

OPTIMIZATION OF PROCESS PARAMETERS FOR ADSORPTION OF CHROMIUM (VI) FROM TIE AND DYE INDUSTRY WASTEWATER USING BANANA PEELS ACTIVATED CARBON: RESPONSE SURFACE METHODOLOGY APPROACH

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ABSTRACT

Recent research has linked the use of synthetic adsorbents for wastewater treatment to potential risks of neurotoxic and neurodegenerative diseases. With reference to this, the current study focused on the use of an agricultural by-product and eco-friendly natural activated adsorbent, banana peels for the treatment of tie and dye industrial wastewater. In a bid to obtain an optimum percentage amount of Chromium (VI) adsorbed by the adsorbent and the permissible level of Chromium (VI) that should be discharged to the environment, optimization of process variables affecting the adsorption of metal ion was undertaken using a Response Surface Methodology (RSM). Four parameters were carried out viz; mass of adsorbent, pH of the solution, temperature and contact time and their effects on the adsorption of Chromium (VI) on the tie and dye wastewater were established. The obtained experimental data was fitted to a quadratic model that was also validated. The RSM model predicted 95.496 % as value of Chromium (VI) adsorbed with optimal conditions of 71.0 °C, 5.41, 76.58 minutes and 1.009 g for temperature, pH, contact time, and mass of adsorbent respectively. The predicted optimal conditions were validated in triplicates in the laboratory with 96.5 % of Chromium (VI) adsorbed. The study revealed the efficacy of Banana Peels Activated Carbon (BAPC) as agricultural waste adsorbent for removal of Chromium (VI) metal ion from tie and dye wastewater.

Keywords: Tie and dye wastewater, adsorption, optimization, RSM.

INTRODUCTION

The importance of water to human life, flora and fauna cannot be overemphasized. It was reported by Onifade and Ilori, (2008), that about 70 % of human body weight is made up of water. However, access to this clean and safe drinking water is of great concern to the Nigerian government and the entire world which necessitated the sustainable development goal (SDG – 6) that seeks to ensure safe drinking water and sanitation for all, focusing on sustainable management of water resources, wastewater and ecosystems, and acknowledging the importance of the environment. The inaccessibility of potable drinking water and the dearth of a

sufficient supply of safe water has been a critical task that required awareness in developing countries like Nigeria and Africa as a whole. Water treatment is very essential for water to be within the consumption standard (Baptista et al., 2015). There are numerous techniques for water treatment processes such as coagulation and flocculation, distillation, reverse osmosis separation and adsorption process. The most usual approach is the use of inorganic coagulants such as alum for coagulation while the use of synthetic activated carbon or carbon black are widely used for adsorption process.

In the environment, Chromium is mostly stable in its trivalent and hexavalent forms but Chromium in its zero oxidation state is biologically inert and does not occur naturally in the earth's crust, whereas Cr⁺³ and Cr⁺⁶ are derived from industrial sources. The available forms of chromium are halides, oxides, and sulfides. Cr (VI) gets reduced to Cr (III) in the lower respiratory tract by pulmonary alveolar macrophages (Gao et al., 2017). Chromium (VI) enters the bloodstream, where it is taken up by Red Blood Cells (RBCs), reduced, and bound to haemoglobin.

There are three excretion half-lives for Cr (VI): 7 hours, 15-30 days, and 3-5 years (Kirti Shekhawat et al., 2015). In some recent studies, Gorzin and Abadi (2018) used paper mill sludge as an adsorbent to remove Cr (VI) from aqueous solutions, Khan et al. (2017) investigated how chemical adsorption could increase the effectiveness of banana peels as an adsorbent for the treatment of wastewater.

Also, Subashree et al. (2017), Priyatharishini et al. (2019), and Ibrahim et al. (2012), investigated banana and lemon peels for the treatment of wastewater, the effectiveness of banana peels as a coagulant in reducing turbidity in synthetic wastewater, and bio-sorption of Cr (VI) from wastewater onto maize cobs respectively. However, none of these authors have optimized the process factors affecting the adsorption of Cr (VI) from tie and dye industrial wastewater using Banana Peels Activated Carbon (BPAC).

Modeling and optimization are of utmost priority in any process unit as they tend to improve the yield and maximize the performance of the process with resultant less waste. The use of a One Factor At a Time (OFAT) is very obsolete, time-consuming, large number of experiments and does not give level of interactions between the other variables in a process. RSM is a method of optimization that is

made up of experimental design, modeling, analysis through the partial regression fitting of the experimental factors (Wang et al., 2011 and Adesina et al., 2019).

The RSM can combine many variables at a time and display reciprocal interaction on the response of a process, RSM also decreases the number of investigational runs needed to offer adequate knowledge for acceptable results statistically (Adesina et al., 2019)

This contemporary work optimized the process factors that have been reported already in literature affecting the adsorption of Cr (VI) from wastewater (Subashree et al., 2017; Priyatharishini et al., 2019; and Ibrahim et al., 2012). In a bid to maximize the amount of Cr (VI) adsorbed from tie and dye wastewater onto BPAC. This study ascertained the process condition in maximizing the percentage amount of Cr (VI) adsorbed by BPAC from tie and dye wastewater which can be adapted to industrial and local wastewater treatment.

MATERIALS AND METHOD

Materials

The tie and dye wastewater was obtained at a different discharged point from the tie and dye factory (Adire) in Ogun State while the banana peel sample used as a precursor for the activated carbon adsorbent preparation in this study was obtained from Ekiugbo market at Iyede in Isoko North Local Government Area of Delta State, Nigeria.

The chemicals used are 85% phosphoric acid (H₃PO₄) (for the chemical activation of the adsorbent), sodium hydroxide (for pH adjustment), and hydrochloric acid (for pH adjustment). The chemicals are of analytical grades (Sigma Aldrich), procured from an accredited chemical dealer in Effurun, Delta State, Nigeria. The chemicals are used without any other purification.

Methods

Preparation Of Precursor (Banana Peels, Bps)

Banana peel samples were washed with water to remove the debris and sun-dried for two days. Thereafter, dried in an oven (DHG-9053) for 24 hours at 103 °C to remove any inherent moisture content. The BPs were crushed using a laboratory blender (LG-3002) and sieved to 250 μm prior to carbonization and activation (Afshin et al., 2021).

Carbonization And Activation Of The Banana Peels (Adsorbent)

The milled BPs were carbonized using the method of Olaoye et al., (2018) which was modified. 500 g of the prepared dried banana peel was charged into a muffle furnace to carbonize at 600 °C for 1 hour and subsequently cooled in a desiccator. At room temperature, 0.5 M of phosphoric acid was used to chemically activate the carbonized material. The impregnation ratio for the activating agent and the precursor was 2:1. This was left to stand for 24 hours and stirred with a magnetic stirrer for 30 minutes. This Banana Peel Activated Carbon (BPAC) was dried in the oven at 103 °C for 12 hours before and thereafter classified with a sieve into 0.25 mm. It was then kept in an airtight container prior to use (Afshin et al., 2021).

Batch Adsorption Procedure

100 ml of wastewater effluent sample with different experimental conditions (Table 1) was put into the conical flask, certain dosage of BPAC as suggested by the design of the experiment was added. The mixture was then agitated using a magnetic stirrer, after the completion of the reaction, the samples were then filtered using 0.4 μm of Whatman PTFE filter paper and the filtrate collected was analyzed for change in concentration of Chromium (VI) by measuring the change in absorbance using a UV-Vis spectrophotometer (Spectrumlab 755s) at a

wavelength of 480 nm. The metal ions uptake on the adsorbent and percentage removal of metal ions were calculated by applying equations (1) and (2), respectively:

$$q = \frac{(c_o - c_f)}{m} * V \quad (1)$$

$$\% \text{ metal ion removal} = \frac{(c_o - c_f)}{c_o} * 100 \quad (2)$$

Where C_o is the initial ion concentration in contact with the adsorbent (mg/L), C_f is the metal ion concentration at time t , or the metal ion concentration following the batch adsorption process, V is the volume of aqueous solution (adsorbate) in contact with the adsorbent in (L), and m is the adsorbent dosage in (g).

Optimization studies of adsorption of chromium (vi) from tie and dye wastewater.

Optimization of the process variables affecting the adsorption of Chromium (VI) from tie and dye wastewater was carried out using RSM. Four variables were varied viz; mass of adsorbent, pH of the solution, temperature, contact time and their effects on the amount of Chromium (VI) adsorbed on the tie and dye wastewater was examined. A set of 29 experimental runs were made using Box Behnken Design (BBD). The model fitness was evaluated via a test of significance and Analysis of Variance (ANOVA). The chosen process variables temperature, contact time, pH, and mass of adsorbent represent X_1 , X_2 , X_3 , and X_4 respectively. The method of coding has been reported by Betiku and Adesina (2013) and Adesina et al., (2019) as shown in Table 1, the choice of the process variables selected in Table 1 was arrived at after literature review. The multiple regressions were used to fit the coefficient of the polynomial model of the amount of chromium (VI) adsorbed (response) which is shown in Eq 3.

$$Y = b_o + \sum_{i=1}^k b_i X_i + \sum_{i=1}^k b_{ii} X_i^2 + \sum_{i<j}^k b_{ij} X_i X_j + e \tag{3}$$

where Y is the amount of chromium adsorbed, b_o is the intercept, b_{ij} is the interaction effect, and b_{ii} is the quadratic coefficients of x_i and e is the randomized error. Design Expert 11 software was used to design and analyze the experimental data. Design Expert is a statistical software package that performs the design of experiments, comparative tests and optimization of process variables. It also contains the graphical tools that can help to know the impact of each parameter considered on the yield of the process.

Table 1: Experimental factor codes and levels

Variable	Symbol	Coded variable levels		
		-1	0	+1
Temperature, °C	X ₁	30	55	80
Contact time (mins)	X ₂	20	70	120
pH	X ₃	2	5	8
Mass of adsorbent, (g)	X ₄	0.5	1.50	2.50

Characterization of the adsorbent (bpac)

Scanning Electron Microscope (sem)/energy Dispersive x - rays

The surface morphology of the sample (BPAC) was analyzed with the aid of a 52-cm scanning electron microscope (SEM) at a magnification of 100 μm and 10 μm. The EDX gives an overall map of the sample by analyzing the near-surface elements and estimating the elemental proportion at different positions. EDX is used in conjunction with SEM.

When an electron beam with an energy of 10–20 keV contacts the surface of the conducting sample, the energy of the X-rays that are emitted depends on the material being investigated. This was carried out in the Chemical Analysis Laboratory of the Department of Chemical Engineering at Ahmadu Bello University, Zaria.

Characterization of the Wastewater

The wastewater pH was carried out using a pH meter model HI 2215 which was used to measure the alkalinity and acidity of the wastewater. The pH meter was calibrated with buffer solutions of 4.00, 7.0, and 10.0 before use. A portable conductivity meter DDS-307 was used to measure the conductivity of the waste water sample. A thermostatic sieve shaker HY – 4A cycling vibrator was used for the agitation of the wastewater sample. The turbidity of the tie and dye wastewater sample was determined using JBN.911010 portable turbidity meter. The dissolved oxygen (DO) of the wastewater sample was determined using DO meter model JPB-607A.

RESULTS AND DISCUSSION

The chemical composition of tie and dye wastewater effluent as presented in Table 2 and in comparison to Table 3 shows that the chromium level in the tie and dye effluent water was found to be above the permissible limit for any industrial wastewater that can be discharged into the water body which necessitated this study. Figure 1a gives the SEM micrograph of the raw Banana Peels before treatment which demonstrates a very fine suspended particle in the wastewater. Figure 1b shows the SEM micrograph of the banana peel activated carbon (BPAC) which displays the presence of pores of varying sizes on the surface of the activated carbon, this is a sign of its potential as an adsorbent. Figure 1c describes the SEM micrograph of the BPAC after

adsorption of metal ions agglomeration of the loosely bonded particle was noticed which resulted

Table 2: Chemical Composition of Tie and dye wastewater

Parameters	Results	
	Raw WW	Treated WW
pH	9.32	7.2
Conductivity, $\mu\text{s}/\text{cm}$	223	100
Temperature, $^{\circ}\text{C}$	26.90	27
DO, mg/l	1.30	4.50
Specific gravity	1.007	1.005
Density, g/cm^3	1.0046	1.0046
Chromium, mg/l	1.610	0.041
Turbidity, NTU	5.60	3.85

Table 3: WHO, USEPA and NESREA Permissible Limits for Heavy Metal ions

Heavy metals Metals, (ppm)	Permissible limits		
	WHO	US EPA	NESREA
As (III)/As (V)	0.05	0.01	0.1
Pb(II)	0.05	0.015	0.01
Cd(II)	0.005	0.005	0.1
Cr(VI)/Cr(III)	0.05	0.05	0.05
Hg(II)	0.001	0.002	0.01
Zn(II)	5.00	5.00	3.00
Cu(II)	1.50	1.30	1.0
Co(II)	0.01	-	-
Ni	0.1	-	0.1

Source: (WHO, 2004; Griffiths et al., 2012, Butu et al., 2022; Egbere et al., 2023).

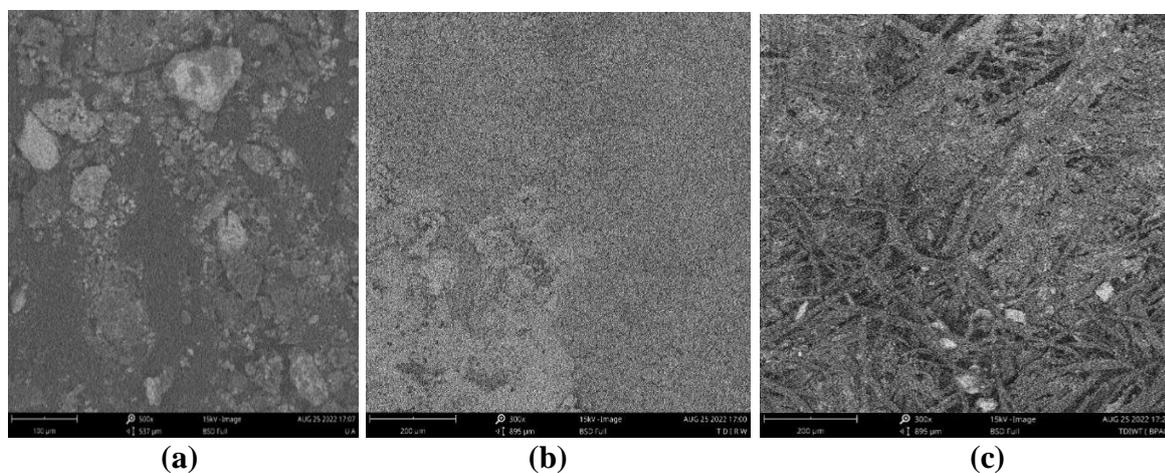


Figure 1 SEM micrograph of (a) Raw Plantain Peels (b) Activated BPAC (c) BPAC after Cr (IV) adsorption

from adsorption of Chromium (VI) from the tie and dye wastewater

Optimization Of Cr (Vi) Adsorption Process Of Tie And Dye Wastewater

The observed, predicted, and residual values of the experiment are presented in Table 4. The low residual values indicate a good fit between the model and the experimental data. Table 5 shows the results of the ANOVA test and the regression coefficients of the significant variables. The F-value of 21.17 (with a p-value much less than 0.0001) indicates that the model is statistically significant. The achieved R² value of 0.9549 indicates a good fit for the model. This suggests that 95.49% of the variation in the dependent variable within the sample can be attributed to the independent variable.

Therefore, the obtained model could be used for theoretical predictions of the dependent variable, as supported by previous research (Betiku and Adesina, 2013; Adesina et al., 2019). Table 5 further details the results of a significance test for each regression coefficient. The results revealed that the p-values of the model terms X₃, X₄, X₃X₄, X₃², and X₄² were significant at 95 % confidence level (p< 0.05). The model equation for the prediction of Cr (VI) adsorbed from the tie and dye wastewater is expressed as Eq. (4).

$$\% \text{ Cr (VI) adsorbed, } Y = 90.77 + 0.7225X_1 + 1.41X_2 + 12.74X_3 - 14.01X_4 - 0.0550X_1X_2 + 0.3925X_1X_3 + 0.015X_1X_4 - 1.54X_2X_3 + 0.1325X_2X_4 + 19.60X_3X_4 - 0.4206X_1^2 - 2.14X_2^2 - 10.09X_3^2 - 11.69X_4^2 \tag{4}$$

Table 4: Actual, predicted, and residual values for experimental data for adsorbed Cr (VI)

S/N	Temperature (°C)	Contact time (mins)	pH	BPAC dosage (g)	%Actual Cr (VI)	% Predicted Cr (VI)	Residual
1	55	70	8	0.5	82.65	76.14	6.51
2	80	70	8	1.5	89.55	94.11	-4.56
3	55	70	5	1.5	93.65	90.77	2.88
4	80	20	5	1.5	92.09	87.58	4.51
5	55	20	5	0.5	89.91	89.67	0.2358
6	55	70	5	1.5	91.88	90.77	1.11
7	55	70	2	2.5	23.77	22.64	1.13
8	55	120	8	1.5	89.45	91.15	-1.70
9	55	70	8	2.5	95.23	87.32	7.91
10	55	120	5	2.5	60.67	64.48	-3.81
11	80	120	5	1.5	94.02	90.29	3.73
12	30	70	5	0.5	89.45	91.96	-2.51
13	55	20	2	1.5	60.48	62.85	-2.37
14	30	70	8	1.5	87.62	91.88	-4.26
15	55	70	5	1.5	83.9	90.77	-6.87

16	80	70	2	1.5	68.54	67.85	0.6925
17	55	70	2	0.5	89.6	89.86	-0.2613
18	55	120	5	0.5	91.07	92.23	-1.16
19	55	70	5	1.5	89.76	90.77	-1.01
20	30	70	5	2.5	62.65	63.91	-1.26
21	80	70	5	2.5	63.83	65.39	-1.56
22	55	20	5	2.5	58.98	61.39	-2.41
23	30	20	5	1.5	89.94	86.02	3.92
24	55	120	2	1.5	68.56	68.75	-0.1879
25	55	70	5	1.5	94.67	90.77	3.90
26	30	70	2	1.5	68.18	67.19	0.9925
27	55	20	8	1.5	87.52	91.41	-3.89
28	30	120	5	1.5	92.09	88.96	3.13
29	80	70	5	0.5	90.57	93.38	-2.81

Table 5: ANOVA and Regression coefficient test of significance

Source	Sum of Squares	Df	Mean Square	F – value	P – value
Model	7254.67	14	518.19	21.17	<0.0001
Residual	342.76	14	24.48		
Lack of fit	269.80	10	26.98	1.48	0.3762
X ₄	2355.36	1	2355.36	96.21	<0.0001
Pure error	72.95	4	18.24		
X ₃ X ₄	1537.03	1	1537.03	62.78	< 0.0001
X ₃ ²	660.78	1	660.78	26.99	0.0001
X ₄ ²	886.32	1	886.32	36.20	< 0.0001
Cor Total	7597.43	28	R ² =0.9549	Adj.R ² =0.9098	

Figure 3 - 8 gives the surface plot for the adsorption of Cr (VI) using the BPAC adsorbent from tie and dye wastewater. The curvature nature of Figures 3 and 5 indicates the interface between the response (% Cr (VI) amount adsorbed) and the process variables studied in this work. This also confirms the significance of the adsorbent dosage, the interactive terms (pH and adsorbent dosage), and the

quadratic term (pH) as depicted in Table 5. Figure 3 shows a decrease in bio-sorption of Cr (VI) with increase in pH levels and adsorption dosage. This is because Cr (VI) exists in solution as HCrO⁴⁻, Cr₂O₇²⁻ and CrO₄²⁻ at lower pH, the adsorbent surface (Activated Banana Peels) is also protonated and there is a strong electrostatic attraction between the positively charged adsorbent surfaces with

oxyanions of Chromium (VI) (Hamadeen et al., 2022). Higher pH results in the negatively charged surface of the adsorbent becoming less reactive with

these ions (HCrO_4^- , $\text{Cr}_2\text{O}_7^{2-}$, and CrO_4^{2-}), and higher pH also results in an abundance of hydroxyl ions in the aqueous solution (Ali et al., 2016).

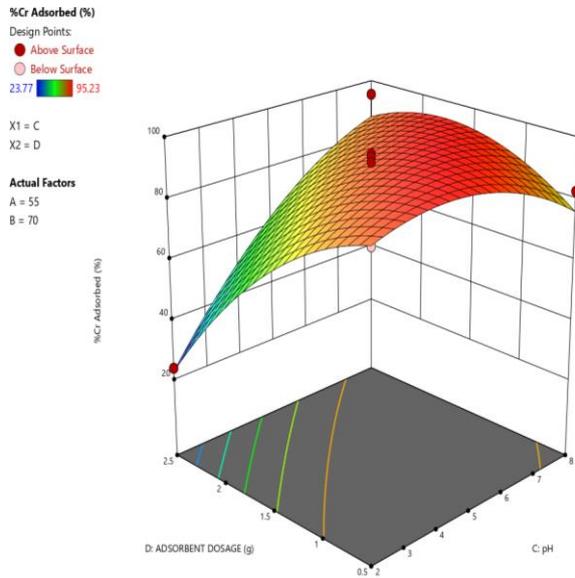


Figure 3 Amount of chromium adsorbed (%) as a function of adsorbent dosage and pH

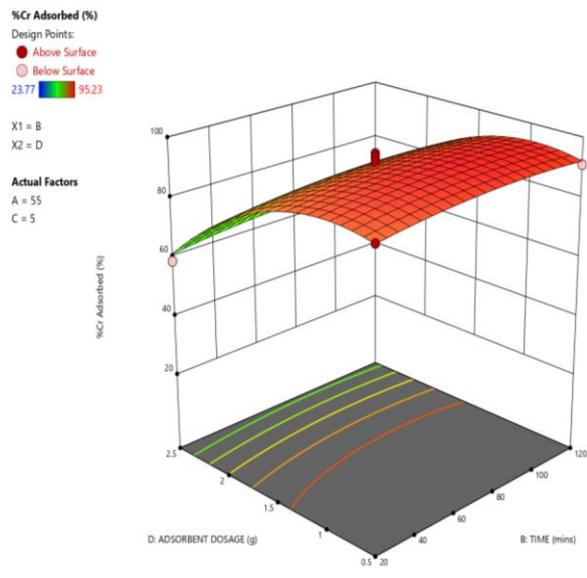


Figure 4 Amount of chromium adsorbed (%) as a function of adsorbent dosage and contact time

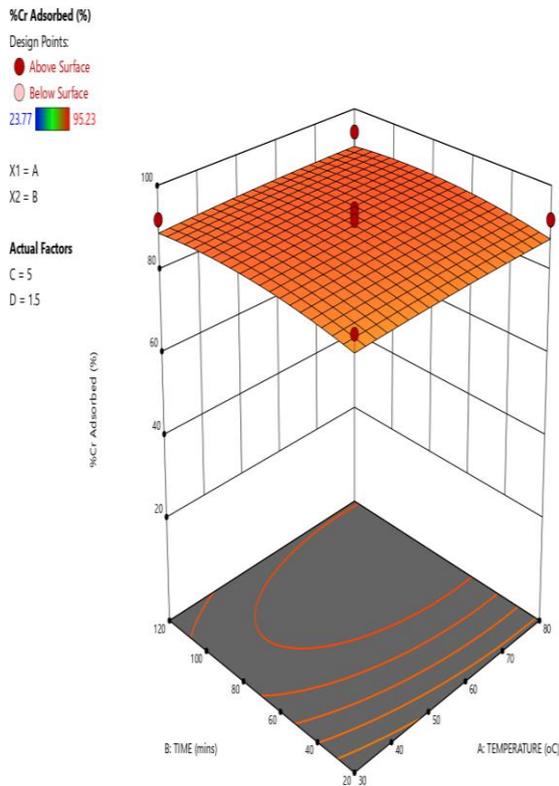


Figure 6 Amount of chromium adsorbed (%) as a function of contact time and temperature

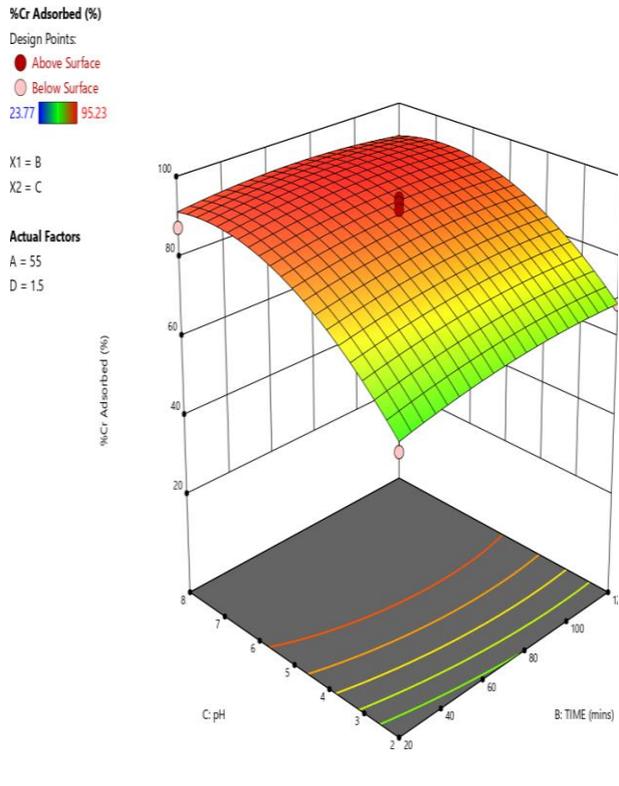


Figure 5 Amount of chromium adsorbed (%) as a function of pH and contact time

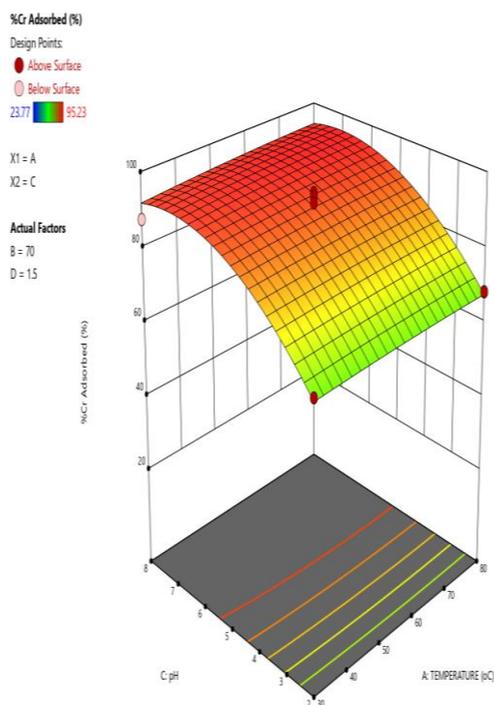


Figure 7 Amount of chromium adsorbed (%) as a function of temperature and pH.

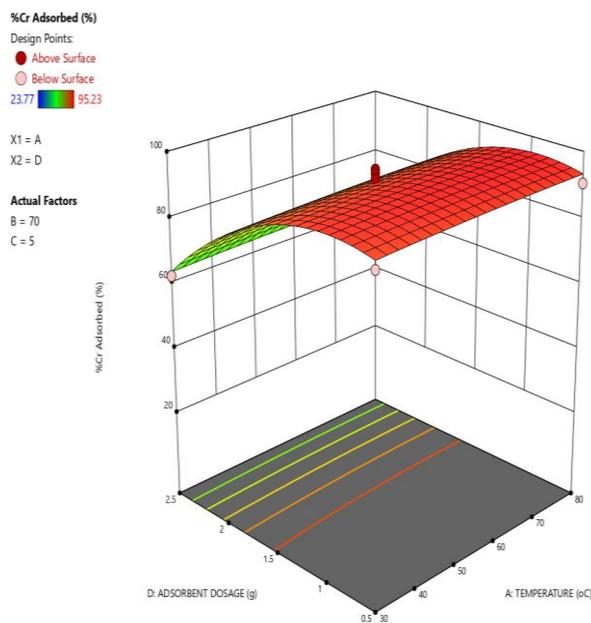


Figure 8 Amount of chromium adsorbed (%) as a function of adsorbent dosage and temperature

It was observed in Figure 4 that an increase in both contact time and BPAC dosage favours a high amount of Cr (VI) adsorbed. As the change in the value of pH has a significant effect on chromium adsorbed, the trend of Figure 3 was also noticed in Figure 5. An increase in both contact time and temperature corresponds to a high amount of Chromium (VI) adsorbed by BPAC as seen in Figure 6. This is a result of a high collision of adsorbed metal ions molecules as a result of high temperature caused by lowering the energy barrier. There was a high mutual interaction between pH and the amount of chromium adsorbed as shown in Figure 7. It was also observed that as the temperature and adsorbent dosage increase, the quantity of chromium adsorbed increases correspondingly as depicted in Figure 8. This shows that more active sites are now available in the BPAC for Cr (VI) adsorption and the rise in temperature speeds up the reaction process for the chromium

adsorption. Suganya et al., (2019), noted that an increment in temperature increases the adsorption of Chromium ions. A similar observation was also reported by Badessa et al., (2020). The increment in the bio-sorption of Chromium (VI) as the temperature increases is an indication that the process is endothermic (Ali et al 2019). Similar findings were also reported by Sharma et al., (2018).

The optimization tool of Design Expert 11 software was used for the optimization. The purpose of the optimization was to optimize the % amount of Cr (VI) adsorbed using the constraint indicated in Table 7. The optimal process conditions as obtained from the software were 71.0 °C, 5.41, 76.58 minutes and 1.009 g for temperature, pH, contact time, and BPAC adsorbent dosage respectively which gave 95.496 % value of Chromium (VI) adsorbed as optimum. In other to validate this optimum process variable as predicted, three replicates of the optimal

process conditions were experimented in the laboratory, the percentage of Cr (VI) adsorbed achieved was 96.5 %. The obtained optimum condition from this study could be scaled up and applied to other related industrial wastewater effluent.

CONCLUSION

Modeling and optimization of Cr (VI) adsorption process using BPAC were undertaken in this work. The effects of process variables such as temperature, contact time, pH, and BPAC dosage on the amount of chromium adsorbed were carried out using the Response Surface Methodology (RSM). It was ascertained that pH and BPAC dosage are the significant process variables in maximizing Cr (VI) adsorbed from tie and dye wastewater. The experimental data obtained fitted well with the second-order mathematical model used. A contact time value of 76.58 minutes, temperature value of 71.0 °C, pH value of 5.41, and BPAC value of 1.009 g were the optimum conditions established. The optimum process condition was validated in three replicates in a laboratory experiment and 96.5 % Cr (VI) adsorbed was achieved.

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