Optimization of Acrylonitrile Removal by Activated Carbon-Granular Using Response Surface Methodology

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A statistical Box–Behnken design of experiments was performed to evaluate the effects of individual operating variables and their interactions on the acrylonitrile (AN) removal of $C_0 = 100$ mg/L as fixed input parameter. The variables examined in this study included activated carbon-granular (AC) dosage, *w*, temperature, *T*, and time of contact, *t*. The significant variables and optimum conditions were identified ($w = 4$ g/L, $T = 30°C$, and $t = 120$ min with AN uptake of 23.97 mg/g of AC) from statistical analysis of the experimental results using response surface methodology (RSM).

Un plan des expériences statistiques de Box–Behnken a été conçu afin d'évaluer les effets des variables de fonctionnement individuelles et leurs interactions sur l'élimination de 100 mg/L d'acrylonitrile (AN) comme paramètre d'entrée fixe. Les variables examinées dans le cadre de cette étude comprenaient le dosage de charbon granulaire activé (*w*), la température (*T*) et le temps de séjour (*t*). Les variables importantes et les conditions optimales ont été déterminées (et minimales avec une absorption de 23,97 mg/g d'AN) à partir de l'analyse statistique des résultats expérimentaux au moyen de la méthodologie de la surface de réponse (MSR).

Keywords: Box–Behnken, acrylonitrile (AN), response surface methodology (RSM), adsorption, granular activated carbon (AC)

INTRODUCTION

Acrylonitrile (AN) is a colourless, volatile liquid that is soluble in water and most common organic solvents such as acetone, benzene, carbon tetrachloride, ethyl acetate, and toluene (Klein et al., 1957). The technical-grade product always contains a polymerization inhibitor. AN is a reactive chemical that polymerizes spontaneously and can explode when exposed to flame (Hazardous Substances Database, 2002). AN is produced commercially by the process of propylene ammoxidation, in which propylene, ammonia and air are reacted in a fluidized bed in the presence of a catalyst (EPA, 1984, 1985). AN is an important industrial chemical. It is used extensively in the manufacture of synthetic fibres, resins, plastics, elastomers, and rubber for a variety of consumer goods such as textiles, dinnerware, food containers, toys, luggage, automotive parts, small appliances, and telephones (SRI, 1984).

AN is reasonably anticipated to be a human carcinogen based on sufficient evidence of carcinogenicity in experimental animals (IARC, 1979, 1982, 1987, 1999; SRI, 1984; ATSDR, 1990). The primary routes of potential human exposure to AN are inhalation and dermal contact. AN, an EPA priority pollutant, is among the top 50 most widely used industrial compounds in the USA (Keith and Telliard, 1979). AN induced permanent toxicity (Carrera et al., 2007) and symptoms of chemical bronchitis/asthma (Mohan et al., 2006).

Exposure to AN may occur during its manufacture and production; greater potential for exposure exists for workers using AN to make other products in factories where the compound is not easily contained (DPIM, 1989). AN is found in the wastewater mainly because of industrial activities (IARC, 1979). Several examples of water contamination following spills have been reported (Miller and Villaume, 1978). In most of the industrial effluents, AN concentration was observed in the range of 50–100 mg/L (World Health Organization, 1983).

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Several treatment methods, particularly biological or a blend of wet oxidation followed by biological treatment, have been used for the removal of AN from wastewaters. Adsorption is also a very attractive treatment method for the removal of toxics from wastewaters (Mishra et al., 1995; Desai and Ramakrishna, 1998; Kumar et al., 2008a,b). Activated carbon (AC) is an adsorbent most widely used for various purification processes. Adsorption on activated carbon is one of the well-established and effective methods for the removal and preconcentration of herbicides and metal ions from a solid liquid system (Huang and Blankenship, 1984; McBay and Murad, 1987). This is owing to its distinguished properties such as extensive pore surface area, developed internal pore structure, and unique surface chemistry. The unique and versatile adsorptive efficiency arises from the large surface area, micropore structure, high absorption capacity, and high degree of surface reactivity (Pal et al., 2006).

The objective of the present study is to investigate the feasibility of using granular activated carbon (GAC) for the adsorptive removal of AN from aqueous solution having AN concentration of 100 mg/L. The study uses the Box–Behnken design in the optimization of experiments using response surface methodology (RSM) to understand the effect of important parameters and their interactions on the adsorption process (Box and Behnken, 1960). The parameters used are the GAC dosage (w), temperature (T), and contact time (t). Since, the AN gets polymerized in the acid and/or basic medium, hence the effect of pH was not studied in the present work.

EXPERIMENTAL

Adsorbent and Adsorbate

Adsorption of (AN) was studied using GAC. The commercial grade activated carbon-granules was obtained S.D. Fine Chemicals Ltd (Mumbai, India). Detailed physico-chemical and surface characteristics of the GAC have been presented in Table 1 (Kumar et al., 2008b). Laboratory grade AN, inhibited with 200 mg/L hydroquinone mono methyl ether and supplied by S.D. Fine Chemicals Ltd, was used for the preparation of synthetic aqueous solution of AN of initial concentration $C_0 = 100$ mg/L. The required quantity of the adsorbate was accurately weighed and dissolved in a small amount of double distilled water (DDW) and subsequently made up to 1 L in a measuring flask by adding DDW. Fresh stock solution as required was prepared every day and was kept at ambient conditions in a glass stoppered glass container. The C_0 was ascertained before the start of each experimental run.

Table 1. Physico-chemical and surface characteristics of adsorbent

Characteristic	GAC
Proximate analysis (sample as received)	
Moisture (%)	7.70
Ash (%)	9.73
Volatile matter (%)	6.49
Fixed carbon (%)	76.08
Bulk density (kg/m^3)	725
Carbon pH	10.38
pH_{PZC}	10.33
Heating value (mJ/kg)	6.88
Average particle size	3–5 mm
Ultimate analysis (dry basis) (%)	
C	77.16
H	5.105
N	0.025
S	0.741
Chemical analysis of ash (%)	
Insoluble matter	2.90
Silica	2.50
Ferric and alumina	3.90
CaO	84.0
Mg	2.90
Surface area (m^2/g)	
BET	870.57
Langmuir	1015.43
t-plot micropore	965.29
t-plot external	50.14
Single point	863.19
BJH adsorption cumulative	76.16
Pore volume (cm^3/g)	
Single point total pore volume	0.53
t-plot micropore volume	0.33
BJH adsorption cumulative	0.07
Pore size (Å)	
BET Adsorption average pore width	24.67
BJH adsorption average pore diameter	42.00
Functional groups (meq/g)	
Carboxylic	1.55
Lactonic	0.10
Phenolic	0.15
Carbonyl	0.10
Phenol number	21
Iodine number	875

Batch Experimental Program

For each experiment, 50 mL of AN solution of known C_0 and a known amount of the GAC were taken in a 100 mL air-tight conical flask with a glass stopper. This mixture was agitated in a temperature-controlled shaking water bath at a constant shaking speed of 250 rpm. The uptake (q_t (mg/g)) of AN by GAC at any time, t was calculated as:

$$q_t = \frac{(C_0 - C_t)V}{w} \quad (1)$$

where C_0 is the initial adsorbate concentration (mg/L), V the volume of the wastewaters in the flask (L), C_t the adsorbate concentration (mg/L) after time t, and w the mass of the GAC (g) used in the experiment.

Analytical Measurements

The concentration of AN in the aqueous solution was determined by using high performance liquid chromatography (HPLC) (Waters, Bangalore, India) at 196 nm wavelength (Kumar et al., 2008b). Noval Pack, C_{18} column (size: 3.9 mm × 150 mm) was used in the HPLC for the measurement of AN. Degassed organics-free water was used as the solvent, keeping a flow rate of 1 mL/min as per specifications given in the user manual of the instrument. The calibration curve of the peak area versus AN concentration was used for the determination of the unknown concentration of AN from a sample. Wherever needed, the sample was appropriately diluted to have the AN concentration in the calibration range.

Box–Behnken Design

Box and Behnken (1960) have proposed some three-level designs for fitting response surfaces. Box–Behnken design requires an

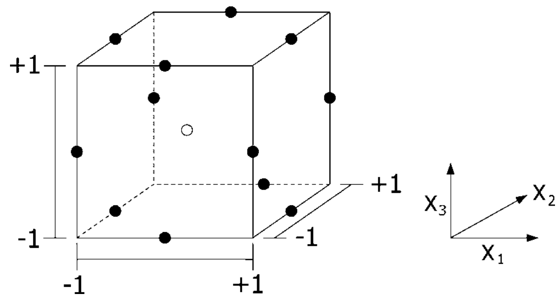

Figure 1. A Box–Behnken design for three factors ($k = 3$).

experiment number according to $N = k^2 + k + c_p$, where k is the factor number and c_p the replicate number of the central point. Such design is formed by combining 2^k factorials with incomplete block design. Box–Behnken is a spherical, revolving design, viewed as a cube and consists of a central point and the middle points of the edges. As a result Box–Behnken design is usually very efficient in terms of the less number of runs required, and is either rotatable or nearly rotatable as shown in Figure 1. A three-variable Box–Behnken design is presented by Evans (2003).

It has been applied for optimization of several chemical and physical processes (Tan et al., 2008a,b,c). This design is generally used for fitting the second order model as given below by manual regression method

$$q = \beta_0 + \sum_{i=1}^{n} \beta_i x_i + \left(\sum_{i=1}^{n} \beta_{ii} x_i \right)^2 + \left(\sum_{i=1}^{n-1} \sum_{j=i+1}^{n} \beta_{ij} x_i x_j \right) + \varepsilon \qquad (2)$$

where $\beta_0 =$ constant, β_i the slope or linear effect of the input factor x_i, β_{ij} the linear by linear interaction effect between the input factor x_i and x_j, β_{ij} the quadratic effect of input factor x_i (Benyounis et al., 2005; Hameed et al., 2008). The detail about the Box–Behnken design and second order model has been presented (Kumar et al., 2008a).

Cubic model was found to be aliased and cannot be used for further modelling of experimental data. A model is aliased means that not enough experiments have been run to independently estimate all the terms for that model. Whenever, there are fewer independent points in the design than there are terms in the model, some parameters cannot be estimated independently. A model is aliased means that model is inappropriate for further investigation (Kumar et al., 2008).

In the present study, the three-level, three-factorial Box–Behnken experimental design is applied to investigate and validate adsorption process parameters, affecting the removal of AN onto GAC. The adsorbent dose (4–36 g/L), temperature (30–60°C) and agitation time (5–295 min) are variable input parameters, while the AN concentration of 100 mg/L was maintained as a constant input parameter, at its natural pH value of 6.46. The factor levels were coded as −1 (low), 0 (central point), and +1 (high) (Evans, 2003).

Table 2 shows the experimental parameters and the experimental Box–Behnken design levels used. RSM was applied to the experimental data using statistical software, Design-expert V6 (trial version).

Table 2. Experimental design levels of chosen variables

Variables	Levels		
Coded level	Low (−1)	Middle (0)	High (+1)
w: dose (g/L)	4	20	36
T: temp (°C)	30	45	60
t: time (min)	5	150	295

Table 3. Experimental and predicted values of q_t (mg/g) for AN onto GAC

Std order	Run order	w	T	t	$q_{t,exp}$	$\ln(q_{t,exp})$	$q_{t,pre}$	$\ln(q_{t,pre})$
8	1	36	45	295	2.53	0.93	2.32	0.84
10	2	20	60	5	3.31	1.20	3.19	1.16
4	3	36	60	150	2.56	0.94	2.89	1.06
1	4	4	30	150	23.62	3.16	20.91	3.04
2	5	36	30	150	2.54	0.93	2.66	0.98
16	6	20	45	150	4.55	1.52	4.57	1.52
7	7	4	45	295	18.49	2.92	19.89	2.99
17	8	20	45	150	4.50	1.50	4.57	1.52
15	9	20	45	150	4.61	1.53	4.57	1.52
11	10	20	30	295	4.53	1.51	4.76	1.56
3	11	4	60	150	17.55	2.86	16.61	2.81
5	12	4	45	5	10.24	2.33	11.25	2.42
13	13	20	45	150	4.60	1.53	4.57	1.52
9	14	20	30	5	2.99	1.10	3.06	1.12
14	15	20	45	150	4.61	1.53	4.57	1.52
6	16	36	45	5	2.31	0.84	2.14	0.76
12	17	20	60	295	4.06	1.40	3.94	1.37

q_t is the response (acrylonitrile uptake) corresponding to the AN-GAC system used in the present study. $q_{t,exp}$ and $q_{t,pre}$ are experimental and predicted responses.

RESULT AND DISCUSSION

Box–Behnken Statistical Analysis

The most important parameters, which affect the efficiency of AN removal by GAC, are adsorbent dose (w), temperature (T), and time of contact (t) of adsorbate-adsorbent. To study the combined effect of these factors, experiments were performed for different combinations of the physical parameters using statistically designed experiments. The range of values for the input variables is given in Box–Behnken Design Section.

The uptake of AN onto GAC is varying from 2.32 mg/g (minimum) to 23.62 mg/g (maximum). The ratio of minimum to maximum uptake of AN is 10.181, which is greater than 10 suggesting that transformation is required for the present system. Further, it is also found that the AN removal onto GAC represents the growth data, so in the present investigation, natural log transformation is suggested. This can be written as follows:

$$q_t' = \ln(q_t + y)$$

where y is constant and taken to be zero. The results of the q_t (response) for AN onto GAC were measured according to design matrix (Kumar et al., 2007) and the measured responses are listed in Table 3. Analyzing the measured responses by the Design expert software, the fit summary of the output indicates that the

Table 4. Selection of adequate model for AN-GAC adsorption system

Source	Sum of squares	d.f.	Mean square	F	P	Remark
Sequential model sum of squares						
Mean	45.25	1	45.25			
Linear	7.48	3	2.49	27.13	<0.0001	
2FI	0.098	3	0.033	0.30	0.8263	
Quadratic	1.03	3	0.34	35.05	0.0001	Suggested
Cubic	0.068	3	0.023	173.31	0.0001	Aliased
Residual	5.22E−004	4	1.307E−004	—	—	
Total	53.93	17	3.17	—	—	
Source	**Sum of squares**	**d.f.**	**Mean square**	**F**	**P**	**Remark**
Lack of fit tests						
Linear	1.19	9	0.13	1015.61	<0.0001	
2FI	1.10	6	0.18	1398.46	<0.0001	
Quadratic	0.068	3	0.023	173.31	0.0001	Suggested
Cubic	0.000	0	—	—	—	Aliased
Pure error	5.227E−004	4	1.307E−004	—	—	—
Source	**Std. Dev.**	**R^2**	**Adj. R^2**	**Pre. R^2**	**Press**	**Remark**
Model summary statistics						
Linear	0.30	0.8623	0.7185	0.7185	2.44	
2FI	0.33	0.8736	0.7977	0.3788	5.39	
Quadratic	0.099	0.9921	0.9820	0.8746	1.09	Suggested
Cubic	0.011	0.9999	0.9998	—	—	Aliased

quadratic model is statistically highly significant for the present adsorbate-adsorbent system. Cubic model was not recommended for AN-GAC system as the Box–Behnken matrix has sufficient data to interpret the outcome of the present system.

To decide about the adequacy of model for AN removal by GAC, three different tests viz., Sequential model Sum of Squares, Lack of Fit Tests and Model Summary Statistics were carried out in the present study. P values for all the regressions were lower than 0.01 (Table 4). This means that at least one of the terms in the regression equation has a significant correlation with the response variable. As will be seen later, the interaction of two factors (2FI) was not significant using the RSM. The ANOVA table also shows a term for residual error, which measures the amount of variation in the response data left unexplained by the model. The analysis shows that the form of the model chosen to explain the relationship between the factors and the response is correct (Kim et al., 2003).

The statistical significance of the ratio of mean square variation due to regression and mean square residual error was tested using the analysis of variance (ANOVA). ANOVA is a statistical technique that subdivides the total variation in a set of data into component parts associated with specific sources of variation for the purpose of testing hypotheses on the parameters of the model (Huiping et al., 2007). According to ANOVA (Table 5), the F values for all regressions were found to be very high indicating that most of the variation in the response can be explained by the regression equation. The associated P-value is used to estimate whether F is large enough to indicate statistical significance. If P-value is lower than 0.05, then it indicates that the model is statistically significant (Segurola et al., 1999).

The ANOVA result for the AN-GAC system shows the model F-value to be 97.79, which implies that the terms in the model have a significant effect on the response. The model gives R^2 value of 0.99 and an adjusted R^2 value of 0.98. Therefore, it can be

Table 5. ANOVA table for AN-GAC adsorption system

Source	Sum of squares	d.f.	Mean square	F	P
Model	8.61	9	0.96	97.79	<0.0001[a]
w	7.26	1	7.26	742.28	<0.0001[a]
t	0.21	1	0.21	21.58	0.0024[b]
w^2	0.87	1	0.87	88.47	<0.0001[a]
t^2	0.20	1	0.20	20.83	0.0026[b]
wt	0.062	1	0.062	6.37	0.0395[b]
Residual	0.068	7	9.78E−003	—	—
Lack of fit	0.0068	3	0.023	173.31	0.0001[b]
Pure error	5.227E−004	4	1.30E−004	—	—
Cor. total	8.68	16	—	—	—

$R^2 = 0.9921$, predicted $R^2 = 0.9820$; adjusted $R^2 = 0.8746$, adequate precision = 30.03.
[a] Highly significant.
[b] Significant.

assumed that the proposed model does not explain at least 1% of the experimental results. The probability $P < 0.05$ indicates that the model terms are significant at 95% of probability level. Any factor or interaction of factors with $P < 0.05$ is significant. The ANOVA table obtained from the response surface quadratic model shows that w, t, w^2, t^2, and wt, lack of fit and the constant, whose value is 3.09 (see Eq. 4), are significant. The value S/N, which is a measure of the adequate precision is 30.09. A value greater than 4 is desirable in support of the fitness of the model (Muthukumar et al., 2003).

The ANOVA analysis indicates a linear relationship between the main effects of the dose, time, the quadratic relationship with dose, time and the interaction between dose and time. The final mathematical equation in terms of actual factors (confidence level above 95%) as determined by Design-expert software is given below:

$$\ln(q) = +3.09 - 0.14 \times w - 5.60E - 003 \times T + 6.46E - 003 \times t + 1.78E - 003 \times w^2 + 4.96E - 006 \times T^2 - 1.05E - 005 \times t^2 + 3.23E - 004 \times w \times T - 5.39E - 005 \times w \times t - 2.49E - 005 \times T \times t \quad (3)$$

Eliminating the insignificant terms and interactions, the above empirical model equation may be simplified in terms of actual factors to

$$\ln(q) = +3.09.14 \times w + 6.46E - 003 \times t + 1.78E - 003 \times w^2 - 1.05E - 005 \times t^2 - 5.39E - 005 \times w \times t \quad (4)$$

A normal probability plot and a dot diagram of these residuals are shown in Figure 2. The data points on this plot lie reasonably close to a straight line, lending support to the conclusion that w, t, w^2, t^2, and wt are the only significant effects and that the underlying assumptions of the analysis are satisfied. Figure 3 shows the relationship between the actual and predicted values of $\ln(q_t)$ for adsorption of AN onto GAC. It is seen in Figure 3 that the developed models are adequate because the residuals for the prediction of each response are minimum, since the residuals tend to be close to the diagonal line.

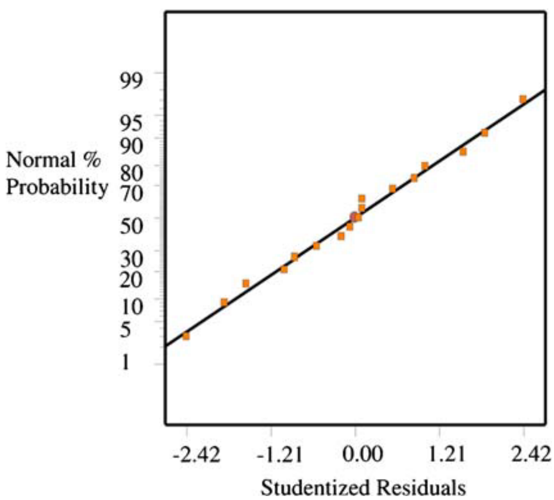

Figure 2. Normal % probability versus studentized residuals. [Colour figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

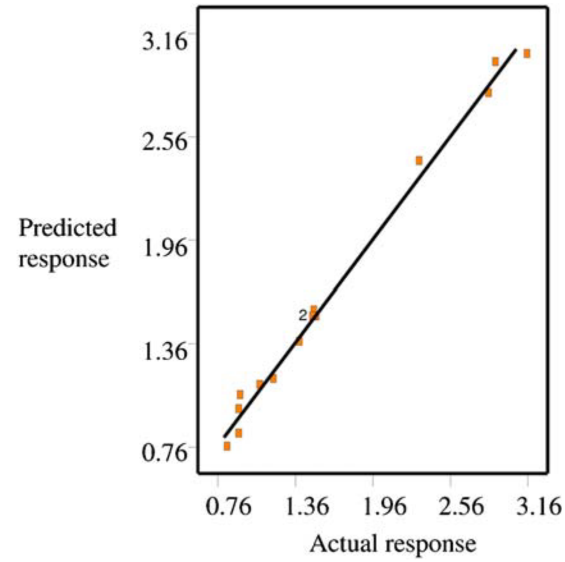

Figure 3. Scatter diagram of predicted response versus actual response for AN-GAC adsorption system. [Colour figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Figures 4a,b and 5a,b show contour and 3D graph for the relationship between dose versus temperature and time of contact for the uptake of AN onto GAC at optimum condition respectively and their values are presented in Table 6.

For the optimization of the process parameters, the point prediction option in the software is used. The optimized parameters obtained from statistical software are $w = 4$ g/L, $T = 30$°C, and $t = 120$ min with uptake of 19.33 mg/g of AN. The temperature induces negative effect for the uptake of AN onto GAC (Kumar et al., 2008b) (Figure 4a,b) and hence it is kept to be the lowest among the range selected. The removal gradually increases with time (Figure 5a,b), so it is taken to be large for the model validation and duplicate confirmatory experiments were

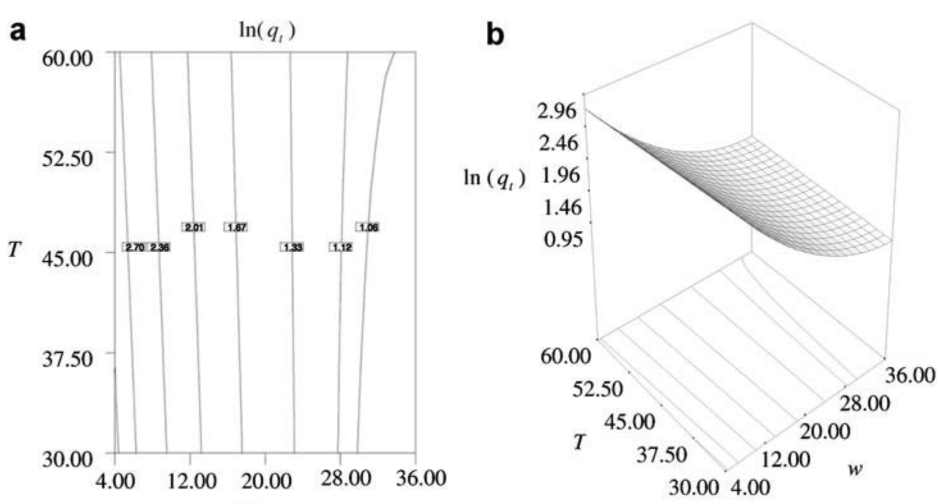

Figure 4. a,b: Contour and 3D graph for AN removal versus adsorbent dose and temperature for AN-GAC adsorption system.

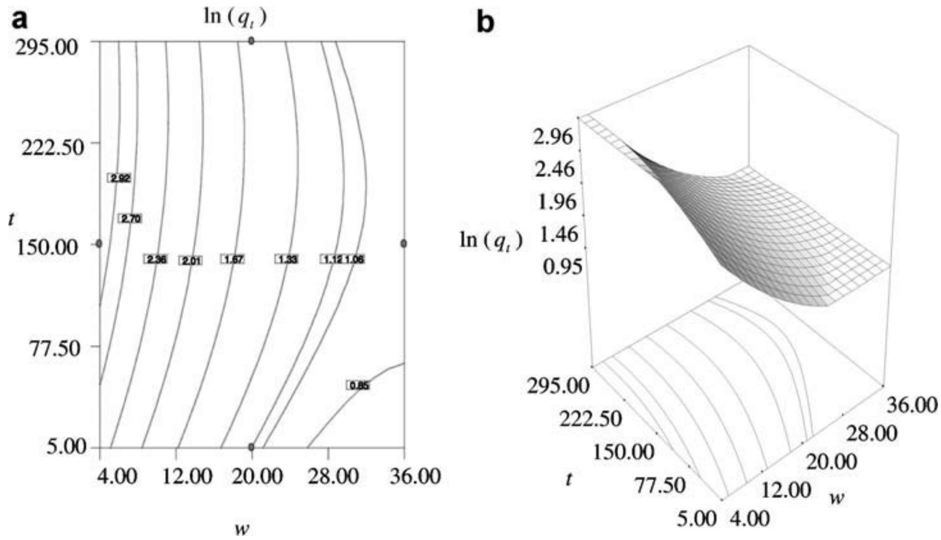

Figure 5. a,b: Contour and 3D graph for AN removal versus adsorbent dose and time for AN-GAC adsorption system.

Table 6. Optimum and confirmative values of the process parameters for maximum efficiency

Processes parameters	Optimized parameter (predicted value)	Confirmation experiment (actual value)
q_t': AN uptake (mg/g)	19.33	23.98
w: dose (g/L)	4	4
T: temperature (°C)	30	30
t: time (min)	120	120

conducted using the above optimized parameters; and the value of q_t is found to be 23.98 mg/g. The experimental result obtained (Table 3, std order experiment no. 1) in the present investigation shows the good agreement with the result obtained by Kumar et al. (2008b).

CONCLUSION

The present study shows the efficacy of GAC for AN removal. The study clearly showed that Box–Behnken design was one of the suitable methods to optimize operating conditions to maximize the AN removal. Graphical response surface and contour plots were used to locate the optimum point. Box–Behnken design was successfully employed for experimental design and analysis of results. Satisfactory prediction equation was obtained to optimize the parameters.

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