#### **ORIGINAL ARTICLE**



# Application of response surface methodology for optimization of dissolved solids adsorption by activated coal

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#### Abstract

The adsorptive removal of total dissolved solids by activated coal using response surface methodology was investigated. A four-variable central composite experimental design was applied to correlate the adsorption variables (effluent pH, adsorbent dosage, contact time, and adsorption temperature). The adsorption variables were optimized based on the removal of total dissolved solids from fibre cement industry effluent. Three-dimensional surface plots were generated to estimate the effect of the combinations of the independent variables on the adsorption efficiency. The results of the model validation gave experimental yield 96.2%, predicted yield 96.5% obtained at effluent pH 6.27, adsorbent dosage 27.60 mg L<sup>-1</sup>, contact time 48.00 min, and adsorption temperature of 31.00 °C. The good agreement found between observed and predicted values supports the suitability of the applied model to predict the adsorption treatment.

 $\textbf{Keywords} \ \ Surface \ plots \cdot Optimization \cdot Dissolved \ solids \cdot Adsorbent \cdot Experimental \ design \cdot Response \ surface \ methodology$ 

### Introduction

Coal is an organic sedimentary rock consisting of a complex mixture of substances, organic and mineral, derived from plant debris deposited in the earth's crust. Coal can be used to produce activated carbon resulting from the carbonization of the substance. This process involves the heat treatment of coal in an inert atmosphere to required temperature leading to increase in carbon contents and decreases in contents of heteroatoms (Harry and Francisco 2006). Available data show that coal occurrences in Nigeria have been indicated in more than 22 coal fields (Falade and Adeyeye 2016). Nigerian coal is one of the most bituminous in the world owing to its low sulphur and ash content and therefore

rated environmentally friendly (Adedinni 2018). This has prompted research into conventional and non-conventional uses of the substance. Coal is used for heating, electricity generation, and as precursor for numerous industrial chemicals. Activated carbon adsorbent has been produced from agricultural by-products such as rice husk and rubber seed shell (Okiemen et al. 2004), coconut shell and cattle bone (Baba and Saba 2006), corncob (El-Sayed et al. 2014), kenaf core fibre (Shamsudinn et al. 2016), plantain (*Musa paradisiaca*) fruit stem (Ekpete et al. 2017).

The limitation of fresh water resources in most parts of Nigeria has necessitated the use of unconventional water resources. Wastewater effluent is one of these unconventional sources. However, the environmental impacts of wastewater reuse should be carefully studied (Abedi et al. 2013). The discharge of effluent contaminated with dissolved solids to the environment is distressing for both toxicological and aesthetical reasons as dissolved solids impede light penetration, damage the quality of the receiving streams and are toxic to food chain organisms. Since dissolved solids are composed of cations (calcium, magnesium, lead, etc.) and anions (carbonates, chlorides, phosphates, etc.), it is difficult to biodegrade when discharged into waste streams. Adsorption has been successfully employed for the removal of organic constituents (Muyibi et al. 2014; Amosa et al. 2016, Akpomie 2018), and inorganic

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contents such as dissolved gases, dissolved solids, suspended solids, turbidity and colour (Alkhatib et al. 2014). A total dissolved solid is a parameter which gives the index of dissolved compounds both organic and inorganic present in wastewater (Dhall et al. 2013). Adsorption processes are usually thought of as a polishing process for wastewater streams having a number of persistent pollutants after biological treatment (Amosa et al. 2015).

The application of statistical experimental design techniques such as response surface methodology, in the analysis of adsorption process, as was performed in this paper, has been associated with the following benefits: (1) all experimental units are used in evaluating effects, resulting in the most efficient use of resources; (2) the effects are evaluated over a wide range of conditions with minimum of resources: (3) a factorial set of treatments is optimized for estimating main effects and interactions (Annadurai et al. 2002). Response surface method (RSM) can be defined as a collection of statistical techniques for designing experiments, building models, evaluating the effects factors and searching for the optimum conditions (Nwabanne and Igbokwe 2012). RSM is based on the use of second-order equation. However, not all processes fit to a second-order polynomial. Also, not all systems containing curvature are well accommodated by the second-order polynomial. Conventional and classical methods of studying a process involve studying of one factor at a time by maintaining other factors at unspecified constant levels. This approach does not represent the combined effect of all the parameters involved in the process (Jia-Hong et al. (2012). This method is not only tedious but time-consuming. These limitations of a classical method can be effectively eliminated by optimizing all the parameters collectively by statistical experimental designs such as response surface methodology (Arenas et al. 2006). These designs reduce the total number of experiments in order to achieve the best overall optimization of the process. Full factorial design determines the effect of each factor on response as well as how the effect of each factor varies with the change in level of the other factors (Montgomery 1997; Brasil et al. 2005). Interaction effects of different factors could be attained using design of experiments (Brasil et al. 2005). The aim of this work is the removal of dissolved solids from fibre cement industry effluent by adsorption onto activated carbon prepared from coal by optimization of process variables such as effluent pH, adsorbent dose, temperature and time using response surface methodology.

### **Materials and methods**

# Adsorbent preparation

Activated carbon was prepared as reported in Ani et al. (2012). Coal was collected from Enugu Coal Mine,



south-east of Nigeria. The coal was first washed with distilled water several times to remove dirt and impurities and sun-dried for 72 h to remove moisture until constant weight was obtained so as to facilitate easy crushing and grinding. The washed and dried raw material was pulverized using a laboratory milling machine and sieved to particle sizes of 2.5–5.0 mm. One hundred grams of dry pulverized coal was impregnated overnight with concentrated phosphoric acid (60%) at ratio of acid to coal of 1:1 on weight basis and dried in an oven at 110 °C for 1 h. The prepared coal sample was carbonized at 700 °C for 2 h, using Vecstar muffle furnace model LF3. The carbonized coal sample was washed and dried. Finally, the prepared activated coal adsorbent was tested and used for adsorptive removal of dissolved solids from a fibre cement industry wastewater effluent.

### Physicochemical characterization of adsorbent

Fourier transform infrared (FTIR) analysis was performed using IR Tracer-100 Shimadzu spectrophotometer in the range 400–4000 cm<sup>-1</sup> to give the vibration frequencies of the adsorbents lattice which result from stretching of bending modes of the functional groups present in the activated coal adsorbent particles using the KBr pellet method. The structure and morphology of coal and activated coal adsorbent molecules were examined by scanning electron microscope (SEM) (Phenom Pro X).

## Dissolved solids adsorption studies

Batch adsorption experiments were performed at room temperature (30 °C) to study the effect of effluent pH, adsorbent dosage, and contact time. Each experiment was carried out in Erlenmeyer flasks containing 20 mL effluent solution by shaking the flasks at moderate speed for period contact time of 60 min. Samples were withdrawn at predetermined time intervals (0–60 min) and filtered through Whatman No. 42 filters. The residual DS concentration in the supernatant was determined using the spectrophotometer (Jenway 6305) at 290–292 nm. To evaluate the effect of temperature on the DS adsorption, constant process parameters (pH, adsorbent dosage and DS concentration) were applied at four temperatures (20, 25, 30 and 40 °C). The removal efficiency of DS molecules was calculated by Eq. (1):

$$R = \frac{C_i - C_t}{C_i} \times 100 \tag{1}$$

where R is the removal efficiency (%) of the DS, and  $C_i$  and  $C_t$  are the initial and residual concentrations of DS (mg L<sup>-1</sup>), respectively.

# Experimentation and optimization of adsorption process

Optimum condition for the adsorption of DS by activated coal adsorbent was determined by means of central composite design (CCD) under RSM. Optimization studies were carried out by studying the effect of four variables including effluent pH, adsorbent dosage, contact time and adsorption temperature. Each factor was studied at a two-level and four-factor (2<sup>4</sup> or 2 by 4) central composite design, that is, fractional factorial design  $(2^{4-1}+2\times4+5)$  resulting in 21 experiments. The factor levels are given in Table 1. The matrix for the four variables: effluent pH, adsorbent dosage, contact time and adsorption temperature was varied at two levels (-1 and +1). The lower level variable was designated as "-1" and higher level as "+1." The experiments were performed in random manner to avoid systematic error. To analyse the factorial design, the original measurement units for the experimental factors (uncoded units) were transformed into coded units. The factor levels were coded as -1 (low) and +1 (high). The response was expressed as the adsorption efficiency (%). The licensed software, Design Expert-7.0.7.1, was used to design and analyse the experimental matrix, in order to measure the effect of various factors on the adsorption efficiency. The levels and ranges of the studied factors are presented in Table 1. The empirical equation which explains the behaviour of the system is represented as

$$Y = \beta_0 + \sum_{i=1}^4 \beta_i X_i + \sum_{i=1}^4 \beta_{ii} X_i^2 + \sum_{i=1}^4 \sum_{j=i+1}^4 \beta_{ij} X_i X_j$$
(2)

where i and j are linear, quadratic coefficients,  $\beta_0$  is the constant coefficient,  $\beta_i$  is the linear coefficient,  $\beta_{ii}$  is the interactive coefficient and  $\beta_{ii}$  is the quadratic coefficient.

The transformation of coded value to actual value was obtained using Eq. (3):

$$X_i = \frac{x_i - \overline{x}}{\Delta x} \tag{3}$$

where  $X_i$  the coded value of *i*-th factor,  $x_i$  the current actual value,  $\bar{x}$  mean value for actual values,  $\Delta x$  difference between the mean actual value and actual value.

The design matrix of all the factors in coded and actual values for all the experimental runs is given in Table 2.

Table 1 Studied range of each factor in actual and coded form for adsorption of fibre cement industry effluent using coalbased activated carbon

Factor	Unit	Low level	High level	-α	+α	0 level
pH (A)		5(-1)	7(+1)	4(-2)	8(+2)	6
Adsorbent dosage (B)	mg/L	20(-1)	40(+1)	10(-2)	50(+2)	30
Contact time (C)	Min	30(-1)	50(+1)	20(-2)	60(+2)	40
Adsorption temperature (D)	°C	25(-1)	35(+1)	20(-2)	40(+2)	30

### **Results and discussion**

### Characterization of activated coal

The morphology of the adsorbent was studied by SEM. Figure 1a and b shows the SEM images of raw coal and activated coal after DS adsorption, respectively. It was observed that raw coal particles consists of fine particles which did not have regular and fixed shape and size exhibiting low porosity (Fig. 1a). However, the smooth surface of raw coal became thicker and coarser after DS adsorption (Fig. 1b), exhibiting an irregular and heterogeneous surface morphology with moderately developed porous structure. The formation of these cavities resulted from the evaporation of the dehydrating agent (H<sub>3</sub>PO<sub>4</sub>) during carbonization, leaving the space previously occupied by the reagent (El-Hendawy et al. 2008).

FTIR spectroscopy was used to detect the presence of binding groups in the coal, COBAC (before and after adsorption) (Table 3). In the coal spectra, the absorbance peak appears in 2856 cm<sup>-1</sup> and was assigned to the C–H stretching mode of aldehyde. The spectra show a broadband around 3646–3618 cm<sup>-1</sup> which is due to O–H stretching mode and 1600–1463 which was attributed to C=C aromatic (Anisuzzaman et al. 2015). However, carbonization of the coal resulted in the formation of additional functional groups namely N–H amine, C–H aromatic, C–N amine and C–O alcohol or phenol. After DS adsorption, the chemical groups oxidized to C–H stretch CH<sub>2</sub> or CH<sub>3</sub>, C=O aldehyde, C–O stretch ether and C=C aromatic.

## **Characterization of fibre cement effluent**

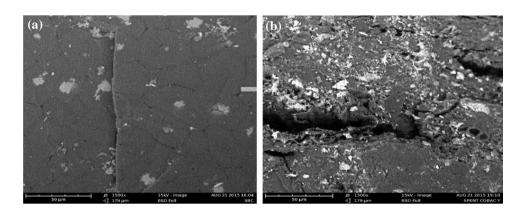
The characteristics of the fibre cement effluent (FCIE) were analysed using standard methods and are presented in Table 4. The table depicts the characteristics of the original effluent stream as well as the characteristics after adsorption. The adsorption treatment ultimately reduced the concentration of the pollutants to levels well below the WHO specifications for recycled effluent streams. The alkaline nature of the FCIE could be attributed to the type of raw materials used in the processing of the product. Silica, cement, cellulose and water were the major raw materials used for the formulation of fibre cement products. The measured value of the total dissolved solids for FCIE was 1578.00 mg/L, the



Table 2 Experimental design matrix for adsorption of fibre cement industry effluent using coal-based activated carbon

Run order	pH (A)		Adsorption dosage (mg/L) (B)		Adsorption time (min) ( <i>C</i> )		Temperature (°C) (D)	
	Coded	Real	Coded	Real	Coded	Real	Coded	Real
1	+1	7	+1	40	+1	50	-1	25
2	+1	7	+1	40	<b>-1</b>	30	-1	25
3	+1	7	-1	20	+1	50	+1	35
4	<b>-</b> 1	5	+1	40	<b>-</b> 1	30	+1	35
5	+1	7	-1	20	<b>-</b> 1	30	+1	35
6	<b>-</b> 1	5	-1	20	+1	50	-1	25
7	<b>-</b> 1	5	+1	40	+1	50	+1	35
8	<b>-</b> 1	5	-1	20	<b>-</b> 1	30	-1	25
9	-2	4	0	30	0	40	0	30
10	+2	8	0	30	0	40	0	30
11	0	6	-2	10	0	40	0	30
12	0	6	+2	60	0	40	0	30
13	0	6	0	30	-2	20	0	30
14	0	6	0	30	+2	60	0	30
15	0	6	0	30	0	40	-2	20
16	0	6	0	30	0	40	+2	40
17	0	6	0	30	0	40	0	30
18	0	6	0	30	0	40	0	30
19	0	6	0	30	0	40	0	30
20	0	6	0	30	0	40	0	30
21	0	6	0	30	0	40	0	30

**Fig. 1** SEM images of **a** raw coal and **b** activated coal after DS adsorption



value being much higher than the WHO recommended limit of 500.00 mg/L, showing that the wastewater was considerably polluted prior to treatment.

# Model formulation and validation for the adsorption of fibre cement industry effluent

The individual run in experimental design plan was carried out based on the adsorption of fibre cement effluent (FCIE) using coal-based activated carbon (COBAC) and the responses were measured and are presented in Table 5. The adsorption efficiency of COBAC for the treatment of fibre cement industry effluent depends on whether there

is significant variation in the combination of the process parameters. The empirical relationship between adsorption efficiency  $(Y_2)$  and the four process variables namely pH, Adsorbent dosage, contact time and adsorption temperature coded A, B, C and D, respectively, which was obtained by Design expert 7.0.7.1, is given in Eq. (4).

$$Y_2 = 94.37 + 3.5A - 0.087B + 1.84C + 2.48D + 3.91AB + 5.44AC - 0.52AD - 2.94BC + 1.44BD - 0.063CD - 7.33A^2 - 0.68B^2 - 2.45C^2 - 2.39D^2$$
(4)



Table 3 FTIR data of coal and COBAC

Sample	IR spectral bands (cm <sup>-1</sup> )
Coal	3646–3618 (O–H stretch alcohol)
	2856 (C-H stretch aldehyde)
	1600-1463 (C=C aromatic)
COBAC (before DS adsorption)	3437 (OH alcohol or phenol)
	3287 (N–H amines NH <sub>2</sub> )
	2910 (C-H aromatic)
	2545 (C-H aliphatic)
	1437 (C=C aromatic)
	1248 (C-N amine)
	1063 (C–O alcohol or phenol)
	769 (Substitution in benzene ring)
COBAC (after DS adsorption)	2953–2853 (C–H stretch $CH_2$ or $CH_3$ )
	1747–1645 (C=O aldehyde)
	1088 (br) (C–O stretch ether)
	1574–1460 (C=C aromatic)

where  $Y_2$  is the response variable (adsorption efficiency) and A–D are the coded values of the independent variables. Equation (4) represents the quantitative effect of the factors (A, B, C and D) upon the response  $(Y_2)$ . Coefficients with one factor represent the effect of that particular factor, while the coefficients with more than one factor represent the interaction between those factors. Positive sign in front of the terms indicates synergistic effect, while negative sign indicates antagonistic effect of the factor.

The adequacy of the proposed model was tested using the Design expert 7.0.7.1 and is given in Table 6. From the sequential test, it can be seen that the model F-value (318.93) of the quadratic model is large compared to the values for the other model terms for the equation. From the statistics test, the coefficient of determination ( $R^2$  = 0.9987) was high; the adjusted  $R^2$  (0.9955) is in close agreement with the predicted  $R^2$  (0.9925) value. Analysis of variance (ANOVA) was applied for estimating the significance of the model at 5% significance level as has been suggested by Lilian and Charles (2008). A model is considered significant if the p value (significance probability value) is less than 0.05. From the p values presented in Table 6, it can be stated that the linear terms A, C and D and interaction terms AB, AC,

BC and BD and quadratic terms  $A^2$ ,  $B^2$ ,  $C^2$  and  $D^2$  are significant model terms. Based on this, the insignificant terms of the model were removed but B was included because of its importance in the process and to maintain the hierarchy; the model reduced to Eq. (5):

$$Y_2 = 94.37 + 3.5A - 0.087B + 1.84C + 2.48D + 3.91AB + 5.44AC - 2.94BC + 1.44BD - 7.33A^2 - 0.68B^2 - 2.45C^2 - 2.39D^2$$
(5)

The experimental data were also analysed to check the correlation between the experimental and predicted adsorption efficiency of COBAC for the treatment of FCIE waste. The actual and predicted plot is shown in Fig. 2. It can be seen from the figure that the data points on the plot were reasonably distributed near to the straight line, indicating a good relationship between the experimental and predicted values of the response, and that the underlying assumptions of the above analysis were appropriate as has been described in a similar work by Chigoziri and Okechukwu (2015). The result also suggests that the selected quadratic model was adequate in predicting the response variables for the experimental data.

# Three-dimensional surface plots for adsorption of fibre cement industry effluent

The three-dimensional response surface plots were generated to estimate the effect of the combinations of the independent variables on the adsorption efficiency. The plots are shown in Figs. 3, 4, 5, 6, 7, 8, 9 and 10. Figure 3 (3-D plot) and Fig. 4 (contour plot) show the dependency of adsorption efficiency on pH and adsorbent dosage. Adsorption efficiency increased as the pH and adsorbent dosage increased. This implies that COBAC was effective in the adsorption of FCIE at pH 4–6 and also as the dosage increased till dosage of about 30 mg/L after which it tended to decrease. Figure 5 (3-D plot) and Fig. 6 (contour plot) show the dependency of adsorption efficiency on pH and adsorption time. Adsorption efficiency increased as both the pH and adsorption time increased up to a point and then decreased. The lowest % removal was recorded at pH 4 (Table 5). Lower adsorption of

**Table 4** Results of the characterization of fibre cement effluent before and after adsorption

Parameter	Before adsorption	After adsorption	WHO standard
pH	12.20	4.40	7.00-8.00
Turbidity (NTU)	450.00	4.00	5.00
TSS (mg/L)	950.00	0.02	50.00
DS (mg/L)	1578.00	8.96	500.00
Alkalinity (mg CaCO <sub>3</sub> /L)	1350.00	60.00	100.00



Table 5 Response (adsorption efficiency, %) data of adsorption of fibre cement industry effluent using coal-activated carbon based on experimental design matrix

Run order	pН	Adsorption dosage (mg/L)	Adsorption contact time (min)	Tem- perature (°C)	Adsorption efficiency (%)
1	7	40	50	25	90
2	7	40	30	25	81
3	7	20	50	35	92
4	5	40	30	35	85
5	7	20	30	35	71.5
6	5	20	50	25	80
7	5	40	50	35	72
8	5	20	30	25	81
9	4	30	40	30	58
10	8	30	40	30	72
11	6	10	40	30	91.8
12	6	40	40	30	88
13	6	30	20	30	81
14	6	30	60	30	88
15	6	30	40	20	79.8
16	6	30	40	40	89.7
17	6	30	40	30	93
18	6	30	40	30	94.6
19	6	30	40	30	94.6
20	6	30	40	30	94.6
21	6	30	40	30	95

obtained a similar trend.

Table 6 Significance of regression coefficients of the adsorption efficiency of coal-based activated carbon in the treatment of fibre cement industry effluent using Design expert 7.0.7.1

Source	Coefficient estimate	Degree of freedom	Sum of square	<i>F</i> -value	p  Value (Prob > F)
Model	94.37	14	1940.17	318.93	< 0.0001
A	3.50	1	98.00	225.53	< 0.0001
B	-0.087	1	1.63	0.16	0.7067
C	1.84	1	0.068	125.17	< 0.0001
D	2.48	1	54.39	112.78	< 0.0001
AB	3.91	1	49.01	140.91	< 0.0001
AC	5.44	1	61.23	544.34	< 0.0001
AD	-0.52	1	1.16	2.67	0.1532
BC	-2.94	1	69.03	158.87	< 0.0001
BD	1.44	1	8.27	19.02	0.0048
CD	-0.063	1	0.031	0.072	0.7975
$A^2$	-7.33	1	1322.48	3043.49	< 0.0001
$B^2$	-0.68	1	23.50	54.08	0.0003
$C^2$	-2.45	1	148.32	341.33	< 0.0001
$D^2$	-2.39	1	140.86	324.17	< 0.0001
Residual		6	2.61		
Cor. Total		19	525.78		

Mean = 84.41; C.V.% = 0.78; PRESS = 14.56; R<sup>2</sup> = 0.9987; Adj. R<sup>2</sup> = 0.9955; Pred. R<sup>2</sup> = 0.9925; Adeq. Precision = 65.190



DS at acidic pH is probably due to the presence of excess H<sup>+</sup> ions competing with the cation groups on the DS for adsorption sites. At pH 4-6, the surface of COBAC may get negatively charged which enhances the positively charged DS cations through electrostatic forces of attraction (Hameed et al. 2008; Cooney 1999). However, increasing initial pH to a value higher than 6 is not a good idea as there has been precipitation of some metal cations to hydroxides leading to electrostatic repulsion. Similar result has been reported by Jumina et al. 2007.

Figure 7 (3-D plot) and Fig. 8 (contour plot) show the dependency of adsorption efficiency on adsorbent dosage and adsorption time. Adsorption efficiency increased speedily as adsorption time increased and gradually as adsorbent dosage increased. Figure 9 (3-D plot) and Fig. 10 (contour plot) show the dependency of adsorption efficiency on adsorbent dosage and adsorption temperature. Adsorption efficiency increased gradually as adsorption temperature and adsorbent dosage increased up to a point and then decreased slightly. The initial faster rate may be due to the availability of the uncovered surface area of the adsorbents, since the adsorption kinetics depends on the surface area of the adsorbents. As a consequence, some portion of the active adsorbent sites may be blocked with the passage of time; hence, the rate becomes slower and reaches equilibrium when the surface becomes almost saturated. Igwe and Abia (2006)

The adsorption process was optimized with the design expert to obtain optimal conditions for the process. Adsorption of FCIE using COBAC under the obtained

Fig. 2 Plot of predicted values versus actual values for adsorption of fibre cement industry effluent using coal-based activated carbon

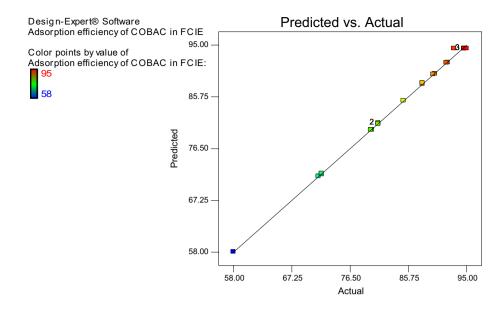


Fig. 3 3D plot showing the effect of pH and adsorbent dosage on adsorption efficiency in the treatment of fibre cement industry effluent using coalbased activated carbon

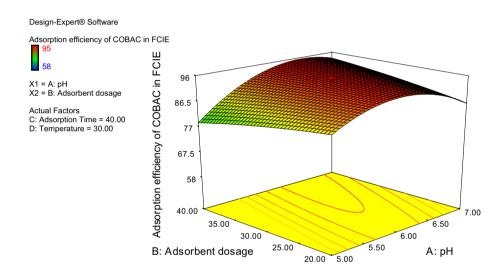


Fig. 4 Contour plot showing the effect of pH and adsorbent dosage on adsorption efficiency in the treatment of fibre cement industry effluent using coalbased activated carbon

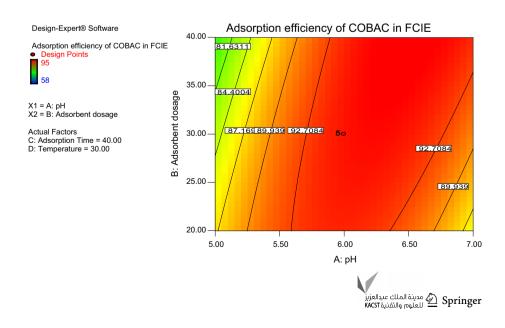


Fig. 5 3D plot showing the effect of pH and adsorption time on adsorption efficiency in the treatment of fibre cement industry effluent using coal-based activated carbon

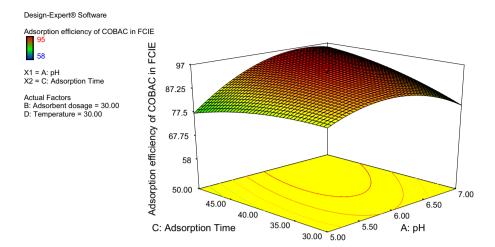


Fig. 6 Contour plot showing the effect of pH and adsorption time on adsorption efficiency in the treatment of fibre cement industry effluent using coal-based activated carbon

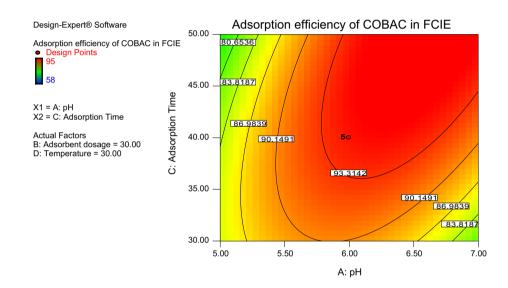


Fig. 7 3D plot showing the effect of adsorbent dosage and adsorption time on adsorption efficiency in the treatment of fibre cement industry effluent using coal-based activated carbon

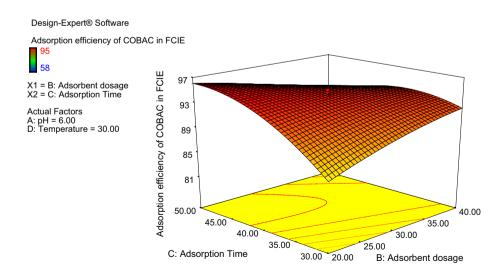




Fig. 8 Contour plot showing the effect of adsorbent dosage and adsorption time on adsorption efficiency in the treatment of fibre cement industry effluent using coal-based activated carbon

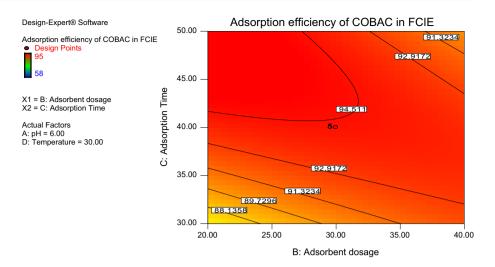


Fig. 9 3D plot showing the effect of adsorbent dosage and adsorption temperature on adsorption efficiency in the treatment of fibre cement industry effluent using coal-based activated carbon

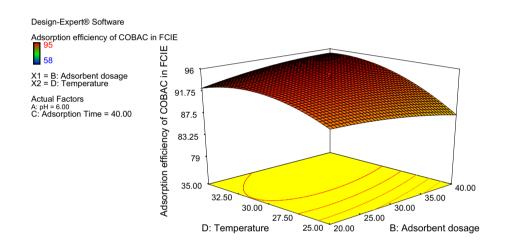
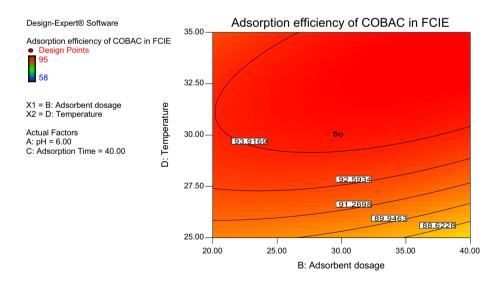


Fig. 10 Contour plot showing the effect of adsorbent dosage and adsorption temperature on adsorption efficiency in the treatment of fibre cement industry effluent using coal-based activated carbon



optimum operating conditions was carried out in order to evaluate the precision of the quadratic model. The experimental value and the predicted values are given in Table 7. Comparing the experimental and predicted results, it can be seen that the error between the experimental and predicted is less than 0.4%; therefore, it can be concluded that the generated model has sufficient



Table 7 Results of the model validation for the adsorption of FCIE using COBAC

Experiment	pН	Adsorbent dosage (mg/L)	Adsorption time (min)	Temperature (°C)	Experimental yield (%)	Predicted yield (%)
	A	В	C	D		
Adsorption of FCIE using COBAC	6.27	27.60	48.00	31.00	96.20	96.50

accuracy to predict the adsorption efficiency for the process (Leng and Pinto 1996; Chen et al. 1997).

### **Conclusions**

Scanning Electron microscopy showed the adsorption of dissolved solids on the coal-based activated carbon. FTIR spectrophotometer confirmed the presence of functional groups on the investigated samples. The selected quadratic model can be used in predicting response variables under the same conditions of the experiments. COBAC was effective in the adsorption of FCIE at pH 4–6 and also as the adsorbent dosage increased till dosage of about 30 mg/L after which it tended to decrease. Adsorption efficiency increased speedily as adsorption time increased and gradually as adsorbent dosage increased. Adsorption efficiency also increased gradually as adsorption temperature and adsorbent dosage increased up to a point and then decreased slightly. The generated model has sufficient accuracy to predict the efficiency of the process.

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### References

- Abedi KJ, Eslamian SS, Khaleghi M (2013) Performance of tire powder for absorption of lead, zinc and manganese heavy metals and determination of kinetic and adsorption isotherms. Irrig Sci Eng (JISE) Sci J Agric 36(3):23–30
- Adedinni A (2018) Coal: revenue source in need of revitalization. business A.M. www.businessamlive.com/coal-revenue-sourc e-in-need-of-revitalization/. Accessed 28 Jan 2019



- Akpomie KG, Ezeofor CC, Olikagu CS, Odewole OA, Ezeorah CJ (2018) Abstraction and regeneration potential of temperature enhanced rice husk montmorillonite combo for oil spill. Environ Sci Pollut Res https://doi.org/10.1007/s11356-0183425-9
- Alkhatib M, Mamun A, Akbar I (2014) Application of response surface methodology (RSM) for optimization of colour removal from POME by granular activated carbon. Int J Environ Sci Technol 12:1295–1302. https://doi.org/10.1007/s1376 2-014-0504-4
- Amosa MK, Jami MS, Alkhatib MFR, Jimat DN, Muyibi SA (2015) A two-step optimization and statistical analysis of COD reduction from biotreated POME using empty fruit bunch-based activated carbon produced from pyrolysis. Water Qual Expo Health 7:603–616. https://doi.org/10.1007/s1243-015-0176-4
- Amosa MK, Jami MS, Alkhatib MFR (2016) Electrostatic biosorption of COD, Mn and H<sub>2</sub>S on EFB-based activated carbon produced through steam pyrolysis: an analysis based on surface chemistry, equilibria and kinetics. Waste Biomass Valoriz 7:109–124. https://doi.org/10.1007/s12649-015-9435-7
- Ani JU, Nnaji NJ, Okoye COB, Onukwuli OD (2012) Factorial analyses, equilibrium studies, kinetics and thermodynamics of the removal of suspended particles from an industrial effluent on coal based activated carbon. Int J Chem Sci 10(3):1765–1784
- Anisuzzaman SM, Joseph CG, Daud W, Krishnaiah D, Ye H (2015) Preparation and characterization of activated carbon from *Typha* orientalis leaves. Int J Ind Chem 6:9–21
- Annadurai G, Juang R, Lee D (2002) Factorial design analysis for adsorption of dye on activated carbon beads incorporated with calcium alginate. Adv Environ Res 6:191–198
- Arenas LT, Lima EC, Santos AAD, Vaghetti JCP, Coasta TMH, Benvenutti EV (2006) Use of statistical design of experiments to evaluate the sorption capacity of 1,4-diazoniabicycle [2,2,2] octane silica chloride for Cr(VI) adsorption. Coll Surf A Physiochem Eng Aspects 297:240–248
- Baba AH, Saba AM (2006) Effect of process variables on the saponification, acid and iodine values of local palm kernel oil purified using locally processed activated carbon. J Niger Soc Chem Eng 21:10–18
- Brasil JL, Martins LC, Ev RR, Dupont J, Dias SLP, Sales JAA, Airoldi C, Lima EC (2005) Factorial design for optimization of flow injection preconcentration procedure for copper (II) determination in natural waters, using 2-aminomethylpyridine grafted silica gel as adsorbent and spectrophotometric detection. Int J Environ Anal Chem 15:475–491
- Chen J, Wey N, Yan M (1997) Theoritical and experimental study of metal capture during incineration process. J Environ Eng 123:1100–1106
- Chigoziri NJ, Okechukwu EO (2015) Response surface optimization of the inhibition efficiency of *Gongronema latifolium* as an inhibitor for aluminium corrosion in HCl solutions. Int J Mater Chem 5:4–13
- Cooney DO (1999) Adsorption design for waste water treatment. CRC Press, Boca Raton
- Dhall P, Girdhar M, Mohan A, Kumar R, Kumar A (2013) Biological removal of pulp and paper TDS. Biosci Biotechnol Res 10(2):691–697

- Ekpete OA, Marcus AC, Osi V (2017) Preparation and characterization of activated carbon obtained from plantain (*Musa Paradisiaca*) fruit stem. J Chem 2017:6. https://doi.org/10.1155/2017/8635615
- El-Hendawy ANA, Alexander AJ, Andrews RJ, Forrest G (2008) Effect of activation schemes on porous, surface and thermal properties of activated carbon prepared from cotton stalks. J Anal Appl Pyrol 82:272–278
- El-Sayed GO, Yehia MM, Asaad AA (2014) Assessment of activated carbon prepared from concorb by chemical activation with phosphoric acid. Water Resour Ind 7–8:66–75
- Falade MO, Adeyeye P (2016) A profile of Nigeria's solid minerals a detailed desk review. https://doi.org/10.13140/rg.2.1.4233.9600
- Hameed BH, Mahmood DK, Ahmed AL (2008) Equilibrium modeling and kinetic studies on the adsorption of basic dye by a low-cost adsorbent: coconut (*Cocos nucifera*) bunch waste. J Hazard Mater 158:65–72
- Harry M, Francisco R (2006) Activated carbon. Elsevier Science & Technology Books, London
- Igwe JC, Abia AA (2006) Sorption kinetics and intraparticulate diffusivity of As (III) bioremediation from aqueous solution, using modified and unmodified coconut fibre. ECLETICA Quim 31:23-29
- Jia-Hong K, Kaimin S, Chiou Liang L, Ming-yen W (2012) Simulation of agglomeration/defluidization inhibition process in aluminium—sodium system by experimental and thermodynamic approaches. Powder Technol 224:395–403

- Leng CC, Pinto NG (1996) An investigation of the mechanisms of chemical regeneration of activated carbon. Ind Eng Chem Res 35:2024–2031
- Lilian SK, Charles EG (2008) Analysis of variance: is there a difference in means and what does it mean? J Surg Res 144:158–170
- Montgomery DC (1997) Design and analysis of experiments, 4th edn. Wiley, New York, pp 1–26
- Muyibi SA, Tajari T, Jami MS, Amosa MK (2014) Removal of organics from treated palm oil mill effluent (POME) using powdered activated carbon (PAC). Adv Environ Biol 8(3):590–595
- Nwabanne JT, Igbokwe PK (2012) Application of response surface methodology for preparation of activated carbon from palmyra palm nut. N Y Sci J 5(9):18–25
- Okiemen FE, Okojah FI, Okeimen CO, Wuana RA (2004) Preparation and evaluation of activated carbon from rice husk and rubber seed shell. ChemClass J 191–196
- Sarjono Jumina RE, Paramitha B, Hendaryani I et al (2007) Adsorption characteristics of Pb(II) and Cr(III) onto C-4-methoxyphenylca-lix[4]resorcinarene in batch and fixed bed column systems. J Chin Chem Soc 54:1167–1178
- Shamsudinn MS, Yusoff NRN, Sulaiman MA (2016) Synthesis and characterization of activated carbon produced from Kenaf core fiber using H<sub>3</sub>PO<sub>4</sub> activation. Proced Chem 19:558–565

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