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Adsorption of crystal violet from quaternary dye mixture onto sawdust: statistical analysis and optimization studies

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ORIGINAL RESEARCH ARTICLE

ABSTRACT

The selective adsorption of crystal violet from dye mixture of methylene blue, malachite green, and Rhodamine B using sulphuric acid-treated sawdust of *Parkia biglobosa* was investigated through crossed composite Design of Experiments. The competitive effects of mixture components and process parameters on the adsorption process were studied and optimized using response surface methodology. The optimum contact time, pH, adsorbent dose and temperature were found to be 275.10 min, 9.94, 0.99g and 60 °C respectively for the maximum decolorization of 68.39 mg/L CV (97.2%). A linear model was obtained for the dye decolourization through this design. The experimental values were in good agreement with predicted values and the model developed was highly significant with the correlation coefficient of 0.985. Experimental results were analyzed by Analysis of variance (ANOVA) statistical concept.

KEYWORDS

adsorption; crossed composite design; crystal violet; dye mixture; *Parkia biglobosa*

1. INTRODUCTION

Wastewaters from textile and related industries contain wide range of dyes and chemical additives; and thus act as a source of pollution (Hussain et al., 2015). Upon their discharge into the environment, the effluents from these industries could have adverse carcinogenic and toxic effects, in addition to aesthetic dissatisfaction. They are therefore harmful to animals and plants or even to human beings (Mahvi et al., 2009; Mahvi and Heibati, 2012; Ashrafi et al., 2013). Adsorption using commercial activated carbon has proven to be very effective for the removal of dyes and other pollutants from wastewater but is still considered to be expensive (Hameed et al., 2008). The removal of basic dyes from aqueous solutions has been studied using activated carbons prepared from various alternative precursors (Hameed et al., 2007; Tan et al., 2007; Tan et al., 2008; Kavitha and Namasiviyan, 2007). In particular, studies have been conducted on the removal of crystal violet from aqueous media using various types of adsorbents (Kulkarni et al., 2017; Mashkoor et al., 2018; Tahir et al., 2017; Shoukat et al., 2017; Jayganesh et al., 2017; Jayasantha Kumari et al., 2017; Miyah et al., 2017). The widely used traditional step-by-step approach of batch adsorption involves a large number of independent runs and does not consider the simultaneous interaction of multiple parameters. Apart from this, the method is also time and material consuming and requires a large number of experimental trials to capture the effects of all the factors. These limitations can be minimized through corresponding interactions among the variables studied and optimized using crossed composite design and response surface methods with contour plots (Jaikumar and Ramamurthi, 2009).

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Only limited works were reported on the optimization studies of the adsorption of dyes using single dye system where only one dye was present in aqueous environment and was attempted to be removed from the solution (Jaikumar and Ramamurthi, 2009; Ahmad et al., 2015; Khamparia et al., 2015). However, since wastewaters rarely contain only a single substance, it is more appropriate to consider adsorption of pollutants from multi-component solutions. In this study therefore, optimization process for the removal of a basic dye, crystal violet, by the sulphuric acid-charred sawdust of *Parkia biglobosa* from a quaternary mixture of basic dyes at different operating conditions, was performed using Design of Experiment and Response Surface methodologies (Abdulsalam et al., 2017).

2. MATERIALS AND METHODS

2.1. Preparation and characterization of adsorbent

The adsorbent was prepared and characterised as reported elsewhere (Abdulsalam et al., 2017). Sawdust of *Parkia biglobosa* collected from Ogbomoso saw mill was the precursor for preparation of the adsorbent. It was sieved with 10-20 mesh and charred with concentrated sulphuric acid in ratio 1:1 (w/v). It was charred further and activated in an electric oven at 160 ^{o}C for 15 h. Excess acid on the adsorbent was neutralised with $NaHCO_3$ solution (5% w/v), then washed with distilled water till a stable pH was obtained. It was dried, stored and labeled as sulphuric acid-modified sawdust (SAMS). Scanning Electron Microscopy (SEM), Fourier Transform Infrared (FTIR) Spectroscopy and Elemental Diffraction X-ray Spectroscopy (EDS) were employed to characterize the adsorbent.

2.2. Preparation of adsorbate

An aqueous solution of a mixture of four dyes; malachite green (MG), methylene blue (MB), crystal violet (CV) and rhodamine B (RB) was used as artificial wastewater in this study, with crystal violet (CV) as the analyte. The wavelength of maximum absorbance (λ_{max}) of crystal violet was determined. A stock solution (1000 mg/L) of each dye was prepared and the working solutions of different desired concentrations were obtained by appropriately diluting the stock.

2.3. Crossed Composite Experimental Design

The design selected for the adsorption of dye mixture was crossed composite design because it can simultaneously cater with the mixture of dyes and process parameters. Experiments were performed according to the crossed composite design (CCD) matrix given in Table 1. The response was expressed as percentage color removal, %R, calculated as

$$\%R = (C_o - C_e)/C_o \times 100 \qquad (1)$$

Where, C_o and C_e are the initial and equilibrium concentrations of the dye in mg/L respectively.

Table 1. Design variables for crossed composite design

Factors	Units	Type	Actual Value		Coded Value	
			Low	High	Low	High
Methylene blue	mg/L	Mixture	10	100	0	1
Malachite blue	mg/L	Mixture	10	100	0	1
Crystal violet	mg/L	Mixture	10	100	0	1
Rhodamine B	mg/L	Mixture	10	100	0	1
Dosage	g	Numeric	0.1	1	-1	1
Temperature	^{o}C	Numeric	30	60	-1	1
pH		Numeric	4	10	-1	1
Time	min	Numeric	30	300	-1	1

3. RESULTS AND DISCUSSION

3.1. Characterization of adsorbent and adsorbate

The surface textural morphology of the adsorbent, SAMS, is presented as scanning electron micrograph observed at a magnification of × 200 (Figure 1). The SEM image shows that the surface of the adsorbent is highly porous with evenly distributed pores throughout the surface.

The FTIR spectrum of SAMS (Figure 2a) shows some absorption peaks that indicate the complexity of the material. The spectrum indicates the presence of the OH, COOH, C = O, C = C and C = S functional groups which are potential adsorption sites. The C = S functional group was not present in the raw sawdust (Figure 2b) thereby confirming the chemical modification of the precursor material with sulphuric acid. The result of the EDX elemental analysis of SAMS is given in figure 3. It shows a high percentage of carbon (66.77 %) which makes SAMS a potentially good adsorbent.

The wavelength of maximum absorbance (λ_{max}) of the adsorbate (crystal violet) was determined to be 580 nm.

3.2. Model evaluation and selection

The evaluation of different models for the adsorption of crystal violet from a quaternary dye mixture is shown in Table 2. The standard deviation showed the degree of disparity between the predicted values and the mean experimental values. The correlation coefficient (R^2) value reflects the efficiency of the prediction relative to the actual experimental data. The closer

Table 2. Summary of Model Evaluation

Source	Standard		Adjusted	Predicted		
[Mix]*Process	**Deviation**	**R-Squared**	**R-Squared**	**R-Squared**	**Press**	**Comment**
[Mean]*Linear	1.197252	0.654263	0.630823	0.59318	99.51279	
[Mean]*2FI	1.260391	0.655801	0.590858	0.498099	122.7707	
[Mean]*Quadratic	1.260391	0.655801	0.590858	0.498099	122.7707	Aliased
[Mean]*Cubic	1.306909	0.657855	0.560099	0.416316	142.7757	Aliased
[Linear]*Mean	1.652222	0.330406	0.296927	0.238151	186.3568	
[Linear]*Linear	0.291944	0.984669	0.978049	0.967564	7.934255	Suggested
[Linear]*2FI	0.335418	0.990801	0.971024	0.905806	23.04102	
[Linear]*Quadratic	0.335418	0.990801	0.971024	0.905806	23.04102	Aliased
[Linear]*Cubic	0.250012	0.998978	0.983901	0.738334	64.0064	Aliased
[Quadratic]*Mean	1.652222	0.330406	0.296927	0.238151	186.3568	Aliased
[Quadratic]*Linear	0.291944	0.984669	0.978049	0.967564	7.934255	Aliased
[Quadratic]*2FI	0.335418	0.990801	0.971024	0.905806	23.04102	Aliased
[Quadratic]*Quadratic	0.335418	0.990801	0.971024	0.905806	23.04102	Aliased
[Quadratic]*Cubic	0.250012	0.998978	0.983901	0.738334	64.0064	Aliased
[Special Cubic]*Mean	1.652222	0.330406	0.296927	0.238151	186.3568	Aliased
[Special Cubic]*Linear	0.291944	0.984669	0.978049	0.967564	7.934255	Aliased
[Special Cubic]*2FI	0.335418	0.990801	0.971024	0.905806	23.04102	Aliased
[Special Cubic]*Quadratic	0.335418	0.990801	0.971024	0.905806	23.04102	Aliased
[Special Cubic]*Cubic	0.250012	0.998978	0.983901	0.738334	64.0064	Aliased
[Cubic]*Mean	1.652222	0.330406	0.296927	0.238151	186.3568	Aliased
[Cubic]*Linear	0.291944	0.984669	0.978049	0.967564	7.934255	Aliased
[Cubic]*2FI	0.335418	0.990801	0.971024	0.905806	23.04102	Aliased
[Cubic]*Quadratic	0.335418	0.990801	0.971024	0.905806	23.04102	Aliased
[Cubic]*Cubic	0.250012	0.998978	0.983901	0.738334	64.0064	Aliased

the R-squared and adjusted R-squared value to unity, the better the model. The predicted R-squared also shows the efficiency of the model prediction within the experimental range. Linear model was selected for both the mixture components and process parameters as a result of its high correlation coefficients (R^2 = 0.985; adjusted R-square= 0.978 and predicted R- square= 0.968) (Table 2).

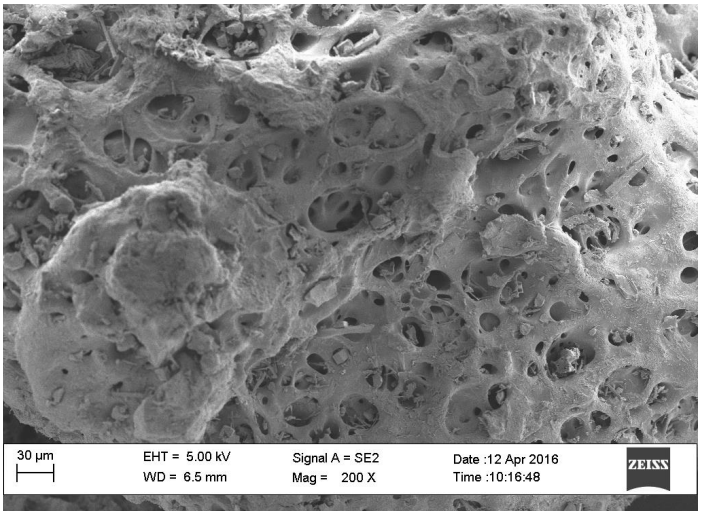

Figure 1. Scanning electron microscopy (SEM) image of SAMS

3.3. Evaluation of model parameters using Analysis of Variance (ANOVA)

The selection of model terms for Percentage Removal (%R) of crystal violet from quaternary dye mixture was done using the analysis of variance. The analyses were done using Fisher's 'F'-test and Student 't'-test. The student 't'-test was used to determine the significance of the regression coefficients of the parameters. The P-values were used as a tool to check the significance of the main variables and interactions among the variables, which in turn may indicate the patterns of interactions among the variables. It also evaluates the desirability of the model through adequate precision (the signal to noise ratio). If the model is desirable, it can be used to navigate the design space.

Table 3 shows the analysis of variance for the model and parameter selection. The Model F-value of 148.74 implies the model is significantly relative to the pure error. There is only a 0.01% chance that a "Model F-Value" as large as this could occur due to noise. The values of "Prob > F" for all the model parameters is less than 0.0500 which indicates that the terms are significant. The final model equation is presented in equation 2 below.

$$\% R = 92.019MB + 92.045MG + 88.299CV + 92.076RB + 2.222MB*dose + 0.033MB*temp + 0.1667MB*pH + 7.466E\text{-}003MB*time + 2.222MG*dose + 0.033MG*temp + 0.1667MG*pH + 7.420E\text{-}003MG*time + 2.222CV*dose + 0.033CV*temp + 0.1667CV*pH + 7.466E\text{-}003CV*time + 2.222RB*dose + 0.033RB*temp + 0.1667RB*pH + 7.466E\text{-}003RB*time \quad (2)$$

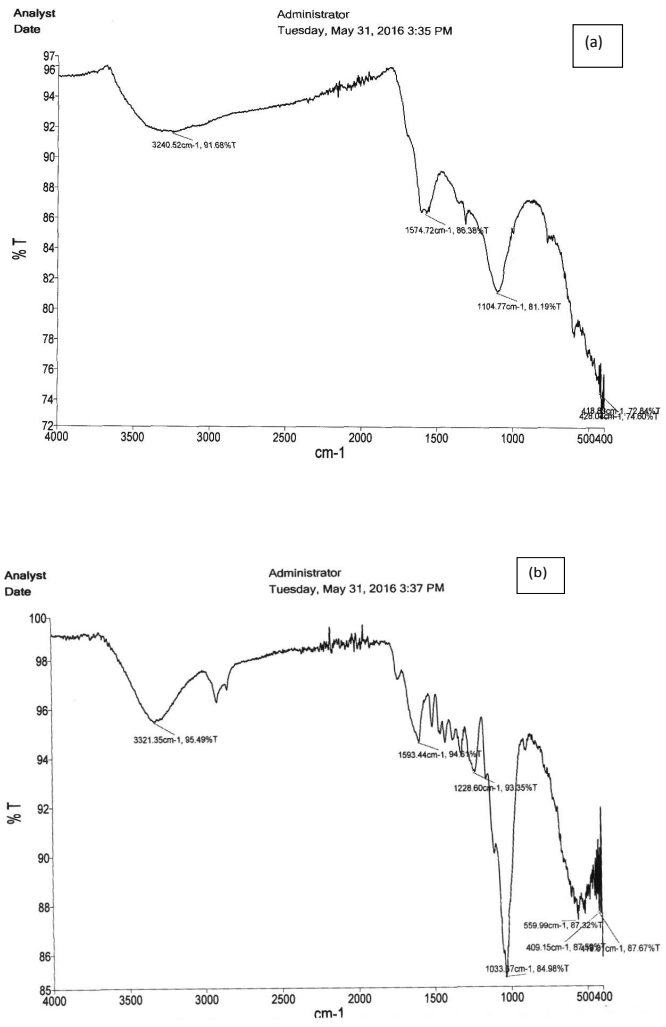

Figure 2. (a)Fourier Transform Infrared (FTIR) Spectrum of SAMS. (b) Fourier Transform Infrared (FTIR) Spectroscopy Micrograph of sawdust before modification

3.4. Diagnostic Case Study

The results of the diagnostic case study are presented in Table 4. The actual values on the table indicate the percentage of dye removed and the predicted values indicate the standard generated by the response surface. The residual shows the closeness of the actual

Table 3. Analysis of variance for experimental responses of Crystal violet in mixture

Source	Sum of Squares	Degree of Freedom	Mean Square	F Value	Prob > F	Comment
Model	240.8611	19	12.6769	148.7354	<0.0001	Significant
Linear mixture	80.8211	3	26.94037	316.0856	<0.0001	
AE	16	1	16	187.7246	<0.0001	
AF	4	1	4	46.93114	<0.0001	
AG	4	1	4	46.93114	<0.0001	
AH	16	1	16	187.7246	<0.0001	
BE	16	1	16	187.7246	<0.0001	
BF	4	1	4	46.93114	<0.0001	
BG	4	1	4	46.93114	<0.0001	
BH	16.04003	1	16.04003	188.1942	<0.0001	
CE	16	1	16	187.7246	<0.0001	
CF	4	1	4	46.93114	<0.0001	
CG	4	1	4	46.93114	<0.0001	
CH	16	1	16	187.7246	<0.0001	
DE	16	1	16	187.7246	<0.0001	
DF	4	1	4	46.93114	<0.0001	
DG	4	1	4	46.93114	<0.0001	
DH	16	1	16	187.7246	<0.0001	
Residual	3.750175		0.085231			
Cor Total	244.6113					

values to the predicted values. Negative values of residuals indicate that the actual value is greater than the predicted value while the positive value indicates that the predicted value is greater than the actual value. Residual value of zero is an indication that the actual amount is equal to the predicted value on which the comparison is made.

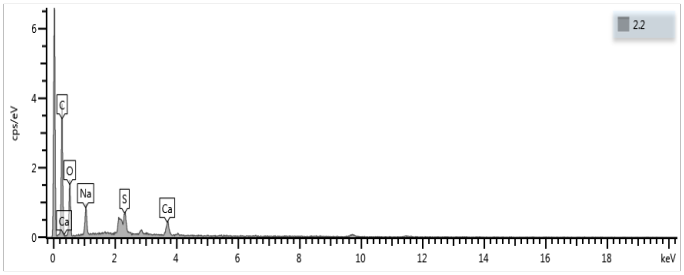

Figure 3. Elemental Diffraction X-ray Spectrum of SAMS

3.5. Main and interaction effects of variables

Table 3 shows the linear mixture for the process parameters and dye mixture. Every process parameter and mixture component is significant as shown in Table 3. The effect of various experimental conditions (adsorbent dose, pH, contact time, temperature and interactive effect the compostion of dye mixture) on percentage removal of dye is shown in Figure 4. The percentage removal increased with increase in adsorbent dose (Figure 4a). This is as a result of increase in adsorption site with increasing amount of the adsorbent. Similar observations were reported by other studies (Hassanein and Koumanova, 2010; Giwa et al., 2013). The percentage removal of the dye also increased with increase in pH (Figure 4b) as observed in other study (Sharma and Uma, 2010). The lower adsorption of CV observed at acidic pH may

Table 4. Diagnostic case study of crossed composite design matrix for adsorption of CV from dye mixture

Standard Order	Actual Value	Predicted Value	Residual	Student Residual	Cook's Distance	Outlier
1	93.89	93.89	0	0	0	0
2	95.89	95.89	0	0	0	0
3	94.89	94.89	0	0	0	0
4	96.84	96.84	0	0	0	0
5	94.84	94.84	0	0	0	0
6	96.89	96.89	0	0	0	0
7	95.89	95.89	0	0	0	0
8	97.89	97.89	0	0	0	0
9	95.89	95.89	0	0	0	0
10	97.89	97.89	0	0	0	0
11	96.89	96.89	0	0	0	0
12	98.89	98.89	0	0	0	0
13	96.89	96.89	0	0	0	0
14	98.89	98.89	0	0	0	0
15	97.89	97.89	0	0	0	0
16	99.89	99.89	0	0	0	0
17	93.85	93.85	0	0	0	0
18	95.85	95.85	0	0	0	0
19	94.85	94.85	0	0	0	0
20	96.85	96.85	0	0	0	0
21	94.85	94.85	0	0	0	0
22	96.85	96.85	0	0	0	0
23	95.85	95.85	0	0	0	0
24	97.85	97.85	0	0	0	0
25	95.85	97.85	0	0	0	0
26	97.85	97.85	0	0	0	0
27	96.85	96.85	0	0	0	0
28	98.85	98.85	0	0	0	0
29	96.85	96.85	0	0	0	0
30	98.85	98.85	0	0	0	0
31	97.85	97.85	0	0	0	0
32	99.85	99.85	0	0	0	0
33	93.87	93.86875	0.00125	0.005164	6.06E-07	0.00510484
34	95.87	95.86875	0.00125	0.005164	6.06E-07	0.00510484
35	94.87	94.86875	0.00125	0.005164	6.06E-07	0.00510484
36	96.87	96.86875	0.00125	0.005164	6.06E-07	0.00510484
37	94.87	94.86875	0.00125	0.005164	6.06E-07	0.00510484
38	96.87	96.86875	0.00125	0.005164	6.06E-07	0.00510484
39	95.86	95.86875	-0.00875	-0.03615	2.97E-05	0.03573441
40	97.87	97.86875	0.00125	0.00516	6.06E-07	0.00510484

Table 4. Contd.

41	95.87	95.87125	-0.00125	-0.00516	6.06E-07	0.00510484
42	97.87	97.87125	-0.00125	-0.00516	6.06E-07	0.00510484
43	96.67	96.87125	-0.00125	-0.00516	6.06E-07	0.00510484
44	98.87	98.87125	-0.00125	-0.00516	6.06E-07	0.00510484
45	96.87	96.87125	-0.00125	-0.00516	6.06E-07	0.00510484
46	98.87	98.87125	-0.00125	-0.00516	6.06E-07	0.00510484
47	97.88	97.87125	0.00875	0.036147	2.97E-05	0.03573441
48	99.87	99.87125	-0.00125	-0.00516	6.06E-05	0.00510484
49	90.9	91.275	-0.375	-1.54916	0.0545329	1.57500692
50	93.99	93.275	0.625	2.581929	0.1515081	2.77094733
51	92.9	92.275	0.625	2.581929	0.1515081	2.77094733
52	93.9	93.275	-0.375	-1.54916	0.0545329	1.57500692
53	92.9	92.275	0.625	2.581929	0.1515081	2.77094733
54	93.9	93.275	-0.375	-1.54916	0.0545329	1.57500692
55	92.9	92.275	-0.375	-1.54916	0.0545329	1.57500692
56	94.9	94.275	-0.375	-1.54916	0.0545329	1.57500692
57	92.9	92.275	-0.375	-1.54916	0.0545329	1.57500692
58	95.9	95.275	0.625	2.581929	0.1515081	2.77094733
59	93.9	93.275	-0.375	-1.54916	0.0545329	1.57500692
60	95.9	95.275	-0.375	-1.54916	0.0545329	1.57500692
61	93.9	93.275	-0.375	-1.54916	0.0545329	1.57500692
62	95.9	95.275	-0.375	-1.54916	0.0545329	1.57500692
63	95.9	95.275	0.625	2.581929	0.1515081	2.77094733
64	97.9	97.275	0.625	2.581929	0.1515081	2.77094733

be due to the presence of excess H^+ ions competing with the cationic dye species for adsorption sites. The surface charge density decreased with an increase in the solution pH, leading to a reduced electrostatic repulsion between the dye particles and the surface of the adsorbent (Wang et al., 2005).

Increase in contact time resulted in increase in the percentage removal of the dye (Figure 4c). This is because as time increases, it enhances the interaction between the surface of the adsorbent and the dye which results in more uptake of dye molecules. Similar observation was reported by other authors (Jabli et al., 2011; Giwa et al., 2015). The effect of temperature on percentage removal of dye is shown in Figure 4d. The percentage removal increased with increase in temperature (Singh and Srivastava, 2001). As it increases, the dye molecules acquire more energy and move faster thereby resulting in increased adsorption. The increase in percentage removal with increasing temperature may also be due to increased volume of pores on the adsorbent which allows the penetration of more dye molecules (Verma and Mishra, 2010). Figure 4e depicts the interactive effect of concentration of the dye mixture components on the uptake of crystal violet. The percentage removal of CV is high at a high concentration of MB but low at a low concentration of MG and RB.

3.6. Optimization

The optimal values of the experimental variables were obtained to determine the specific combination that maximizes the dye removal efficiency. The results showed that the optimal values of pH, SAMS dose, temperature and contact time for the adsorption of 68.39 mg/L of crystal violet dye, CV (% R =97.97) are 9.94, 0.99 g, 60^{o}C and 275.10 minutes respectively, in a quaternary dye mixture consisting of 11.65 mg/L MB, 10 mg/L MG and 39.96 mg/L RB.

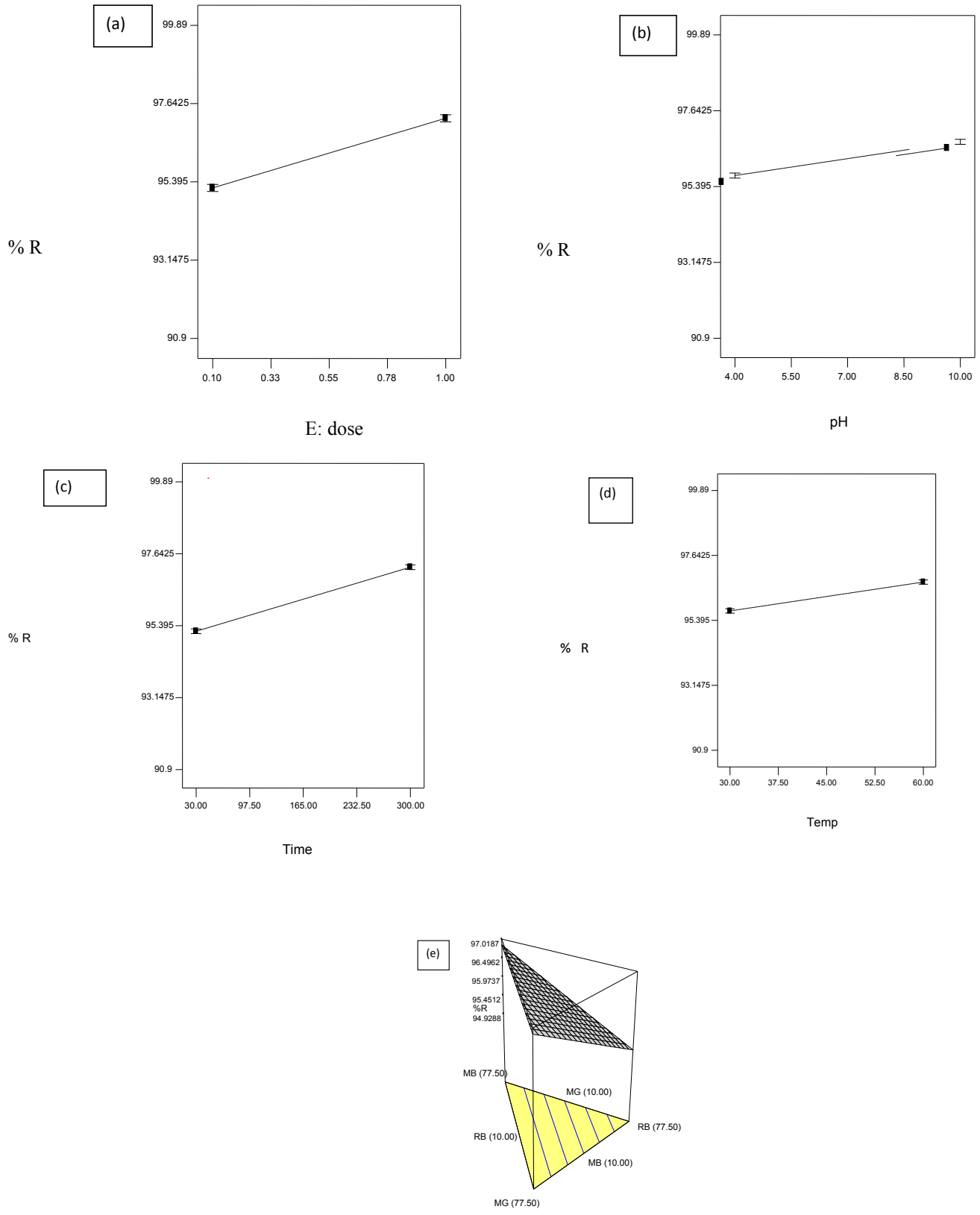

Figure 4. (a) Effects of adsorbent dose (b) Effects of pH (c) Effects of contact time on the removal of crystal violet (d) Effects of temperature on the removal of crystal violet (e) Response surface plot of dye removal (%) showing interactive effect of concentration of the dye mixture.

4. CONCLUSIONS

The adsorption of crystal violet (CV) from quaternary aqueous mixture comprising of Methylene Blue (MB), Malachite green (MG), and Rhodamine B (RB), onto sulphuric acid-charred sawdust of *Parkia biglobosa* (SAMS) was investigated using crossed composite design of Design of Experiments (DoE) with a total of 64 experiments. The combined effects of mixture components (CV, MB, MG, RB) and process parameters (time, pH, adsorbent dosage and temperature) on the dye adsorption was determined and optimized using response surface methodology. The optimum contact time, pH, adsorbent dosage and temperature were found to be 275.10 min, 9.94, 0.99g, 60^{o}C respectively for the maximum decolorization of 68.39 mg/L CV (97.2%) in the presence of 11.65 mg/L MB, 10.00 mg/L MG and 39.96 mg/L RB. A linear model was obtained for dye decolourization through this design. The experimental values were in good agreement with predicted values and the model developed was highly significant, the correlation coefficient being 0.985. Experimental results were analyzed by Analysis of variance (ANOVA) statistical concept.

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