means that as all factors increased, so did the amount of dye desorbed.

3.3.1. Response Surface Plots

3-D response surface plots were constructed to show the effects of the regeneration variables on the response, desorption capacity of methylene blue (Fig 1). Response surface plots explain the nature of the relationship between two factors on the response while holding a third factor constant. Figure 1a shows the response plot for contact time and acetic acid concentration. Within the range of variables, the MB desorption capacity gradually increased with increasing contact time and acetic acid concentration. This finding could be attributed to an increase in the presence of H^+ as a result of the acetic acid displacing dye molecules from the adsorbent pores increasing in concentration. With increased regeneration contact time, it appears that more pores were re-opened. Figure 1b shows the

Table 5. Adsorption capacity of the pristine and regenerated MAC

Adsorption Capacity Pristine MAC (mg/g)	Adsorption Capacity Regenerated MAC (mg/g)
2.0977	2.1912

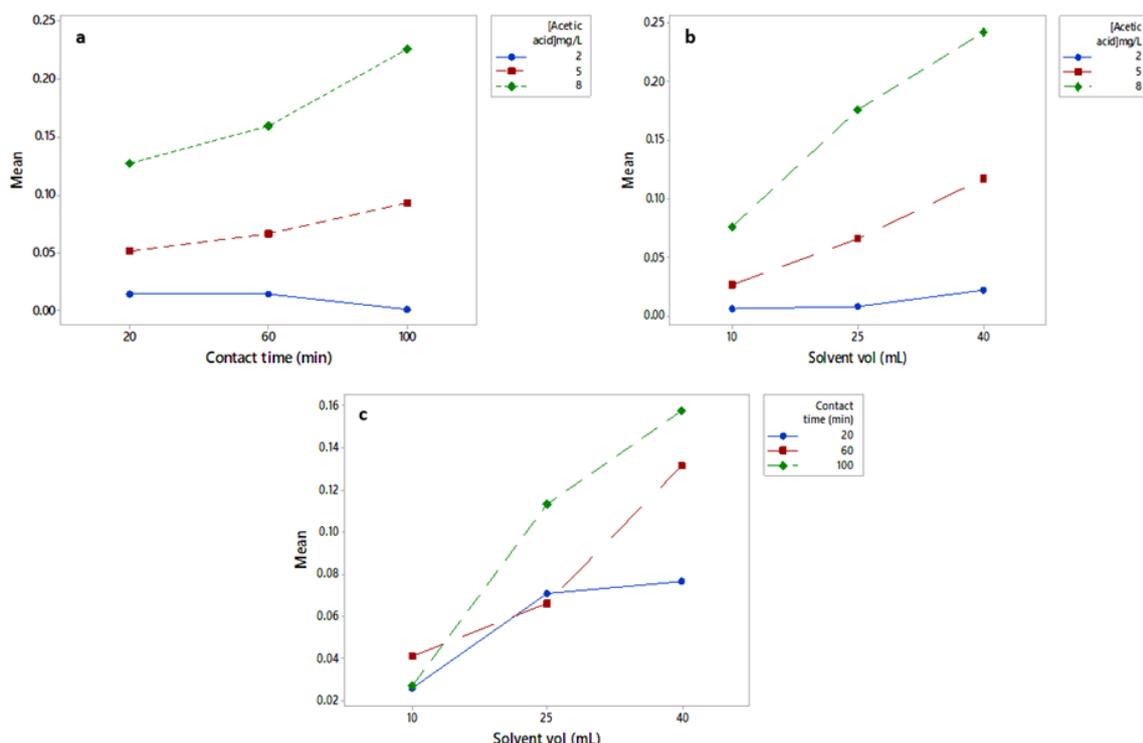


Figure 3. Interaction effects between (a) acetic acid concentration and contact time (b) acetic acid concentration and solvent volume (c) contact time and solvent volume for the Regeneration of MB-saturated MTSPAC.

response plot with respect to acetic acid concentration and solvent volume, which shows a similar response to Figure 1a. The plot demonstrates that the maximum desorption capacity was obtained at the highest solvent volume and concentration. Fig 1 c shows that at a concentration of 5M, contact time had little effect on the response; however, increasing the solvent volume resulted in a significant increase in the response value.

3.3.2. Main Effects and Interaction Plots

The Main effects plots depicted in Fig. 2 show the extent of each factor's individual effects. The main effect plot contains useful information about the relative importance of each factor parameter on the response. Main effect plots compare changes in

level means to determine which factors have the greatest influence on the response: when the line is parallel to the x-axis, there is no main effect. The main effect, however, appears when the line is not parallel to the x-axis. The slope of the plot represents the relative strength of the factors' effect [28]. A steeper slope indicates that the process factor has a greater impact on the response. Based on this premise, the acetic acid concentration had the greatest influence on the regeneration process. The increase in MB desorption from spent carbon with increasing acetic acid concentration could be explained by the accumulation of H^+ in solution, which competes with the dye ions on the adsorbent's surface, resulting in the release of MB^+ ions from the adsorbent thus favoring the desorption process. The mean was higher than average for CH_3COOH concentrations greater than 5M, times greater than 60 minutes, and volumes greater than 25 mL, with a peak value obtained at 8M CH_3COOH concentration. Figure 2 clearly shows that the factors influenced the amount of MB desorbed in the following order: acetic acid concentration > solvent volume > contact time.

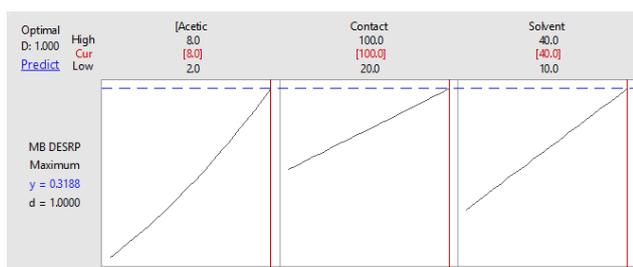


Figure 4. Optimization plots for the regeneration of MB-saturated MTSPAC.

Figure 3 depicts the interaction plots for the response, MB desorbed. Interaction plots show how

the relationship between one factor and the response is affected by the value of a second factor. When the lines are more non-parallel, there is more interaction. It can be seen that there is an interaction between A and B; A and C and B and C due to the non-parallel lines. The variability in MB desorption is greatest when the acid concentration and solvent volume are both increased. The MB desorption is found to be less sensitive to changes in contact time and solvent volume. Also significant are the interactions between A and B, A and C, and B and C (Table 3).

3.3.3. Optimization and Validation

Response optimization with the Minitab software was used to determine the factor settings that could achieve the maximum MB desorption capacity of the spent active carbon. The response optimization plot (Fig. 4) shows that the optimum experimental conditions of 8M acetic acid, 100 min contact time, and 40 mL solvent volume with a predicted response of 0.31881mg MB per gram of activated carbon were obtained with a desirability function of 1.0. The experiments performed to validate these predictions and justify the accuracy of the results yielded an average of 0.3065mg MB per gram of activated carbon.

3.3.3. Adsorption Capacity of Pristine and Regenerated MAC

Table 5 shows that the adsorption capacity of the MB-saturated MAC regenerated under optimal conditions was 2.1912mg/g while that of the pristine MAC was 2.0977mg/g.

This means the regenerated MAC produced at optimal settings outperformed the pristine MAC. The treatment of spent activated carbon with acetic acid may have resulted in the formation of more active sites on the carbon's surface, enhancing the activated carbon's ability to adsorb greater amounts of methylene blue.

3.4. SEM and FTIR Analysis of Regenerated Spent MAC

Fig 5a depicts the surface of MAC before adsorption; it is characterized by a smooth porous surface. After MB adsorption (Fig 5b), the surface appears rough and corroded due to the surface pores being covered by MB dye molecules. The SEM micrograph obtained on regeneration (Fig 5c) shows the reappearance of several pores of different sizes when compared to the saturated MAC (Fig 5 b). This shows evidence of successful regeneration of the carbon when compared to that of the post adsorption micrograph (5b). This indicates that the

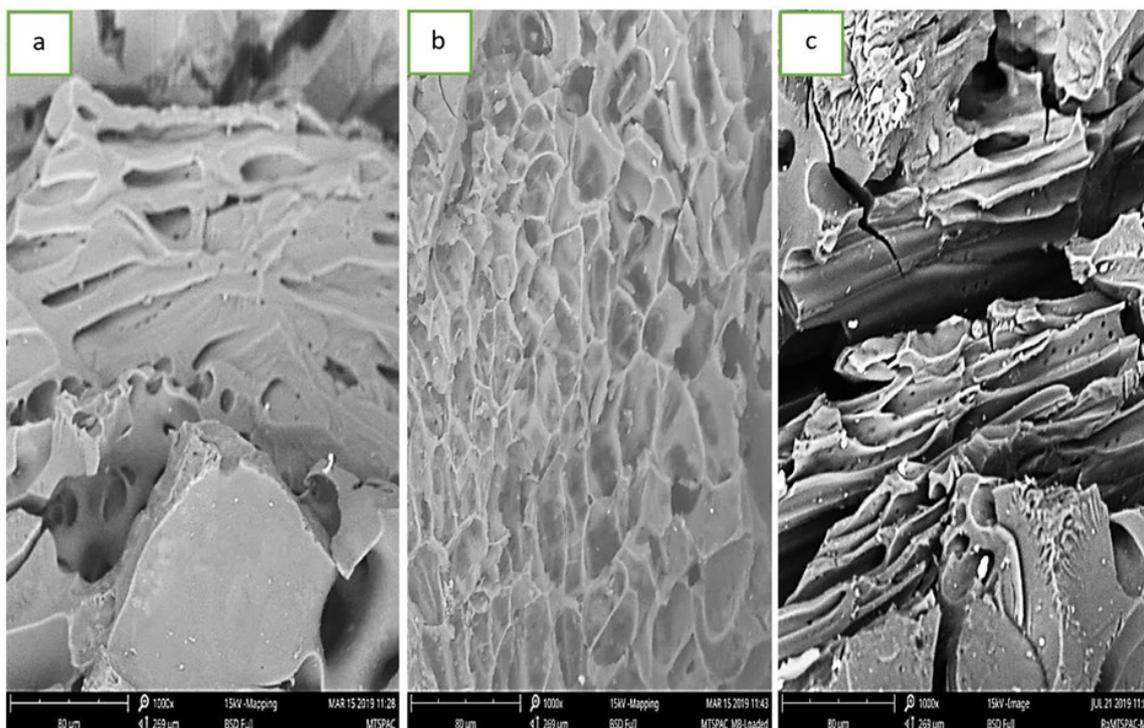


Figure 5. SEM micrographs of (a) fresh MTSPAC (b) MB-loaded MTSPAC and (c) Regenerated MTSPAC-MB at x 1000 magnification.

4. CONCLUSIONS

The regeneration of spent *Millettia thonningii* seed pods' active carbon has been examined and optimized with respect to dye desorption capacity. Box–Behnken experimental design, a subset of Response Surface Methodology was used to locate the optimum operating variables (the concentration of the regenerating agent, acetic acid, solvent volume, and contact time) to maximizing the dye desorption capacity of the spent MAC. While all the factors and their interactions significantly influenced the regeneration process, the concentration of the regenerating agent, acetic acid, had the most significant influence on the process. The optimum conditions for the operating factors for the maximum regeneration of the spent activated carbon within the range of the variables studied were found to be: 8M acetic acid, 100 min, and 40 mL of acetic acid. The maximum amount of methylene blue desorbed approached 0.3065 mg/g under these experimental conditions.. The results reveal that the regenerated MAC could be reused and as such has a potential application as a recyclable adsorbent for the treatment of wastewater. Its reusability should be explored further.

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Author Contributions

E. E. J. conceptualized, designed, and performed the experiments, analyzed the results and drafted the manuscript. J. C. O. participated in designing the study, coordinating the experiments, analyzing the results and drafting the manuscript. E. B. A. participated in designing this research, analyzing the results, and drafting the manuscript.

Conflicts of Interest

The author(s) declared no conflict of interest

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